

TOWARDS CIRCULAR SANITATION SYSTEMS:
RESOURCE RECOVERY VIA PYROLYSIS OF HUMAN EXCRETA

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Statement of originality

I hereby confirm that the work presented in this thesis is my own. The work and contributions of others have been explicitly referenced and acknowledged.

Signature: _____

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Abstract

More people are now using on-site sanitation technologies than sewer connections. Human excreta management remains challenging and requires an understanding of all sanitation service chain components and their interactions: from source conditions (i.e. toilets) to treatment and resource recovery. This research used pyrolysis to recover resources from human excreta, focusing on the effect of two key source interventions (1. urine diversion; 2. biomass addition) aiming to improve the circularity of sanitation systems.

Firstly, the effects of source separation of human excreta on resource recovery were investigated for different pyrolysis temperatures. Source separation maximised nitrogen recovery (70% of N losses avoided) and improved biochar properties relevant to agricultural applications. It also improved the quality of produced solid fuels, including a 50% increase in the biochar's calorific value.

Subsequently, faecal biochars were enriched with nutrients from fully hydrolysed human urine via adsorption and precipitation, creating a combined fertiliser and soil amendment. The main $\text{NH}_4\text{-N}$ adsorption mechanism was ion exchange ($\text{K}^+ \leftrightarrow \text{NH}_4^+$), while precipitation was the primary mechanism for P recovery (>98% P recovered with MgO addition). The combination of MgO (Mg:P=1.5) with the lower biochar dose tested (25 g/L) yielded the most NP-rich fertiliser.

Carbon sequestration potential was investigated for the co-pyrolysis of human faeces with wood biomass. The systematic assessment of biochar stability and the quantification of carbon flows among pyrolysis products were carried out for the first time in the context of human excreta. Synergistic effects took place during co-pyrolysis, and 50% wood content (~30g/toilet user/day) was sufficient to enhance carbon stability and storage.

The laboratory-based findings were applied to assess the resource recovery potential of an operating faecal sludge treatment plant located in Kampala, Uganda. Overall, this research established quantifiable links between sanitation source conditions and resource recovery objectives, contributing towards the design of circular sanitation systems.

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“..to strive, to seek, to find, and not to yield” – Lord. A. Tennyson

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Abbreviations

AD	Anaerobic digestion
ANOVA	Analysis of variance
BET	Brunauer, Emmett, and Teller
BSF	Black soldier fly
CaCO ₃ -eq.	Calcium carbonate equivalence
CBS	Container-based sanitation
CEC	Cation exchange capacity
CHNS(O)	Carbon, hydrogen, nitrogen, sulphur (and oxygen)
CV	Calorific value
DOC	Dissolved organic carbon (water-extractable)
DTG	Derivative thermogravimetry
EBC	European Biochar Certificate
EC	Electrical conductivity
EDX	Energy-dispersive X-ray spectroscopy
FAO	Food and Agriculture Organization
FC	Fixed carbon
FS	Faecal sludge
FSM	Faecal sludge management
FSTP	Faecal sludge treatment plant
FTIR	Fourier-transform infrared spectroscopy
GHG	Greenhouse gas
HAP	Hydroxyapatite
HF	Human faeces
HHT	Highest heating temperature
HHV	Higher heating value
HTC	Hydrothermal carbonisation
IBI	International Biochar Initiative
ICP-OES	Inductively coupled plasma optical emission spectrometry
IQR	Interquartile range
LCA	Life cycle assessment
LDCs	Least developed countries
LMICs	Low- and middle-income countries
MC	Moisture content
MUF	Mixed urine and faeces
NCG	Non-condensable gases
NH ₄ -N	Ammoniacal nitrogen (referring to both NH ₄ ⁺ and NH ₃ forms) expressed as N
P (2% FA)	2% formic acid extractable phosphorus
RTP	Room temperature and pressure
SDG	Sustainable Development Goal
SEM	Scanning electron microscopy
SSF	Source-separated faeces
SSU	Source-separated urine
STA	Simultaneous thermal analyser
TGA	Thermogravimetric analysis
UD	Urine diversion
UDDT	Urine-diverting dry toilet
UN	United Nations
VIP	Ventilated improved pit
VM	Volatile matter
WASH	Water, sanitation and hygiene
WB	Wood biomass
WBC	World Biochar Certificate
WHO	World Health Organization
XRD	X-ray diffraction
XRF	X-ray fluorescence

1 Introduction

1.1 Background and terminology

Currently, more people globally are using on-site sanitation technologies than sewer connections linked to wastewater treatment services. Approximately 3.7 billion people rely on on-site technologies to access sanitation services; including pit latrines, septic tanks, composting toilets and container-based sanitation (World Bank, 2019; WHO/UNICEF, 2023). The World Health Organization (WHO) and the United Nations Children’s Fund (UNICEF) define on-site sanitation facilities as those that “*collect, store and, to some extent, treat excreta in storage tanks or pits*”. They add that “*these may be lined or unlined, permeable or impermeable, and may discharge liquid effluent to on-site treatment (for example, a leach field), a sewer line or into the surface environment*” (WHO/UNICEF, 2023).

Examples of on-site sanitation toilets are shown in **Figure 1.1**. The material that accumulates in such facilities is known as faecal sludge (FS) and consists of human excreta (faeces and urine) with or without the addition of flush water, toilet paper, greywater or other (waste) materials that may be discarded in toilets (Velkushanova et al., 2021). Uncontrolled discharge of human excreta and FS can have detrimental environmental and public health impacts, making access to sanitation and safe FS management (FSM) essential parts of healthy and sustainable communities (Strande et al., 2014).



Figure 1.1: (a) On-site sanitation toilet block at a youth centre in Jinja, Uganda [photograph taken April 2024]; and (b) private on-site toilet at a household in Crete, Greece [photograph taken August 2024].

*All photographs in this thesis are taken by the author, unless otherwise stated.

An overview of the key components of a “sanitation system”, including the containment and FSM service chains, is shown in **Figure 1.2**. These components include the sanitation user interface (i.e. toilet), on-site storage facility (e.g. pit latrine, septic tank), emptying and collection services, FS treatment (as well as transport to and from the treatment facility) and end-use or disposal of the final products. For a sanitation system to be safe, it is essential that all components are present and well-functioning (Tilley et al., 2014).

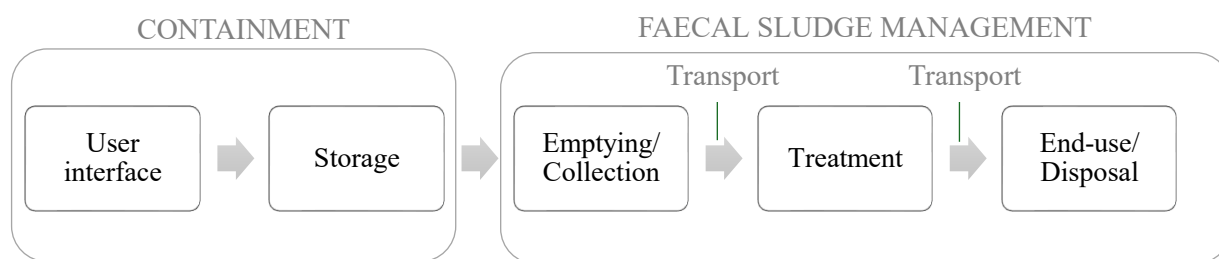


Figure 1.2: Key components of a “sanitation system”, including the faecal sludge management chain, adapted and modified from Strande et al. (2014); WHO/UNICEF (2019).

Terminology is also important to ensure common understanding among researchers, practitioners, decision-makers and other stakeholders. The definition of various sanitation service levels, as provided by the WHO/UNICEF Joint Monitoring Programme (JMP) for data on Water, Sanitation and Hygiene (WASH) is shown in **Table 1.1**. As of 2023, 3.4 billion people do not have access to “*safely managed sanitation*” (>40% of the global population) and 1.5 billion people, mostly living in low- and middle-income countries (LMICs), still do not have access to even “*basic*” sanitation services (WHO/UNICEF, 2023). Of these, 1 billion use “*unimproved*” services that do not separate excreta from human contact or practice open defecation (WHO/UNICEF, 2023). The United Nations (UN) Sustainable Development Goal (SDG) 6 on “clean water and sanitation” highlights the need to work towards both increasing sanitation coverage (i.e. containment phase in **Figure 1.2**) and adopting sustainable management practices for the FS that is being contained and collected (i.e. FSM phase in **Figure 1.2**).

Table 1.1: Sanitation service levels defined by the WHO/UNICEF Joint Monitoring Programme (JMP) for WASH data, their definition and estimates of the number of people using sanitation that meets the criteria of each level, based on data from WHO and UNICEF (2023).

Service level	Definition	No. of people (WHO/UNICEF, 2023)
Safely managed	“Use of improved* facilities that are not shared with other households and where excreta are safely disposed of in situ or removed and treated off-site”	4.6 billion
Basic	“Use of improved* facilities that are not shared with other households”	1.9 billion
Limited	“Use of improved* facilities that are shared with other households”	570 million
Unimproved	“Use of pit latrines without a slab or platform, hanging latrines or bucket latrines”	545 million
Open defecation	“Disposal of human faeces in fields, forests, bushes, open bodies of water, beaches or other open places, or with solid waste”	419 million

*The defined service levels and presented statistics refer to both sewered and on-site sanitation facilities. For on-site sanitation, the JMP classification of “improved on-site sanitation facilities” includes: “a) flush and pour-flush toilets or latrines connected to septic tanks or pits; b) ventilated improved pit (VIP) latrines; c) pit latrines with slabs; and d) composting toilets, including twin pit latrines with slabs and container-based sanitation (CBS) systems”. These different types of facilities are described in detail in **Section 3.1.1**.

At the same time, many researchers have highlighted that human excreta and FS are not just waste to be safely discharged, but also sources of valuable resources for recovery (including nutrients, energy, carbon and water) (Guest et al., 2009; Larsen et al., 2009; Pradel et al., 2016). This change in perspective, and the valorisation of excreta-derived products, can act as incentives for better FSM practices and increased sanitation coverage (Diener et al., 2014). The new paradigm is further supported by the interrelated nature of the SDGs, highlighting the need for an integrated approach to FSM in order to reach a zero-pollution future and achieve multiple resource management targets (see **Section 1.2**) (Clark et al., 2018).

1.2 Sanitation and global resource management

1.2.1 The UN Sustainable Development Goals

The UN SDG 6 calls for universal access to sanitation services by 2030, as well as adopting sustainable management practices for the FS that is being contained and collected, including capacity-building for recycling and recovery of resources (UN, 2020). To this date, no SDG region¹ is on track to achieve SDG 6 and the current rate of progress will require a fivefold increase to achieve universal access to safely managed sanitation (UN, 2023).

Inequalities are maximised for least developed countries (LDCs), isolated areas and fragile contexts, where a ≥ 15 -fold increase in the rate of progress is required to meet targets (UN, 2021). This is driving a rapid increase in on-site sanitation services globally, due to their low cost, robustness and ability to be operated in areas with limited water resources. Strategies towards achieving SDG 6 in these areas need to include compact, novel technologies that are tailored to local needs, user preferences and technological capabilities, whilst also encouraging further capacity-building (Mara & Evans, 2018). Although more people globally are using on-site sanitation than sewer connections, the majority of *safely managed sanitation* services is still attributed to sewer connections, highlighting the need for improved FSM practices (WHO/UNICEF, 2023). Current knowledge suggests that safe FSM solutions are successfully implemented if they can create multiple benefits for the community, including boosting the financial sector, creating job opportunities and recovering resources (Trimmer et al., 2020b; Bagire et al., 2021).

In the context of resource recovery, sustainable sanitation systems can support progress towards multiple SDGs, by recycling water, carbon, energy and nutrients. Several studies have explored the connection between meeting SDG 6 targets and other goals, including food security (SDG 2), energy security (SDG 7), climate action (SDG 13), public health (SDG 3), equality (SDG 5, 10), access to

¹ The SDG regions defined by the UN are: 1) Sub-Saharan Africa; 2) Northern Africa and Western Asia; 3) Central and Southern Asia; 4) Eastern and South-Eastern Asia; 5) Latin America and the Caribbean; 6) Australia and New Zealand; 7) Oceania (excluding Australia and New Zealand); and 8) Europe and Northern America (UN, 2020).

education (SDG 4) and economic growth (SDG 8) (Trimmer et al., 2017; Andersson et al., 2017; Orner & Mihelcic, 2017; WaterAid, 2019). Key resource recovery priorities, and their interactions with human waste management targets, are described below.

1.2.2 Nutrient scarcity

Nutrient retention is a crucial challenge of resource circularity and the connection between SDG 6 and SDG 2 (“Zero Hunger”) has been established by several studies (Trimmer et al., 2017; Orner & Mihelcic, 2017). Particularly the issue of declining phosphorus (P) availability has generated interest in P recovery methods over the past decades, as P is a finite, non-substitutable resource that is essential to life and plant growth and, ultimately, food security (Cordell et al., 2009). Estimates on when P reserves will be exhausted vary, however, the increasing technical difficulty of extraction and the geo-political issues arising from global reserves being limited to a few countries are recognised challenges for global food security (Nedelciu et al., 2020). As phosphate prices fluctuate and uncertainty over availability of reserves increases, the need to recycle this valuable resource is an urgent global sustainability objective (Daneshgar et al., 2018). Nitrogen (N) is also an essential macronutrient, and its synthetic production via the Haber-Bosch process (as ammonia) is very energy-intensive and costly (Canfield et al., 2010). The production of N is said to contribute to up to 90% of the total energy required for fertiliser manufacturing globally (Mudahar & Hignett, 1985).

Still, the wastewater treatment industry (where around 17 Tg of N and 3 Tg P end up annually) generally treats nutrients merely as pollutants to be removed rather than valuable resources to be recovered (Qadir et al., 2020; Preisner et al., 2020). These pollution control measures are essential to prevent eutrophication and maintain water quality, however, they should be considered alongside global challenges of soil depletion and decreased fertility (Pausta et al., 2024). The valorisation of excreta-derived nutrients for agricultural production is becoming increasingly urgent as the global demand for food increases in line with a growing population. It has been estimated that approximately 13% of the global agricultural nutrient demand could be recovered from the human excreta management sector (Qadir et al., 2020).

The production of excreta-derived fertilisers is therefore an attractive opportunity for combined nutrient recovery and eutrophication prevention. The Food and Agriculture Organization (FAO) estimates that current agricultural production rates will need to be increased by approximately 60% to match the expected food demand (Alexandratos & Bruinsma, 2012). While this figure corresponds to achievable rates of increase for developed countries with stable or declining populations (e.g. China, European countries, etc.), it requires a significant increase for developing countries with high population growth and increasing food demand. This inequality needs to be addressed by focusing global efforts to the developing world and efficiently utilising widely available, economical and sustainable nutrient

sources, such as human excreta (Alexandratos & Bruinsma, 2012). Photographs of agricultural applications using excreta-derived fertilisers or fertigation techniques in sub-Saharan African countries are shown in **Figure 1.3**.



Figure 1.3: (a) Fields in Uganda being fertigated with treated wastewater [photograph taken April 2024]; and (b) maize production in rural Rwanda after fertilisation with treated faecal sludge [photograph taken March 2023].

In parallel, the need for safely managed sanitation services will only be heightened due to the expected population increase in LDCs and LMICs, leading to large quantities of FS to be managed (Mara & Evans, 2018). These parallel challenges create an opportunity for sanitation-driven nutrient recovery, particularly for outputs of on-site dry toilets that are more nutrient-dense compared to other wastewater sources (Larsen et al., 2013). While sewerage sanitation infrastructure usually involves the mixing of human excreta with large amounts of flush water and other wastewater streams, decentralised toilets provide opportunities to recover valuable nutrients from pure – and often source-separated – human excreta (Martin et al., 2020).

1.2.3 Energy demand

The rising urban population also creates a rapidly increasing demand for reliable and affordable energy. For LDCs and LMICs there is still a strong reliance on solid fuels for heating and cooking and it has been estimated that 80% of the population in East African countries relies on wood-derived charcoal (Haysom et al., 2021). Wood-burning stoves are also a commonly used cooking method, with significant impact on air quality and health, especially for indoor kitchen stoves (**Figure 1.4**). Previous research has reported on the injustices of the current wood and charcoal supply chain, both environmental and social, and highlighted the need to diversify the portfolio of solid fuel sources (Fløytrup et al., 2022). Demand is rising for alternative waste-derived briquettes produced either from raw/dried biomass or from charred materials, including human excreta and FS (Otieno et al., 2022) (**Figure 1.5**).

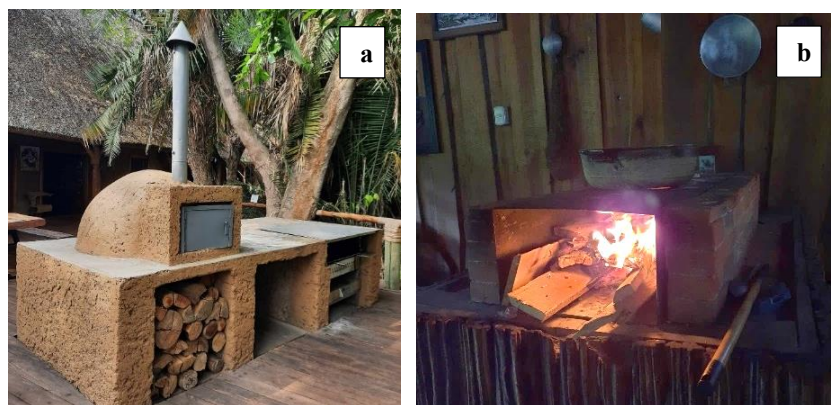


Figure 1.4: (a) Outdoor wood-burning cooking stove in Rwanda [photograph taken March 2023]; and (b) indoor wood-burning cooking stove in Costa Rica [photograph taken April 2021].

The advantages reported for uncharred fuels (**Figure 1.5a**) are mostly related to their lower ash content and quick ignition time (Andriessen et al., 2019; Kizito et al., 2022). Nevertheless, the high volatility of raw biomass and, in the case of human excreta, considerable N and S content, are substantial disadvantages leading to increased emissions during use, including CO₂, CO, NO_x and SO_x gases (Obernberger et al., 2006; Lohri et al., 2016). Therefore, uncharred biomass waste can only be considered a sustainable fuel source when used for industrial furnaces that include suitable emissions controls and do not cause issues with odour or indoor air quality deterioration in households (Diener et al., 2014; Gold et al., 2017).

Briquettes produced from charred biomass (and/or FS) (**Figure 1.5b**) are more suitable for household use due to their low emissions, slow burning properties, abundant availability of materials and affordability compared to normally used charcoal briquettes (Fløytrup et al., 2022). Environmental benefits of using waste feedstock materials rather than wood-based charcoal are even more significant when considered under the prism of global reforestation and climate change mitigation efforts. Therefore, the parallel challenges to achieve universal access to sanitation services and affordable clean energy, create an opportunity to combine waste management and energy provision objectives.



Figure 1.5: (a) Sample of a briquette produced from raw sawdust; and (b) sample of a briquette produced from carbonised sawdust mixed with faecal sludge in Nyamagabe, Rwanda [photographs taken March 2023].

1.2.4 Carbon management

Carbon management is a critical frontier for sustainable development, as climate change has been a global driver of scientific, environmental and socio-political developments in recent decades. Related global challenges due to increasing levels of anthropogenic greenhouse gas (GHG) emissions (mainly CO₂, CH₄, N₂O and fluorinated gases) include atmospheric and ocean warming, extreme weather events (including heatwaves and droughts, as well as heavy rain and flash floods), ice melting and sea level rise (Redlin & Gries, 2021).

Strategies to manage climate change are broadly categorised as mitigation and adaptation measures. There are several global targets, regulatory guidelines, financial incentives and implications that aim to mitigate climate change impacts. The Paris Agreement, adopted in 2015 under the United Nations Framework Convention on Climate Change (UNFCCC), sets the goal of limiting global warming to <2 °C above pre-industrial levels, with efforts to limit it to the long-term warming limit of 1.5 °C (UNFCCC, 2015). The SDG 13 on “climate action” calls to build capacity both for mitigation and adaptation of climate change impacts, through funding commitments, educational and institutional capacity, global and national planning (including for less developed countries) and maximising resilience and adaptation measures (UN, 2020). Financial incentives and implications are derived both from the regulatory framework, including carbon allowances and taxation for carbon pollution, as well as market driven financial instruments such as green investments, carbon offsets and credits (Shi et al., 2022; Ott & Endrikat, 2023).

The Intergovernmental Panel on Climate Change (IPCC) has highlighted the need to achieve net-zero carbon emissions; meaning to balance carbon emissions with activities that can store carbon long-term, effectively removing it from the atmospheric cycle (IPCC, 2023). In the context of sanitation, the management of human excreta and other biomass waste can create opportunities for climate change mitigation, both via avoided carbon emissions and carbon storage, during containment and treatment of FS (Rowles et al., 2022). Although there are currently very limited studies on the quantification and systematic assessment of carbon flows along the sanitation chain (Manga et al., 2022), a new paradigm is starting to be established whereby on-site sanitation is being recognised as an important sector for carbon management, estimated to contribute to up to 6% of global CH₄ emissions (McNicol et al., 2020). Diverting FS from long-term containment in anaerobic conditions is therefore an important mechanism for avoided GHG emissions (Reid et al., 2014).

Further benefits can be expected when resources are recovered from human excreta and FS, including recovered nutrients and energy, due to avoided emissions from alternative practices such as mineral fertiliser manufacturing or unsustainable energy production technologies. Direct carbon storage benefits are also a viable option when treating human excreta via pyrolysis, through the production of biochar as a stable form of aromatic carbon that can remain stored in the soil for hundreds to thousands of years

(Mandlebaum & Nriagu, 2011). When human excreta are mixed with other carbonaceous waste (e.g. cover materials added in dry toilets) further carbon benefits can be investigated. The carbon flows during the treatment of FS need to be researched, particularly for treatment technologies like pyrolysis where carbon is distributed among various products (solid char, oil and gas – see **Section 3.2.1**).

Overall, carbon is a critical resource that needs to be strategically managed to reduce GHG emissions and facilitate climate change mitigation (McLaughlin et al., 2023). Human waste is a small but not insignificant part of anthropogenic emissions that can contribute to global carbon management efforts (McNicol et al., 2020). Cumulative benefits when FSM is considered within the wider framework of biomass waste management are perhaps even more important. From a sanitation perspective, carbon benefits can provide meaningful incentives, environmental and/or financial, to establish and maintain safe containment, collection and treatment services for on-site sanitation users (CBSA, 2023).

1.2.5 Access to clean water

Water scarcity and declining water quality are critical global challenges with implications for human health, economic development and environmental sustainability (Liu et al., 2017). In 2020, it was estimated that 2.4 billion people lived in “water-stressed” countries; a term based on the ratio of freshwater withdrawals to renewable freshwater resources available (UN, 2023). At the same time, the quality of available water resources is deteriorating as a result of water pollution from industrial and domestic waste streams, including due to inadequate sanitation (Schwarzenbach et al., 2010).

It is therefore evident that water access and sanitation objectives are naturally interrelated, with FSM playing a pivotal role in this relationship. This is highlighted by the structure of SDG 6 which combines targets for access to both services (UN, 2020). To this date, approximately half the population of sub-Saharan Africa still relies on water collection (see **Figure 1.6**) (WHO/UNICEF, 2023). Notably, the responsibility of fetching water is often disproportionately placed on women and young girls, meaning that links with SDGs on gender equality and other social challenges (poverty, education, etc.) are also clearly established (Crawford, 2020).



Figure 1.6: (a) Young adults and children collecting clean water from affordable communal taps in rural Rwanda [photograph taken May 2024] and (b) people washing clothes in a river, a few meters upstream from a water collection point in Jinja, Uganda [photograph taken April 2024].

Climate change effects (e.g. extreme weather events) often exacerbate the implications of poor sanitation on water quality (Hyde-Smith et al., 2022). For example, during flood events surface runoff can overwhelm sanitation systems and lead to overflow of untreated FS, increasing the risk of waterborne diseases. Moreover, the inundation of sanitation facilities can render them unusable, leaving local communities without access to proper sanitation often for extended periods of time (Howard et al., 2016). Therefore, ensuring that water and sanitation systems are robust, and that effective FSM technologies are in place, will be essential to protect public health and environmental sustainability while adapting to intensifying climate change effects (Dickin et al., 2020). Dry on-site sanitation toilets have the benefit of not relying on water supply to function, as well as minimising the usage of treated water in households (Aburto-Medina et al., 2020). Resource recovery from human excreta streams can further alleviate pressure from conventional resource management practices (including nutrients, energy and carbon - see **Sections 1.2.2-1.2.4**) that are often water-intensive (Trimmer et al., 2019).

These links within the water resources cycle, while obvious, are often overlooked when it comes to WASH interventions in areas with parallel water and sanitation challenges (Pahl-Wostl et al., 2013). It is therefore imperative that water resources systems are managed holistically, and that the interactions between their components are understood and addressed (Phan et al., 2021). Efforts to maximise circularity within sanitation and FSM systems will inevitably benefit wider water resource management objectives, both in terms of water quality and water quantity (Silva, 2023).

1.3 Resource recovery via pyrolysis of human excreta

One option for the treatment of human excreta and FS is pyrolysis, the thermochemical conversion of carbonaceous materials in an oxygen-limited environment (Lehmann & Joseph, 2015). Pyrolysis has been established in the treatment of many organic feedstocks, including wood biomass, agricultural waste and sewage sludge (Méndez et al., 2013; Ippolito et al., 2020). Although it has not been widely applied for human excreta/FS treatment, interest is rapidly increasing, as pyrolysis ensures pathogen sterilisation and offers several resource recovery opportunities (Krueger et al., 2020).

The end-products of the process, which include a solid output known as biochar, bio-oil and gas (syngas), have various reuse possibilities (Somorin et al., 2020b; Das & Ghosh, 2021). Recent studies on FS pyrolysis have focused on the investigation of biochar properties, as it can be used for a wide range of applications, including agricultural uses, fuel production, adsorption processes, construction materials and carbon sequestration (**Figure 1.7**) (Gold et al., 2018a; Bleuler et al., 2020). Therefore, pyrolysis is a particularly fitting technology for research on resource recovery, as it allows the direct comparison of alternative recovery routes depending on specific feedstock characteristics, treatment temperatures and operating conditions.

This research focuses on pyrolysis as a treatment technology for human excreta – either from mixed or source-separating toilets (i.e. separating the urine and faeces fractions) – and the associated resource recovery opportunities, particularly in terms of nutrient recovery, solid fuel production and carbon sequestration (**Figure 1.7**).

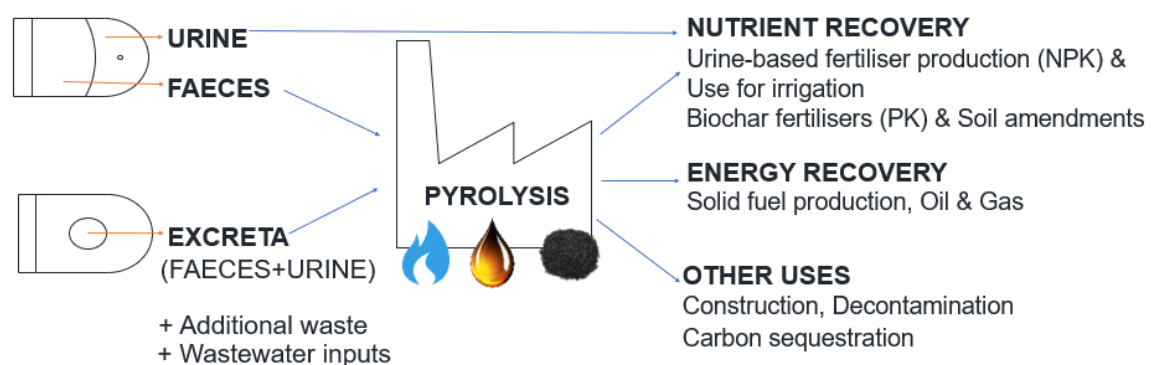


Figure 1.7: Outline of human excreta pyrolysis options and available routes for resource recovery. Pyrolysis products include gas, bio-oil and biochar.

A note on terminology

The term “biochar” is used in this thesis as defined by the International Biochar Initiative (IBI): “a solid material obtained from thermochemical conversion of biomass in an oxygen limited environment” (IBI, 2015). According to the IBI (2015), for a material to be classified as “biochar”, it needs to be produced from a feedstock that consists of biomass and diluents (i.e. inorganic constituents) and includes no more than 2% dry weight contaminants (e.g. fossil fuel-derived compounds, glass, metal objects). Other indicators used for a material to be classified as “biochar” are the H/C (organic) ratio, as an indicator of carbon stability, as well as toxicity indicators to ensure the safety of the final product (IBI, 2015).

Lehmann and Joseph (2015) suggested that the term “biochar” is used to distinguish pyrolysis products intended for environmental management from other materials generated by incomplete combustion processes. Nevertheless, some publications on FS pyrolysis refer to the solid output of pyrolysis as “char” rather than “biochar”. In this thesis the term “biochar” will be used for biochar prepared from human excreta as it can safely be assumed that no more than 2% contaminants are present, based on the IBI (2015) definition.

In **Chapter 8**, however, the term “char” is used for FS feedstocks from unknown sources, as this assumption cannot be safely made from available information on feedstock origin. Moreover, the term “carbonisation” is used in this chapter instead of “pyrolysis”, to highlight the very slow pyrolysis heating rate, aiming to produce solid char rather than oil and gas products (see **Section 3.2.1**).

1.4 Thesis outline

This chapter is followed by an outline of research aims and objectives in **Chapter 2**, while **Chapter 3** presents a detailed review of the existing literature and highlights the identified knowledge gaps. The fundamental methods used for this research are described in **Chapter 4** and supplemented with specific methods included in individual chapters. **Chapter 5** investigates the effects of source separation of faeces and urine on biochar characteristics for the context of resource recovery (including nutrient recovery and solid fuel production). This knowledge is further developed in **Chapter 6**, which presents a comprehensive method to recover additional nutrients (N and P) from separated urine, via adsorption and precipitation onto faecal-derived biochars. This research suggests a circular approach to treating human waste that paves the way towards the production of marketable fertilisers and soil amendments from sanitation waste. **Chapter 7** presents the study of carbon flows and carbon storage potential when co-pyrolysing human faeces with wood biomass cover materials which are often added in sanitation systems. **Chapter 8** involves the assessment of a case study of an operating FS treatment plant in Kampala, Uganda, where the knowledge presented in previous chapters is discussed under the context of a real-world scenario. The final **Chapters 9-11** include the discussion of findings, make recommendations for further research and summarise the conclusions and knowledge contributions.

1.5 List of publications

The author's publications during this PhD are listed below. The findings presented in **Chapters 5, 6 and 7** were included in Koulouri et al. (2023), Koulouri et al. (2024a) and Koulouri et al. (2024b), respectively. A list of the author's presentations at scientific conferences during this PhD is provided in **Appendix A**.

- **Koulouri, M.E.**, Templeton, M.R., Fowler, G.D., 2023. Source separation of human excreta: Effect on resource recovery via pyrolysis. *J. Environ. Manage.* 338, 117782. <https://doi.org/10.1016/j.jenvman.2023.117782>
- **Koulouri, M.E.**, Templeton, M.R., Fowler, G.D., 2024a. Enhancing the nitrogen and phosphorus content of faecal-derived biochar via adsorption and precipitation from human urine. *J. Environ. Manage.* 352, 119981. <https://doi.org/10.1016/j.jenvman.2023.119981>
- **Koulouri, M.E.**, Qiu M., Templeton, M.R., Fowler, G.D., 2024b. Carbon flows and biochar stability during co-pyrolysis of human faeces with wood biomass. *Environ. Sci.: Water Res. Technol.* 10, 2709 – 2722. <https://doi.org/10.1039/D4EW00513A>

2 Research aims and objectives

This research aims to:

optimise resource recovery via pyrolysis of human excreta and faecal sludge

by

exploring the effect of strategic interventions – namely source separation of human excreta and biomass addition in sanitation systems – on biochar properties relevant to resource recovery.

To this end, the specific objectives of this thesis are to a) establish the relationship between source separation of human excreta and resource recovery via pyrolysis; b) investigate a circular method to recover nutrients from stored human urine using faecal-derived biochars; c) assess the carbon sequestration potential when co-pyrolysing human faeces with wood biomass (e.g. when wood is added as a cover material); and d) evaluate an operating FS carbonisation² plant where FS is co-carbonised with biomass waste to produce char fertilisers and solid fuel briquettes. The steps followed to achieve each specific objective are described below:

Objective 1: Establish the relationship between source separation of human excreta (faeces and urine) and resource recovery via pyrolysis.

- Compare the compositional characteristics of the two excreta components and identify their influence upon the thermal decomposition behaviour during pyrolysis, when maintaining all other parameters constant.
- Assess the effect of two feedstock scenarios (with and without source separation) and three pyrolysis temperatures (450, 550, 650 °C) on biochar quality, via the comprehensive characterisation of biochar characteristics.
- Investigate the influence of source separation on potential end-uses of biochars, including agricultural uses and solid fuel production.

² The term “carbonisation” is used here instead of “pyrolysis”, to highlight the very slow pyrolysis heating rate used on-site, aiming to produce solid char rather than oil and gas products (refer to **Section 3.2.1** and **Chapter 8**).

Objective 2: Investigate a circular method to recover nutrients from stored human urine using faecal-derived biochars (via adsorption and precipitation).

- Investigate the evolution of the composition and nutrient content of separated human urine during on-site storage (for up to 6 months).
- Study the mechanisms of $\text{NH}_4\text{-N}$ adsorption from urine onto faecal-derived biochars and model the adsorption isotherms and kinetics.
- Assess the suitability of faecal-derived biochar as a carrier for simultaneous N and P recovery, via batch adsorption and precipitation experiments.

Objective 3: Assess the carbon sequestration potential when co-pyrolysing source-separated faeces with wood biomass.

- Identify potential synergistic effects during co-pyrolysis of faeces and wood biomass, in terms of carbon retention and biochar stability.
- Investigate the effect of various faeces-to-wood mixing ratios (100:0, 75:25, 50:50, 25:75, 0:100) and pyrolysis temperatures (450, 550, 650 °C) on biochar carbon properties.
- Determine the distribution of carbon flows among pyrolysis products (biochar, bio-oil, non-condensable gases).

Objective 4: Evaluate an operating faecal sludge (FS) treatment plant where FS is co-carbonised with biomass waste to produce char fertilisers and solid fuel briquettes.

- Determine the key properties of FS-derived fertilisers and solid fuels, and how these vary statistically.
- Investigate the influence of feedstock composition and pyrolysis operational conditions on end-product quality.
- Assess how the investigated source interventions can improve the resource recovery potential of at-scale FS carbonisation applications.

3 Literature review

3.1 Human excreta and faecal sludge as feedstocks for resource recovery

On-site sanitation facilities produce large quantities of FS that need to be safely contained and treated to avoid environmental pollution and protect public health (Strande et al., 2014), while also being a potential source of valuable resources to be recovered (Guest et al., 2009; Pradel et al., 2016). It is therefore essential to develop FSM solutions that consider all sanitation service chain components and their interactions, from toilet source conditions to treatment technologies and resource recovery. The following sections describe the different sources of FS, highlight key parameters that influence FS characteristics, and summarise the properties of human excreta and FS that are most relevant to resource recovery research.

3.1.1 Sources of faecal sludge

So far, significant progress has been made towards achieving a better understanding of FS composition and its distribution among different toilet types and containment systems (Krueger et al., 2021a), as well as establishing standardised methods of FS sampling and analysis (Velkushanova et al., 2021). Here, relevant literature is reviewed to identify the main types of on-site sanitation facilities and types of FS commonly encountered. For this exercise, terminology and different definitions that may be used across publications are particularly important, to ensure literature knowledge is interpreted consistently. The term “faecal sludge” is very generic and corresponds to anything from liquid wastewater collected from non-sewered sanitation (e.g. outputs of flushed on-site toilets or septic tanks) to thick sludge from dry pit latrines and human excreta sludges from container-based sanitation systems (Strande et al., 2023). It is therefore incorrect to assume that sludges originating from different types of sanitation facilities have similar properties and treatment requirements. The most common sources of FS are described below.

3.1.1.1 Traditional on-site sanitation technologies

One of the most commonly used on-site sanitation technologies is the pit latrine, i.e. a simple pit in the ground used to collect and store human excreta (Tilley et al., 2014). Pit latrines can be broadly categorised as “wet” and “dry” depending on whether water is used for flushing (e.g. pour-flush latrines) (Mamera et al., 2021). Another broad categorisation can be defined depending on whether the pit latrine has a slab to ensure the safe separation of excreta from human contact. It is important to note that pit latrines without slabs are defined as “unimproved” sanitation facilities that will ultimately need to be altered or replaced to achieve SDG 6 (WHO/UNICEF, 2023). Similarly, pit latrines may be lined, unlined or partially lined, meaning that they can either be fully contained or (partly) drain into the

surrounding soil (Buckley et al., 2008; Nabateesa et al., 2017). Therefore, not all pit latrines provide the same service level of “safely managed” sanitation. Depending on construction details and emptying practices, the separation of untreated human excreta from the surrounding environment may not be achieved, especially in areas with a high groundwater table (Graham & Polizzotto, 2013).

Apart from these broad categories, there are several different types of pit latrines which were developed as improvements to the “simple pit latrine”. The ventilated improved pit (VIP) latrine is a well-established design that includes a vent pipe with a fly screen, aiming to minimise odour and insect nuisance for on-site sanitation users (see **Figure 3.1a**) (Obeng et al., 2023). Another design modification is the twin (or double) pit latrines (i.e. two latrines used in alternation), which allow the on-site decomposition of FS during longer retention times, aiming to extend the periods between collections and improve safety for the emptier as the FS will be (partially) treated (Hussain et al., 2017). Significant variation can be expected between FS produced from single versus double latrines, due to the different level of on-site decomposition that has an effect on its organic composition and pathogen content (Dey et al., 2016). For example, hemicellulose and cellulose degrade quicker than lignin, meaning that the lignin content in FS can be expected to increase with sludge age (Krueger et al., 2021b). Twin latrines may also emit more GHGs due to FS decomposition (both anaerobic and aerobic) during extended retention times (Cheng et al., 2022).

Another commonly used on-site sanitation facility is the urine-diverting dry toilet (UDDT). UDDTs collect faeces and urine in separate compartments and operate without flush water (see **Figure 3.1b**) (Tilley et al., 2014). The diversion of urine has several benefits including reduced odour, lower moisture content and slower sludge accumulation rate (Mkhize et al., 2017). In terms of resource recovery, urine diversion has often been reported as being beneficial for nutrient recovery, as most of the nutrients excreted from the human body are concentrated in urine (Larsen, 2020; Chipako & Randall, 2020b). In theory, the outputs of UDDTs are separated faeces and urine, while usually a cover material is also added to the faecal stream to aid on-site decomposition and promote drying (Getahun et al., 2020). Nevertheless, the source separation achieved in UDDTs is highly dependent on user behaviour, as often people may not be sufficiently trained or motivated to use UDDTs properly (Hennigs et al., 2021).

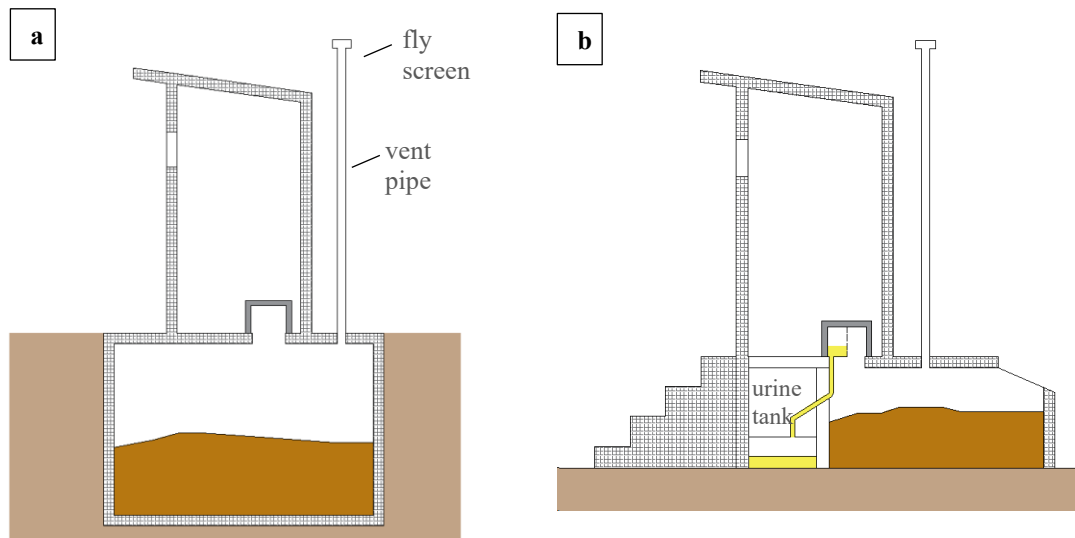


Figure 3.1: Ventilated improved pit (VIP) latrine (a) and urine-diverting dry toilet (UDDT) (b) schematics, adapted and modified from Tilley et al. (2014).

In terms of wet on-site sanitation technologies, the most common facilities are septic tanks. These are underground watertight chambers made of concrete, plastic, fiberglass or other materials, where settling and anaerobic processes occur as primary treatment (see **Figure 3.2**) (Tilley et al., 2014). In contrast to pit latrines and UDDTs, septic tanks can also contain greywater and blackwater sources, and therefore their outputs are often more similar to the consistency of “wastewater”, rather than the usual perception of the term “faecal sludge” (WHO/UNICEF, 2023; Strande et al., 2023). Although “faecal sludge” is often used as an umbrella term referring to any on-site sanitation output, its consistency greatly varies depending on the containment facility and type of inputs. Even the term “septic tank” can refer to a variety of infrastructure, from unsafe permeable cess pits to engineered tanks that are safely contained and managed (Strande et al., 2023). Therefore, on-site sanitation technologies need to be assessed on a case-specific basis.

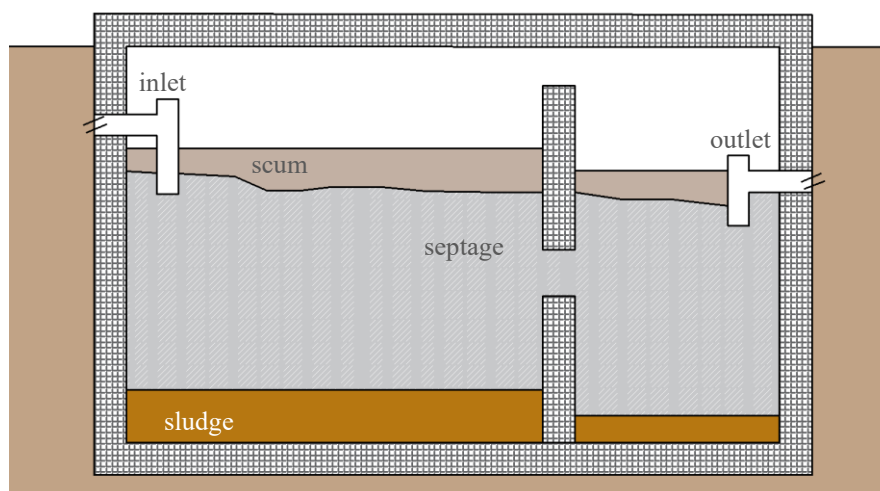


Figure 3.2: Septic tank schematic, adapted and modified from Tilley et al. (2014).

3.1.1.2 Container-based sanitation and human excreta

Container-based sanitation (CBS) systems have been gaining popularity in recent years as an alternative safely managed sanitation solution for low-income settings (World Bank, 2019; Dewhurst et al., 2019). Their business model typically includes collection and transport services, as well as treatment and resource recovery processes, and therefore encourages circularity and safe management of excreta. CBS systems are service-based and offer several benefits compared to conventional on-site sanitation systems, including regular collection at a household level (usually weekly) and urine separation (Velkushanova et al., 2021). CBS toilets are fully enclosed, which means that no sand or grit can enter the toilet and are usually waterless (see **Figure 3.3**) (Russel & Montgomery, 2020).

Apart from human excreta, other organic material, such as sawdust, coconut coir or rice husk, are usually added by users as a cover material (World Bank, 2019). Unlike other on-site sanitation facilities, additional influencing parameters (e.g. whether a pit latrine is lined, how often it is emptied, additional liquid or solid waste being dumped into the toilet, etc.) are minimised due to the household-level management of CBS toilets. Therefore, the theoretical outputs of UDDTs mentioned in **Section 3.1.1.1** (i.e. faeces mixed with biomass cover materials and separated urine) are in fact more realistic for CBS toilets, due to their service-based approach that creates incentives for correct use and ensures no additional waste materials enter the containment chamber. Based on these characteristics, the outputs of CBS toilets can realistically be replicated by pure human excreta (mixed or separated, and with or without the addition of biomass cover materials).

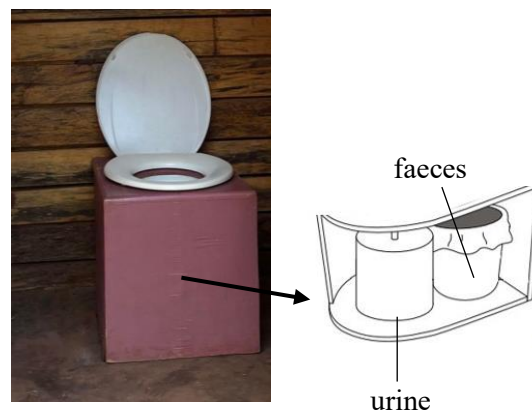


Figure 3.3: Photograph of a container-based toilet [taken in Jinja, Uganda – April 2024] and cross-section schematic adapted and modified from Gensch et al. (2018).

3.1.2 Parameters influencing faecal sludge properties

Previous sections have established that FS composition is very variable and therefore, ensuring uniform treatment standards and consistent quality of recovered products is challenging (Strande et al., 2018). Similar issues have been faced with other types of waste, such as municipal solid waste, and the effective control of source conditions has been crucial in achieving consistent recovery of marketable end-products (e.g. through separation of recyclables) (World Bank, 2012). Some of the key parameters that influence FS properties are described here, based on a critical review of existing literature. Urine diversion and addition of cover materials are highlighted as parameters of interest, as they constitute potential household-level interventions that can be deployed as source control measures in the context of resource recovery.

3.1.2.1 Household-level interventions

3.1.2.1.1 Source separation of human excreta

Source separation of faeces and urine has often been reported as being beneficial for treatment efficiency and resource recovery, as it allows the separate treatment of the two excreta streams based on their distinct characteristics and chemical composition (Larsen, 2020; Chipako & Randall, 2020b). Notably, the urine stream contains most of the nutrients excreted from the human body (80%–90% of N, 50%–65% of P and 50%–80% of K) but only accounts for ~1% of wastewater flows reaching treatment plants (Larsen et al., 2013). In its undiluted form, as the output of source-separating toilets, urine has an NPK ratio similar to that of commercial fertilisers (Viskari et al., 2018). Faeces, on the other hand, are rich in carbon (~50% on a dry weight basis) and hence have significant soil improving properties, while also containing around half of the P excreted from the human body (Rose et al., 2015). Separated faeces exhibit high calorific value (>20 MJ/kg on a dry weight basis) and are suitable for energy recovery (Ward et al., 2014; Hübner et al., 2019).

At present, the investigation of how a specific parameter, like source separation, affects FS and end-product characteristics cannot easily be quantified based on existing knowledge, as there are many influencing parameters that are challenging to monitor for on-site sanitation systems (see **Sections 3.1.2.2-3.1.2.3**). For example, a previous study that compared FS from VIPs and UDDTs did not observe significant differences in nutrient content and noted that the studied UDDTs are often not used correctly and likely include urine inputs (Krueger et al., 2021a). Therefore, there is a need for more research on the quantification of urine diversion benefits for resource recovery under controlled feedstock conditions, as well as on the development of novel, affordable technologies for treatment and recovery (Maurer et al., 2006; Larsen et al., 2009; Larsen, 2020; Chipako & Randall, 2020a).

3.1.2.1.2 Addition of cover materials

On-site dry toilets that do not use water for flushing usually involve the addition of a biomass material to cover human excreta – to prevent odour and insects and to facilitate on-site treatment (Hübner et al., 2019; VanRiper et al., 2022). Often this cover material is some form of wood (e.g. sawdust) or agricultural waste (including coconut shells, coffee husk, etc.), depending on local availability (World Bank, 2019). The addition of these biomass feedstocks alters the material balance in FS treatment, but there is currently limited understanding and control of the relative quantities of materials in sanitation facilities (Hashemi et al., 2019).

The organic composition of lignocellulosic biomass sources, such as wood, is different to that of human excreta, hence their systematic addition to sanitation facilities is expected to alter the behaviour of carbon within FSM systems (Ippolito et al., 2020). Nevertheless, very little is known about the flow of carbon during FS treatment and end-uses of recovered products, as well as on the effect of cover material addition on carbon dynamics (Johnson et al., 2022). Further research is needed to quantify potential synergistic benefits and map the distribution of carbon when biomass materials are added in sanitation systems as cover materials.

3.1.2.2 Type of containment facility

In terms of containment type, a clear distinction can be made between fully enclosed technologies (e.g. CBS or properly constructed septic tanks) versus latrine-based containment facilities (see **Section 3.1.1**). Pits may be partially lined or unlined and often allow the infiltration of soluble compounds into the soil, as well as the entry of foreign materials (e.g. sand, water, etc.) inside the pit (Dzwaïro et al., 2006; Endris et al., 2024). Therefore, FS properties are expected to differ, not only between different sanitation technologies, but also between facilities of the same category, depending on case-specific construction characteristics (Velkushanova et al., 2021).

An indirect effect of different containment types, and containment unit sizes, is the variation in emptying frequency and hence, on-site storage time (Conaway et al., 2023). As a baseline, sewerage sanitation systems transfer wastewater directly to treatment plants and therefore allow for minimal or no decomposition before treatment. For on-site sanitation systems, the emptying frequency can vary between days (e.g. CBS toilets) to months or years (e.g. septic tanks and pit latrines) (World Bank, 2019; Capone et al., 2020). The storage time further varies among types of on-site sanitation facilities due to structural differences, variable uses (e.g. household versus commercial), different collection frequencies and potential additional inputs that may increase the accumulation rate (e.g. solid waste) (Strande et al., 2018).

In turn, storage conditions and duration directly affect the degree of FS decomposition (aerobic or anaerobic) and therefore influence the properties of FS reaching treatment plants (Nwaneri et al., 2008).

In the case of pit latrines this can also depend on the size and depth of latrines, as in many cases only the top (and middle) layers can be effectively emptied, especially for very deep latrines, meaning that the “older” sludge layers are not transferred to treatment (Thye et al., 2011). Container-based toilets differ from other on-site containment types as they usually offer weekly collection services, meaning that little decomposition is expected before treatment (World Bank, 2019). The degree of on-site decomposition affects the organic composition of FS and can determine the energy balance of FS treatment applications (Krueger et al., 2021b).

The addition of flush water in toilets is also an important factor affecting FS properties (Tilley et al., 2014). Flushing is a common practice for sewerage sanitation systems and septic tanks, while pour-flush latrines also include small quantities of added water (Mara, 1985; Welling et al., 2020). Moreover, wastewater and septic tank sludges usually receive inputs from other household uses, including kitchen and shower waste (Strande et al., 2014). Therefore, the moisture content and consistency of FS are directly affected by the relative quantities of water added in sanitation facilities. For pit latrines, the moisture content and rheological properties of FS have implications for pumping requirements during emptying (Septien et al., 2018). Drying requirements, and the need for solid-liquid separation and mechanical dewatering, are also determined by the initial moisture content of the sludge (Tayler, 2018; Septien et al., 2020).

3.1.2.3 Other factors

3.1.2.3.1 Environmental conditions

Geographical characteristics and seasonality can also have an influence upon FS properties (UN, 2019; Lebu et al., 2024). This can be attributed to changes in temperature which may affect the degree of on-site degradation, as well as changes in precipitation rates (Stintzing et al., 2007; Bassan et al., 2013). For tropical climates, rainfall changes are exacerbated due to the occurrence of distinct wet and dry seasons (Kwiringira et al., 2016). Effects are particularly obvious for unlined or partially lined pit latrines, as heavy rainfall can lead to frequent pit filling and cause pollution of surrounding surface water and groundwater during flooding (Sakijige et al., 2012). Climate change effects and extreme flooding episodes are expected to increase the occurrence and severity of such events, making climate change adaptation an important aspect of sustainable sanitation systems (Sherpa et al., 2014). Drought episodes are also more frequent due to climate change and may pose a concern for sanitation systems relying on consistent water supply to operate (i.e. flushed toilets) (Hyde-Smith et al., 2022).

3.1.2.3.2 User behaviour

Sanitation facilities are interactive pieces of infrastructure and as such, they are directly influenced by the behaviour of their users (Changara et al., 2018). Diet habits have been associated with differences in the moisture, organic and inorganic composition of FS, especially due to variance in fibre intake, as

well as protein consumption and fat intake (Rose et al., 2015; Krueger et al., 2021a). In terms of water and/or biomass addition, users' hygiene habits affect expected toilet inputs, e.g. additional water for “washers” versus toilet paper for “wipers” (Tobias et al., 2017). These habits are often a part of cultural norms and as such, can differ between regions, individual countries or religion groups (Ahmed & Ahmed, 2017). Solid waste may also be disposed in toilets and pits, including plastic waste, sanitary products (e.g. pads), textiles and other miscellaneous waste (Ahmed, 2018).

3.1.2.4 Summary

Overall, the characteristics of FS reaching treatment plants depend on a large number of factors that vary with time, geographical characteristics and cultural contexts. Therefore, the investigation of the effect of a single parameter (e.g. urine diversion, cover material addition) is challenging for FS samples, as other influencing factors cannot be kept constant or ignored. From that perspective, pure human excreta samples can be considered as a valuable substrate for fundamental research on FS treatment and resource recovery, as long as a consistent sampling protocol is established. The comparison of FS and human excreta samples is further discussed below.

3.1.3 Properties relevant to resource recovery

The previous sections have established an understanding of the different types of on-site sanitation facilities, their outputs and variability of material properties, as well as the main parameters influencing FS properties. Building on that knowledge, this section provides a critical overview of key FS and human excreta properties relevant to resource recovery.

While properties related to pathogenic indicators are outside the scope of this research, they should be highlighted as an integral part of FS treatment and resource recovery, particularly for environmental applications in agriculture. On-site decomposition during containment can only provide limited pathogen destruction and further FS treatment is essential to ensure safe disposal or reuse (Changara et al., 2019). Biological treatment methods like anaerobic digestion and composting can achieve sufficient pathogen elimination, however their successful application is dependent on the maximum temperature being reached during treatment, which may require long retention times on the scale of days or months (see **Section 3.2**) (Smith et al., 2005; Niwagaba et al., 2009). For high-temperature treatment technologies, such as pyrolysis (operating at $>300^{\circ}\text{C}$), it is safe to assume that pathogen destruction is guaranteed (Krueger et al., 2020).

3.1.3.1 Nutrient content

The nutrient content of FS sources is a significant indicator of their suitability for agricultural uses. While treatment conditions also influence end-product quality, several previous studies have reported that feedstock composition is one of the most important influencing factors for the fertiliser value of

recovered products (Liu et al., 2014; Gold et al., 2018a). To this end, understanding FS properties and source conditions is essential to achieve consistent quality of recovered fertilisers.

The NPK content, as well as calcium (Ca) and magnesium (Mg) concentrations, of different FS and human excreta sources are shown in **Figure 3.4**, based on results presented in previous studies listed in **Tables A1-A2** (Appendix B). Faecal sludge samples from various sources (including pit latrines and VIPs, UDDTs, septic tanks and CBS toilets) are compared against pure samples of faeces and mixed human excreta (i.e. faeces and urine) collected from volunteers. Nevertheless, as noted in **Section 3.1.2**, the variety of influencing factors affecting FS composition (including storage time, additional inputs, environmental conditions, etc.) make the direct comparison between studies challenging. Therefore, this information is presented to provide an overview of FS properties and their variability for the broad categories of FS, faeces and human excreta.

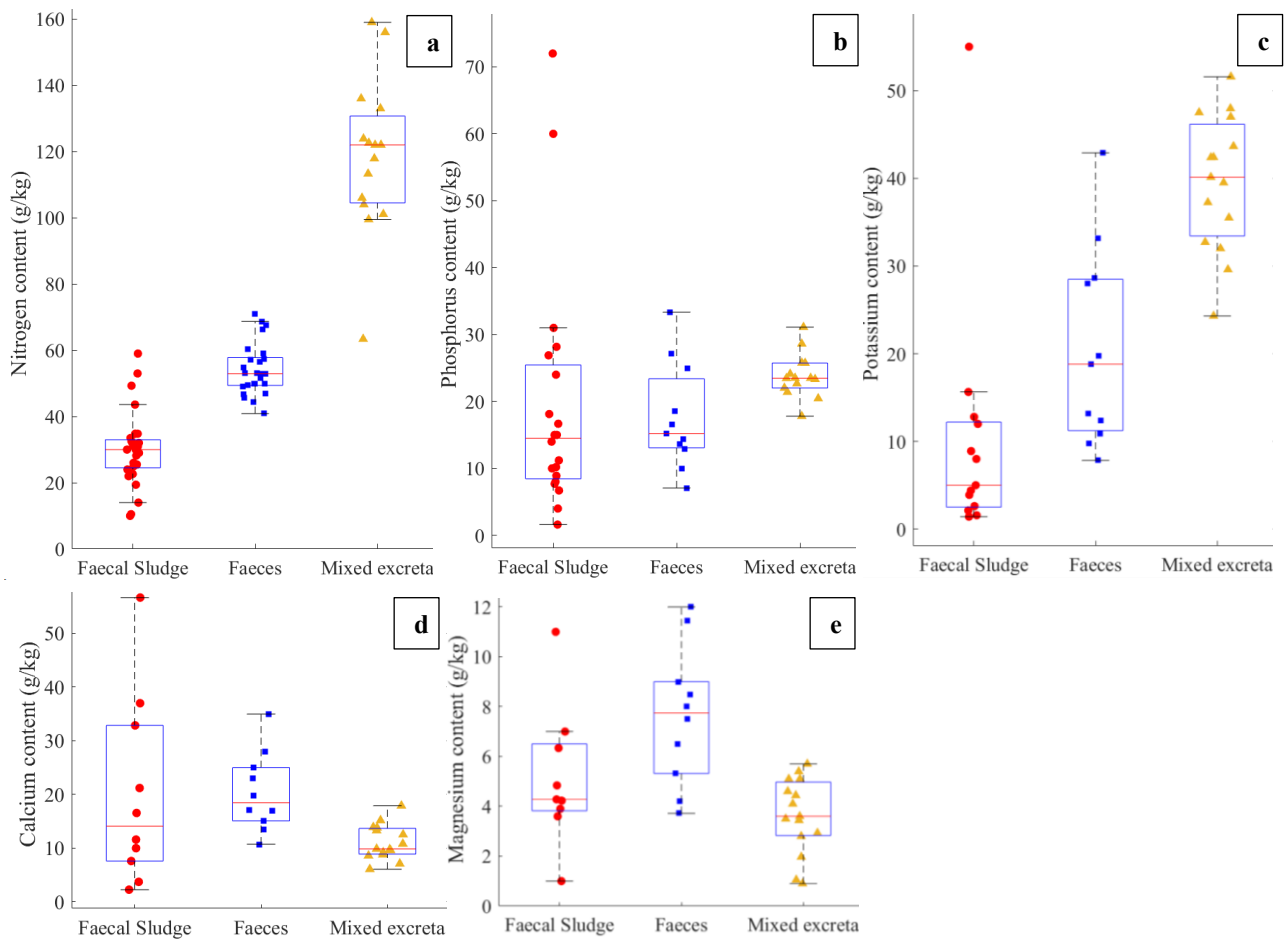


Figure 3.4: Concentration of (a) nitrogen (N); (b) phosphorus (P); (c) potassium (K); (d) calcium (Ca); and (e) magnesium (Mg) (g/kg) in faecal sludge, faeces and mixed excreta samples from published literature. Included studies are shown in **Tables A1-A2** (Appendix B). Results are presented on a dry weight basis (or adjusted based on the reported moisture content).

In terms of N and K concentrations (**Figure 3.4a,c**), mixed human excreta (urine and faeces) are the most nutrient-dense source, followed by separated faeces and FS. This is expected, as most of the nutrients excreted from the human body are found in urine (Viskari et al., 2018). While most FS sources

included in **Figure 3.4** contain urine inputs, these are generally diluted with additional (non-excreta) components found in on-site containment facilities (see **Sections 3.1.1-3.1.2**). Soluble nutrients can also be leached into the surrounding environment (e.g. for unlined pits), explaining the lower values observed for FS samples. For Ca and Mg (**Figure 3.4d,e**), which are also important nutrients for plant growth and soil health, higher concentrations are reported for faeces compared to mixed human excreta, in line with the rate of excretion between faeces and urine (Rose et al., 2015). For P (**Figure 3.4b**), which is generally found in approximately equal parts in faeces and urine, comparable concentrations are expected for sludges derived from toilets with or without urine diversion.

The deviation of values within each feedstock group can be partly attributed to dietary habits, particularly for K which has been reported to vary significantly for different diets (e.g. depending on fibre intake, meat consumption, etc.) and shows large variation in human faeces (Klevay et al., 2007). Extreme outlier values reported for FS can be attributed to potential irregular inputs in on-site sanitation facilities, suggesting that comprehensive sampling protocols and quality control are essential practices for FS research (Krueger et al., 2021a). The overlapping values between FS and faeces can be attributed to the inclusion of studies using CBS sludge, which has similar characteristics to the composition of separated faeces (Krounbi et al., 2018).

For N, which is mostly excreted via urine, it can be expected that the highest variability of concentrations is observed for mixed human excreta, depending on urine quantity, time of day and protein intake of toilet users (Bingham, 2003; Moore et al., 2018). To supplement these data, the nutrient concentrations of separated urine samples from literature (studies included in **Table A3** – Appendix B) are shown in **Figure 3.5** (expressed in mg/L), confirming that N is the most abundant nutrient in human urine and that its concentration is highly variable among samples. The interquartile range (IQR) for N in human urine samples lies between 5000-8580 mg/L, while other nutrients show lower concentrations and more consistent value distributions, with notable outliers.

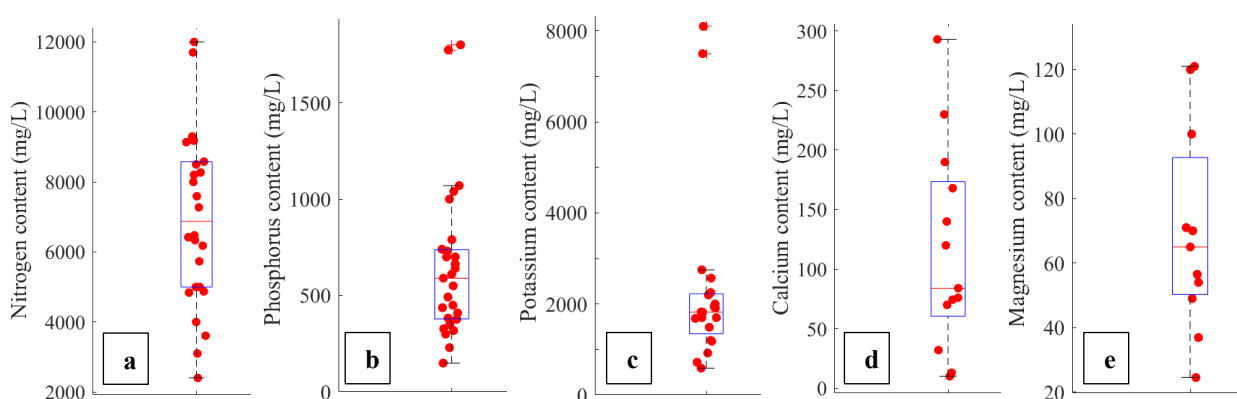


Figure 3.5: Concentration of (a) nitrogen (N); (b) phosphorus (P); (c) potassium (K); (d) calcium (Ca), and (e) magnesium (Mg) in urine samples (mg/L) from published literature. Included studies are shown in **Table A3** (Appendix B).

Overall, the compilation of findings presented in this section provides a visual representation of the nutrient recovery potential of each excreta stream (i.e. faeces and urine) and the deviation between different FS and excreta sources. Therefore, the feedstock source should be carefully considered prior to treatment and recovery applications.

3.1.3.2 Thermally relevant properties

Figure 3.6 includes key components of FS and faeces samples, relevant to thermal behaviour, energy recovery and carbon management (from studies shown in **Tables A1-A2** – Appendix B). Values for mixed human excreta are not included, as relevant data were not found in the literature; a fact that indicates a potentially substantial gap in existing knowledge.

The moisture content (**Figure 3.6a**) is an important parameter to determine the technical and financial viability of treatment via thermal technologies (Septien et al., 2020; Somorin et al., 2020). For FS, moisture content has been found to vary significantly between the different containment systems (Krueger et al., 2021a). Generally, sludge from septic tanks is reported to have the highest moisture content due to additional water added for flushing or other household uses (e.g. greywater from showers) (Strande et al., 2018). Urine-diverting toilets and VIPs have been reported to have similar moisture content, potentially due to the incorrect use of UDDTs by users (i.e. input of urine in the faecal storage component) (Krueger et al., 2021a). Similar to agricultural properties, an overlap of values between FS and faeces is observed, likely due to the inclusion of studies using CBS sludge, which shows similar composition to pure faeces (i.e. lower moisture content compared to traditional FS sources).

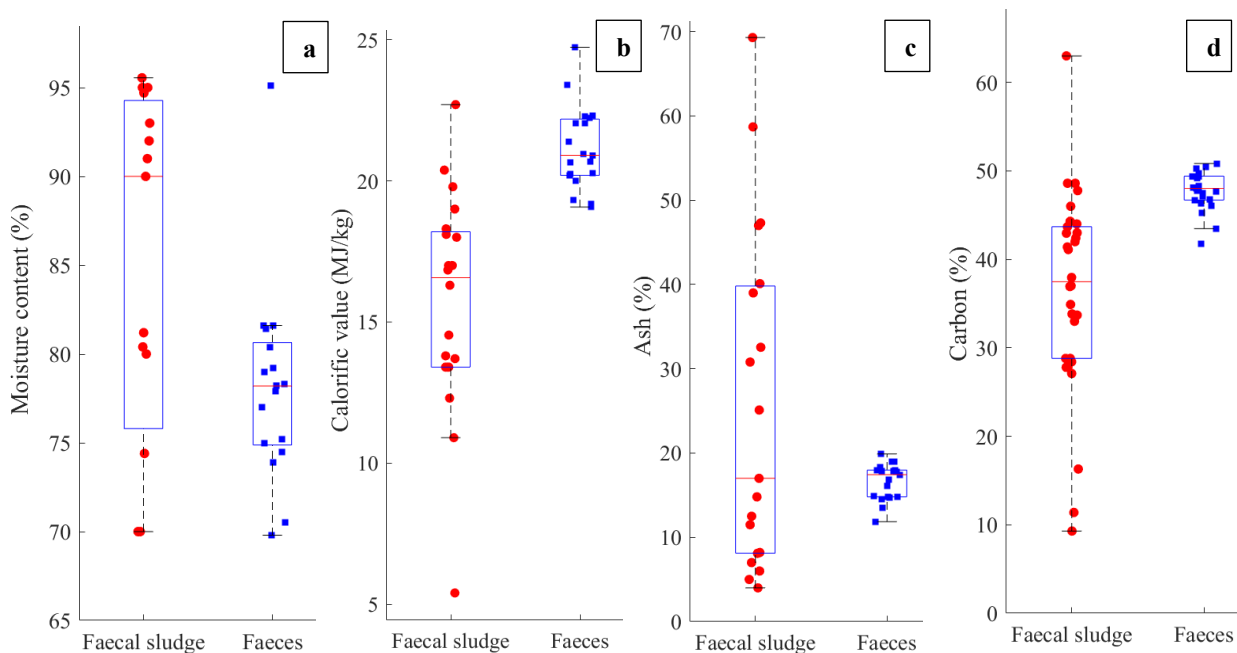


Figure 3.6: Moisture content (a), calorific value (b), ash (c) and carbon content (d) (expressed in % w/w) for faecal sludge and faeces samples from published literature (studies included in **Tables A1-A2** – Appendix B). Results, apart from moisture content, are presented on a dry weight basis.

In terms of energy recovery potential, FS samples from different studies showed significant variability in calorific value, with the IQR lying between 13.4-18.2 MJ/kg (**Figure 3.6b**). This is heavily dependent upon the ash content of the samples, for which the IQR was 8.1%-39.8% (**Figure 3.6c**) (Sharma et al., 2023). The variability of ash content is also dependent on the different types of containment systems (e.g. unlined latrines allowing sand/soil to enter the pit) (Seck et al., 2015; Hafford et al., 2018). Faeces have a much narrower range in calorific value and ash content, with IQRs within 20.2-22.2 MJ/kg and 14.8-18% respectively (**Figure 3.6b,c**). While to date research on urine diversion has primarily focused on nutrient recovery, these results indicate the higher potential for energy recovery from source-separated faeces compared to traditional FS sources. Relevant knowledge is still limited and constrained by the variable composition of different FS sources, creating a need for further investigation under controlled conditions (e.g. analysing separated and mixed human excreta samples from the same sanitation users).

Carbon storage and avoided emissions are also becoming increasingly important aspects of resource management within sanitation systems, particularly for the context of thermal treatment technologies (Gallego-Schmid & Tarpani, 2019; McNicol et al., 2020). Although faeces exhibit a consistently high carbon content (median value 48%), FS samples have a wide range of values with the IQR lying from 28.8 to 43.7% and a large number of outlier values (**Figure 3.6d**). The inconsistent results for FS and the lack of studies on carbon flows during FS treatment highlight the need for further research, particularly on CBS toilet outputs that have similar consistency to pure faeces and can realistically be expected to have higher carbon storage potential (VanRiper et al., 2022; CBSA, 2023). Although hard to quantify based on existing knowledge, source separation of faeces and urine can be a promising strategy to increase carbon sequestration potential by avoiding the dilution of the carbon-rich faecal stream with water and minerals from urine (Rose et al., 2015). The addition of biomass cover materials (see **Section 3.1.2.1.2**), such as wood, to CBS toilets may further increase carbon management benefits and is an interesting field for investigation (Hübner et al., 2019; VanRiper et al., 2022). The next sections will focus on pyrolysis as a thermal technology which offers many opportunities in the FSM nexus.

3.2 Options for faecal sludge and human excreta treatment: Pyrolysis

There are several technologies that can be used for FS treatment including biological; mechanical and physio-chemical; and thermal/ thermochemical processes (Sohail et al., 2016; McConville et al., 2020). A list of several available technologies for FS treatment, and relevant published literature, is included in **Table 3.1**.

Table 3.1: Technologies for faecal sludge treatment and relevant references from literature.

Treatment	References
Biological processes	
Anaerobic digestion (AD)	(Forbis-Stokes et al., 2016; Cheng et al., 2017; Kim et al., 2019; Changara et al., 2019)
Composting	(Vinnerås et al., 2003; Niwagaba et al., 2009; Nartey et al., 2017)
Vermicomposting	(Rodríguez-Canché et al., 2010; Lalander et al., 2015b; Komakech et al., 2016)
Black soldier fly (BSF) treatment	(Lalander et al., 2013, 2015a; Gold et al., 2018c)
Lactic acid fermentation	(Anderson et al., 2015; Andreev et al., 2017; Odey et al., 2018)
Constructed wetlands	(Koottatep et al., 2005; Bui et al., 2019)
Mechanical and physio-chemical processes	
Membrane treatment	(Bakhshayesh et al., 2019)
Sorption	(Kabdaşlı & Tünay, 2018)
Freeze/thawing techniques	(Schmidt & Alleman, 2012; Randall & Nathoo, 2018)
Precipitation	(Shiba & Ntuli, 2017; Zheng et al., 2018)
Thermal and thermochemical processes	
Thermal drying	(Seck et al., 2015; Gold et al., 2017; Septien et al., 2020),
Combustion	(Onabanjo et al., 2016a; Yermán et al., 2017)
Pyrolysis	(Gold et al., 2018a; Krueger et al., 2020; Bleuler et al., 2020)
Gasification	(Onabanjo et al., 2016b; Recalde et al., 2018; Bittencourt et al., 2019)
Hydrothermal carbonisation (HTC)	(Afolabi & Sohail, 2017; Fakkaew et al., 2018)

Of these treatment groups, thermal and thermochemical technologies offer the advantage of quicker process times (e.g. from days/weeks for composting to minutes/hours for pyrolysis or hydrothermal carbonisation), complete pathogen destruction (temperatures typically >300 °C), reduced land requirements (e.g. compared to wetlands and anaerobic digestion), flexibility to operate with inconsistent (or batch) loads, as well as several resource recovery benefits (Liu et al., 2014; Hübner et al., 2019; Somorin et al., 2020b). However, further research is needed as there is still limited availability of research data for full-scale applications (Krueger et al., 2020). Practical challenges for upscaling include the optimisation of process energy efficiency, sophisticated operational control and requirements for emissions control (Liu et al., 2014; Sohail et al., 2016; Andriessen et al., 2019). Usually, pre-treatment steps like drying and dewatering are required for the process to be energy-efficient (Bond et al., 2018).

Although these challenges highlight the need for further research and refinement of operational parameters, thermochemical processes like pyrolysis, gasification and hydrothermal carbonisation (HTC) have been gaining interest in recent years. Particularly pyrolysis, with the production of biochar as a by-product, offers a promising solution to the FSM challenge that can be built around resource recovery and carbon sequestration (Bleuler et al., 2020; Somorin et al., 2020b). This can be directly attributed to the range of resource recovery options that pyrolysis products offer (see **Section 3.3**), while also reducing the volume of material outputs and hence benefiting storage and transport logistics (Graça et al., 2024). The assurance of pathogen elimination at high temperature treatment offers a meaningful advantage, especially for agricultural uses of process outputs (see **Section 3.3.1**) (Nicholas et al., 2023b).

While anaerobic digestion (AD) and composting are well-established technologies that can also successfully eliminate pathogens, this generally depends on the time-temperature combination during treatment (as well as other operational factors and substrate characteristics) and cannot be guaranteed to be a suitable solution for all treatment plants, particularly for feedstocks of varying quantity and quality, such as FS (Smith et al., 2005; Niwagaba et al., 2009; Changara et al., 2019). For example, previous studies on FS composting reported that several unexpected factors (including FS composition) may hinder pathogen inactivation, even at the recommended temperature range (>50-55 °C) (Vinnerås, 2007). Mechanical and physio-chemical processes address the requirements of specific treatment steps or resource recovery objectives but do not generally constitute primary FS treatment technologies. Their operational feasibility is often dependent on the concentration of suspended solids (e.g. for membranes) and the achieved level of sterilisation can vary (e.g. for sorption) (Jimenez et al., 2010; Bakhshayesh et al., 2019; Oishi et al., 2023).

Pyrolysis is therefore a useful technology for resource recovery research as it allows for direct comparison of different recovery routes after treatment (including nutrients, energy, carbon), provides flexibility for the treatment of variable feedstocks, such as FS, and ensures the safety of products in terms of public health implications. In any case, context-specific assessments, including full life cycle assessments are recommended to choose between treatment technologies for FS (Gallego-Schmid & Tarpani, 2019). Several studies have been conducted for biological FS treatment technologies, especially AD and composting (Afifah and Priadi, 2017; Forbis-Stokes et al., 2020), while knowledge on thermochemical technologies and FS pyrolysis is more limited. To ensure that meaningful comparisons between technologies are feasible, it is essential to deepen our understanding of FS pyrolysis, both in terms of operational parameters and in terms of final product quality. This should be done by a combination of fundamental research studies and full-scale case studies.

Over the past decade, publications on FS pyrolysis have been increasing (see **Figure 3.7**), with interest mainly around nutrient and energy recovery. A summary description of articles relevant to FS pyrolysis

that have been published to date is included in **Table A4** (Appendix B). Notably, the majority of existing studies were conducted at laboratory-scale, with very few case studies of at-scale applications currently available (Krueger et al., 2020).

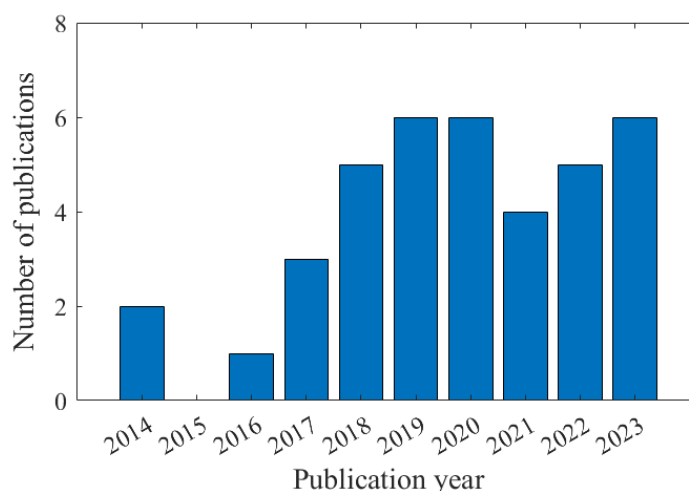


Figure 3.7: Number of publications on faecal sludge pyrolysis to date (per year of publication). List of studies included in **Table A4** (Appendix B).

3.2.1 Pyrolysis description and mechanisms

Pyrolysis has been studied for the treatment of many organic feedstocks, including wood biomass, agricultural waste and sewage sludge (Méndez et al., 2013; Ippolito et al., 2020). Historically, it has been used for thousands of years for charcoal production and within the energy and metallurgical sectors (Lehmann & Joseph, 2015). It is defined as the thermochemical decomposition of carbonaceous materials in an oxygen-free or oxygen-limited environment, usually at temperatures between 300-700 °C (Lehmann & Joseph, 2015). Complex organic materials are broken down to simpler solid, liquid and non-condensable gas (NCG) products that are more amenable for recovery (Basu, 2010). Often an inert gas (e.g. N₂) is used to ensure oxygen is excluded from the process and evolved gases are effectively released (Somerville & Deev, 2020).

When pyrolysis is used to treat biomass feedstock materials, the solid and liquid end-products are referred to as biochar and bio-oil respectively. Pyrolysis can be further categorised based on the range of heating rates used, as “slow”, “fast” and “flash” pyrolysis, each of which will lead to different relative yields and characteristics of end-products (Singh & Kalia, 2017). The yields of different pyrolysis products are also influenced by the composition of the feedstock material, meaning the relative content of cellulose, hemicellulose, lignin and other organic and inorganic constituents (Wang & Luo, 2017).

A graphical representation of the main pyrolysis reactions and products is shown in **Figure 3.8**. The first step of pyrolysis involves the removal of free and bound moisture from the feedstock material during drying and dehydration reactions occurring mostly up to 100 °C and 300 °C respectively (Basu,

2010; Wang & Luo, 2017). The dry feedstock material then undergoes primary decomposition reactions, including depolymerisation and fragmentation, to produce (primary) char, gases and tar (Mohan et al., 2006).

Secondary gas-phase reactions include secondary carbonisation of volatile tars, and repolymerisation during ongoing carbonisation at high pyrolysis temperatures or during longer residence times (Basu, 2010). During these reactions condensable and non-condensable gases are formed and char recondensation occurs (Sahoo et al., 2021). The non-condensable gases are released and condensed gases form oil upon cooling.

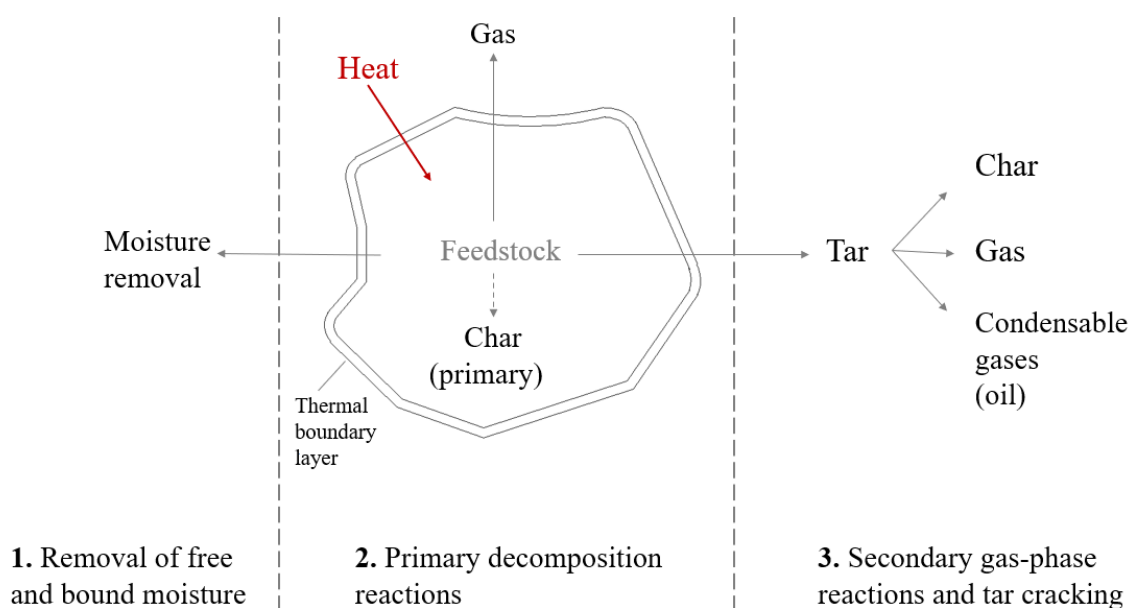


Figure 3.8: Graphical description of pyrolysis reactions and products, adapted and modified from Basu (2010).

A note on terminology and similar processes

There are several overlapping terms of similar but distinct processes that have been used to describe thermochemical conversion technologies, including torrefaction, carbonisation, gasification and pyrolysis (Herak, 2021). These need to be individually understood to avoid misinterpretation of relevant scientific information. Torrefaction refers to a partially complete pyrolysis process, usually at temperatures up to 300 °C, aiming to reduce the mass and volatile content of biomass materials (Dahlquist, 2013). A common application of torrefaction is to produce compact solid residue to be used in coal-fired kilns and boilers (Basu, 2010). Carbonisation is a very slow pyrolytic process, usually in the scale of days, and aims to produce charcoal rather than condensable and non-condensable volatile fractions (i.e. oil and gas) (Basu, 2010). It is still a commonly used term for traditional pyrolytic applications, particularly in developing countries, that use informal kilns (e.g. metal drums or brick kilns) to produce biochar (see **Chapter 8**).

Gasification is the conversion of organic feedstocks into syngas (including CH₄, H₂, CO, CO₂ and N₂) (Aziz et al., 2021). It is a two-step process whereby pyrolysis is an essential pre-step process for the gasification of residual char (i.e. after devolatilisation) to occur at high temperatures (>700 °C) in the presence of a gasification agent (air, steam, etc.) (Speight, 2020).

3.2.2 Pyrolysis operational parameters

Operational conditions during pyrolysis that influence product characteristics include the heating rate, highest heating temperature (HHT), residence time, particle size and inert gas flow rate (Somerville & Deev, 2020; Ippolito et al., 2020). A common finding among previous studies is that feedstock composition and pyrolysis HHT are the most significant influencing factors that determine biochar characteristics (Liu et al., 2014; Lohri et al., 2016; Gold et al., 2018a). The influence of pyrolysis HHT on biochar yield and ash content is shown in **Figure 3.9** for different excreta-derived feedstock sources (FS, faeces (human and synthetic) and CBS sludge), based on published literature.

It is evident that pyrolysis temperature influences biochar production, generally following the same trend for different feedstock sources, due to the release of volatiles causing biochar yield to decrease with increasing temperature (**Figure 3.9a**). However, the actual values of biochar yield vary for different feedstocks of varying ash contents and organic composition. Generally, low pyrolysis temperatures favour char formation, while high temperatures (>600 °C) are known to favour gas formation (Zhang et al., 2023). The ash in the feedstock remains in the biochar fraction and is concentrated with increasing temperature, as volatiles are released (**Figure 3.9b**). Notably, the ash content in the biochar is higher for FS compared to excreta and CBS sludge, in line with the relative ash content of the feedstocks (see **Figure 3.6**).

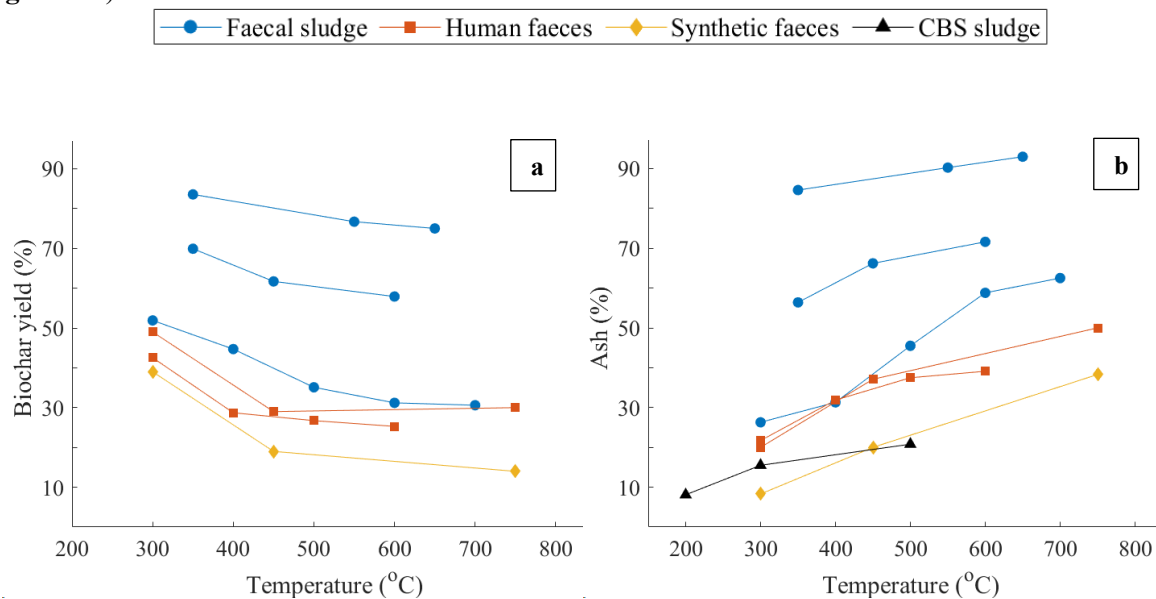


Figure 3.9: The effect of pyrolysis temperature on biochar yield (% w/w) (a) and ash content (% w/w) (b), based on published literature for faecal sludge (Liu et al., 2014; Koetlisi & Muchaonyerwa, 2017; Gold et al., 2018a), human faeces (Ward et al., 2014; Jiang et al., 2023), synthetic faeces (Ward et al., 2014) and container-based sanitation (CBS) sludge (Krounbi et al., 2018). Results are presented on a dry weight basis.

Other influencing parameters include the heating rate, inert gas flow rate and feedstock particle size. The pyrolysis heating rate influences the volatile retention time in the pyrolysis zone and hence determines the distribution of product yields (Basu, 2010). Broadly, slow pyrolysis (0.01 °C/min – 80 °C/min) favours char formation, especially for low temperatures (<500 °C), while fast pyrolysis conditions (up to 10,000 °C/s) can maximise bio-oil or gas production, for moderate (~450-650 °C) or high temperatures (>600-700 °C) respectively (Kan et al., 2016). Therefore, the chosen combination of operational parameters is a determining factor for product yield distribution.

Similarly, the residence time and inert gas flow rate – which influences the vapour residence time in the reactor – affect product yields, in combination with other operational parameters. For example, minimising the solid retention time and maximising the vapour retention time in the condensation system by using a slow inert gas flow rate, can encourage secondary cracking and bio-oil formation and limit NCG formation (Bowles et al., 2023). The effect of feedstock particle size has been reported to be dependent on other operational parameters, such as the inert gas flow rate, due to the changes in intra-particle vapour residence time (Somerville & Deev, 2020).

Therefore, for resource recovery related to biochar applications, slow pyrolysis with adequate retention times and low to moderate temperatures (450-650°C) are generally recommended. Further changes in operational parameters can influence the distribution of products (e.g. between oil and gas) depending on the intended recovery objectives. While it is essential to understand existing literature, the optimisation of operational parameters should be individually examined and validated for different reactor types, on a case-specific basis.

3.3 Resource recovery via pyrolysis of human excreta and faecal sludge

Resource recovery can contribute to sustainable FS pyrolysis, through the adoption of a circular approach that benefits several environmental aspects and encourages the creation of financially viable business models, that can offset front-end sanitation service costs (Krounbi et al., 2019; McConville et al., 2020). Pyrolysis by-products offer a certain flexibility which can further benefit resource recovery (Gold et al., 2018a). Biochars produced from FS can be used in several ways and by different industries, depending on market needs, while at the same time, the produced bio-oil and syngas can offset process energy requirements and help achieve a net-zero or positive energy balance (Liu et al., 2014; Bond et al., 2018). Some of the most widely applied and researched uses of FS-derived biochars are described here.

3.3.1 Agricultural value

Recognition of the agricultural value of biochar materials dates back to the 1800s, when the first “Terra Preta” soils were identified in the Amazonian region (Bezerra et al., 2019). These anthropogenic “black” soils have been attributed to the conversion of biowaste and faecal matter to aromatic carbon-rich soils,

likely via the occurrence of both natural and man-made fires (Glaser et al., 2001; Factura et al., 2010). Remnants of highly fertile carbonaceous soils are still found thousands of years later, suggesting the long-term agricultural potential of these charred materials (Glaser et al., 2002).

Recent literature has investigated several further agronomical benefits of FS-derived biochars. They are a source of essential nutrients, can improve the nutrient and water retention capacity of soils (therefore reducing future fertilising needs), have liming potential and can simultaneously act as a carbon sink by sequestering stable forms of carbon (see **Section 3.3.3**) (Woldetsadik et al., 2017; Krueger et al., 2020). Whilst other biochar types, other than FS-based, have also been recommended for use in agriculture, the inherently high nutrient content of human excreta increases the agricultural value of the end-products (Bleuler et al., 2020). From a practical perspective, FS-derived biochars are generally safe for use on food crops, have no malodour concerns, come in a powder form that is easy to use by farmers, and contain high P and K concentrations (Woldetsadik et al., 2017; Krounbi et al., 2019; Bleuler et al., 2020).

To date, studies on nutrient recovery from FS via pyrolysis have mainly focused on the elemental characterisation of biochars, as well as the effect of the pyrolysis HHT on biochar characteristics (Liu et al., 2014; Gold et al., 2018a). **Figure 3.10** shows the evolution of biochar NPK content with increasing pyrolysis temperature for various FS and human excreta sources found in literature (list of studies included in **Table A4** – Appendix B). The observed trends are consistent, although absolute values are not directly comparable, due to the large number of uncertainties regarding containment conditions highlighted in **Section 3.1.2** (e.g. type of sanitation facility, lining, wet or dry inputs, ash content, etc.). Therefore, there is still a necessity for fundamental research to establish quantifiable relationships between source conditions and biochar agricultural properties, under stable external influencing parameters.

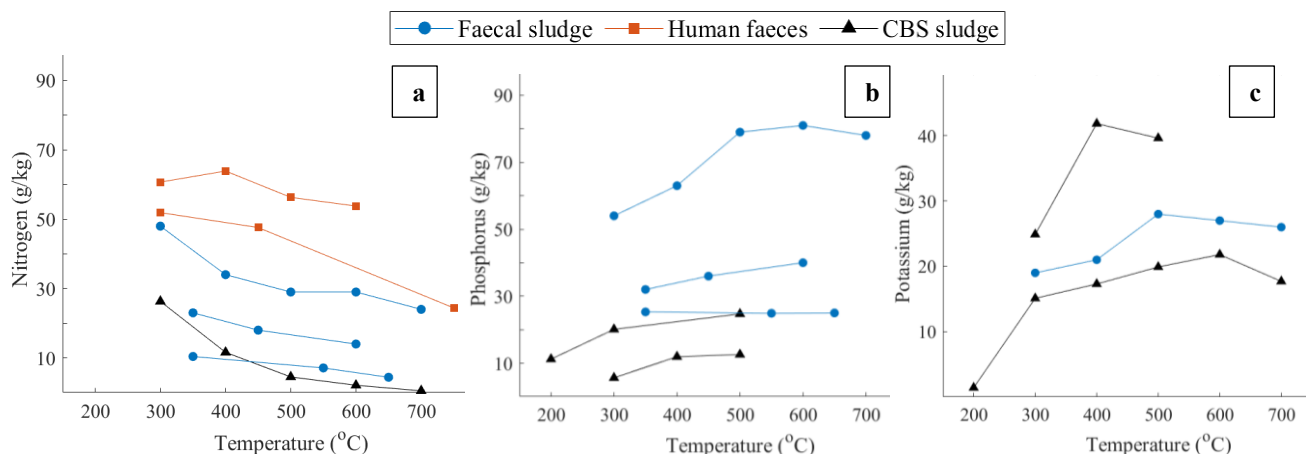


Figure 3.10: The effect of pyrolysis temperature on the nitrogen (a), phosphorus (b) and potassium (c) content (g/kg) of produced biochar, based on published literature for faecal sludge (Liu et al., 2014; Koetlisi & Muchaonyerwa, 2017, 2019; Gold et al., 2018a), human faeces (Ward et al., 2014; Jiang et al., 2023) and container-based sanitation (CBS) sludge (Krounbi et al., 2018, 2019).

Generally, the P and K fractions (**Figure 3.10b,c**) concentrate in the biochar with increasing pyrolysis temperature (up to ~600 °C), while N (**Figure 3.10a**) is released via gaseous emissions at high temperatures and its concentration in the biochar decreases during pyrolysis. There is therefore, a trade-off between PK and N conservation that can affect the agricultural value of biochars, as these nutrients are essential for the production of complete fertilisers (Kutu et al., 2010). Separate collection of faeces and urine before pyrolysis can increase overall nutrient recovery, by avoiding N volatilisation during pyrolysis and allowing for separate N recovery from urine. Although the quantification of these benefits is not possible based on existing knowledge, it could be a significant driver for the production of marketable excreta-based fertilisers of consistent quality. The systemic management of the two excreta streams can further maximise the value of produced agricultural products by combining nutrient supply and soil enhancing properties from urine and faeces respectively (Bai et al., 2018). Further research is needed on combined applications of faecal biochars and urine-derived nutrients, to enable the circular management of human excreta for resource recovery.

3.3.2 Fuel production

The use of FS pyrolysis products for energy recovery and fuel production is also gaining interest, mostly because of the associated financial benefits due to the high demand for affordable fuels (Diener et al., 2014; Andriessen et al., 2017, 2019). Other than bio-oil and gases, the produced biochar can be used as a fuel for cooking and household heating, which is particularly important for LMICs that are currently dependent on solid fuels, such as wood or charcoal, and would need to purchase new boilers/ovens in order to transition to liquid or gas fuels (Ward et al., 2014). Therefore, FS biochar can be an intermediate fuel during the transition to clean energy, as a greener alternative to current unsustainable charcoal production practices (e.g. uncontrolled forest mining) (Lohri et al., 2016).

Nevertheless, several researchers have highlighted the distinction between FS and other lignocellulosic biomass feedstocks historically used for fuel production (Kiwana & Naluwagga, 2016; Gold et al., 2018a). Faecal sludge has higher ash content and increased volatility compared to lignocellulosic biomass, which affects the fuel performance of the produced biochar (Ward et al., 2014). During FS pyrolysis the energy content of the feedstock mostly concentrates in the bio-oil fraction, however, given the additional energy usage of the pyrolysis process itself (including for initial drying and dehydration steps), the feasibility of net energy production largely depends on the initial moisture content of the FS (Liu et al., 2014; Bond et al., 2018).

As shown in **Figure 3.11a**, the calorific value of FS and human excreta biochars decreases with increasing pyrolysis temperature, due to the concentration of ash in the biochar fraction (see **Figure 3.9b**), while the opposite trend is generally observed in literature for lignocellulosic biomass sources such as wood (Hübner et al., 2019). As a result, direct FS combustion has been reported as an alternative

to char production when energy recovery is the main objective, despite obvious environmental and air pollution disadvantages (Bond et al., 2018; Gold et al., 2018a). On the other hand, charred FS has significantly lower volatility compared to raw/torrefied products (**Figure 3.11b**) and is more suitable for household uses, particularly for indoor cooking and heating, due to the smokeless nature of the resulting solid fuels.

Existing data are limited but suggest that FS can realistically be considered as an additional feedstock for solid fuel production when co-pyrolysed with other lignocellulosic biomass sources with better fuel properties (i.e. less ash and higher calorific value) (Atwijukye et al., 2018). Interest in FS-derived fuels is rising rapidly but the transfer of knowledge to real-world applications is currently constrained by the limited available data on feedstock properties and their heterogeneity over time and across locations, as well as operational challenges in low-income settings (Krueger et al., 2020). These challenges need to be addressed through further research to enable the production of sustainable excreta-derived solid fuels at a larger scale.

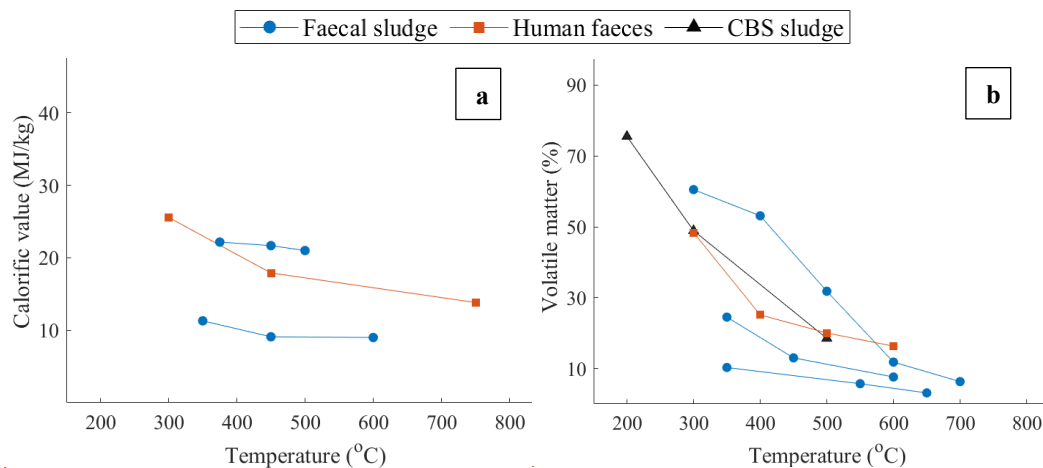


Figure 3.11: The effect of pyrolysis temperature on biochar calorific value (MJ/kg) (a) and volatile matter content (% w/w) (b), based on published literature for faecal sludge (Liu et al., 2014; Koetlisi & Muchaonyerwa, 2017; Gold et al., 2018a; Beik et al., 2023), human faeces (Ward et al., 2014; Jiang et al., 2023) and container-based sanitation (CBS) sludge (Krounbi et al., 2018).

Although agricultural use has been the most applied resource recovery strategy so far, the use of excreta-based feedstocks for energy recovery and fuel production has been reported to provide better revenue generation potential (Diener et al., 2014). An opposing argument, however, is that focus should be placed on wider environmental impacts and benefits associated with resource recovery, rather than solely economic criteria. In any case, it is essential to consider the potential synergies and trade-offs among different resource recovery options. Both the financial and environmental viability of resource recovery business-models are crucial and should be considered on a case-specific basis, depending on feedstock characteristics, available treatment technologies, local environmental and social conditions, spatial characteristics, cost and market analyses (Sohail et al., 2016). Life cycle assessment tools (or equivalent, established methods) are essential for such analyses to produce unbiased results (Peters et al., 2015).

3.3.3 Carbon sequestration

Carbon sequestration is also emerging as an important resource management objective for human excreta and FS. It has been estimated that pyrolysis of human faeces can offset approximately 0.11% of global anthropogenic carbon emissions (Bleuler et al., 2020). Climate change benefits are also expected from avoided emissions that would be released from alternative sludge disposal practices (e.g. untreated disposal or disposal of unstable carbon forms) (Nuagah et al., 2020). Therefore, although small, the climate change mitigation potential during FS pyrolysis is not insignificant, particularly when considered in combination with agricultural and waste management benefits, or as a financial incentive for safe FSM through the use of carbon credit schemes (Bleuler et al., 2020; CBSA, 2023).

Although existing knowledge on carbon sequestration from FS pyrolysis is very limited, relevant biochar characteristics reported from previous studies, namely the carbon and fixed carbon content at various pyrolysis temperatures, are shown in **Figure 3.12**. The fixed carbon portion in the biochar has been reported to increase at higher temperatures (**Figure 3.12a**), due to the release of volatiles and the concentration of aromatic forms of carbon in the solid fraction (Crombie et al., 2013). This indicates the increased stability of FS-derived biochars produced at high temperatures, with potential for long-term carbon sequestration (Fang et al., 2014; Tomczyk et al., 2020). Nevertheless, there are currently no available studies on the comprehensive investigation of carbon stability and storage potential of FS pyrolysis applications.

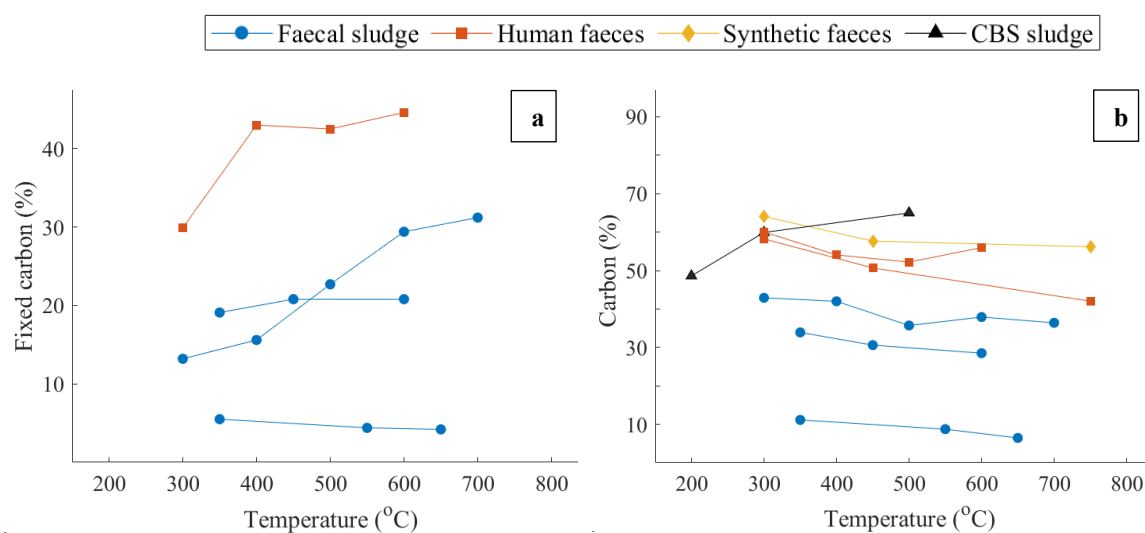


Figure 3.12: The effect of pyrolysis temperature on biochar fixed carbon (% w/w) (a) and (total) carbon content (% w/w) (b), based on published literature for faecal sludge (Liu et al., 2014; Koetlisi & Muchaonyerwa, 2017; Gold et al., 2018a), human faeces (Ward et al., 2014; Jiang et al., 2023), synthetic faeces (Ward et al., 2014) and container-based sanitation (CBS) sludge (Krounbi et al., 2018).

During pyrolysis of FS and human excreta, the total carbon content follows an overall decreasing trend (**Figure 3.12b**) suggesting that a balance needs to be found between carbon retention and biochar stability in FS pyrolysis systems. Sludge from CBS toilets is an exception to this pattern, showing increasing carbon content with increasing temperature, as the addition of wood-based cover materials changes the organic properties of the feedstock, potentially creating a synergistic effect during faeces and wood co-pyrolysis (Somorin et al., 2020b). CBS systems also emit less carbon compared to traditional long-term storage facilities (Reid et al., 2014), as they are enclosed and only store excreta for short periods before treatment (days or weeks) (see **Section 3.1.2.2**), allowing for more carbon to be potentially stored via biochar production (Reid et al., 2014; Rowles et al., 2022).

Therefore, collected data highlight the potential for wood addition to benefit carbon storage during FS pyrolysis, particularly for CBS toilets with outputs consisting of pure human excreta and biomass cover materials, such as wood. Nevertheless, the extent and mechanisms of synergistic effects cannot be quantified based on existing knowledge, as there is limited understanding of the stability and carbon sequestration potential of biochars produced from human excreta and FS, as well as on the effect of co-pyrolysis with other biomass materials.

3.3.4 Other uses

Another promising use of biochars is for pollution remediation and heavy metal immobilisation. Koetlisi and Muchaonyerwa (2019, 2017) found that FS biochar is a suitable substrate for copper (Cu), cadmium (Cd) and zinc (Zn) sorption from industrial effluents and for Cd sorption from wastewater. Woldetsadik et al. (2016) also found FS biochar to be a promising substrate for Cd immobilisation in contaminated soils. Other researchers have studied the adsorption performance of activated sewage sludge char, with positive results (Li et al., 2020). Similar studies for FS are yet to be published but would be an interesting area for future work. Previous research on the production of activated carbons from other waste-derived biomass sources, including coconut shells and corn cobs, show promising results for their adsorption properties (El-Hendawy et al., 2001; Tan et al., 2008; Gratiuto et al., 2008). Therefore, co-treatment of FS with other lignocellulosic biomass (including cover materials used in toilets) can be investigated as a strategy to increase the surface area of the end-products.

Faecal sludge can also be used as a medium for the growing of insect larvae which can then be sold as a source of protein for animal feed (Lalander et al., 2013). For example, Black Soldier Fly (BSF) treatment is currently being used by Sanergy, a company operating in Kenya (World Bank, 2019). Interest in BSF larvae production has been growing over the past years, particularly because conventional feed prices have risen drastically, due to a decline in wild fish stocks globally (Naylor et al., 2009; Diener et al., 2014). The remaining sludge is a compost-like material that can be used in agriculture and offers a second stream of revenue generation for BSF business-models (Diener et al.,

2014). Opportunities to combine BSF treatment and pyrolysis of the remaining sludge can therefore be investigated. Nevertheless, the process performance of BSF treatment can be variable and there are still concerns about the presence of pathogens in the final products (Gold et al., 2018c). Further research is required before the process can be widely applied at scale.

Alternative biochar applications include its use as a building material. The high GHG emissions associated with cement production have raised interest in the incorporation of biochars in cementitious materials (e.g. as a filler alternative) (Akinyemi & Adesina, 2020; Maljaee et al., 2021). However, research has mainly been conducted for wood/plant biomass or sewage sludge derived biochars, with no information currently available for FS-derived biochars. Therefore, future research should assess the feasibility of using FS biochars in the construction industry. Diener et al. (2014) indicated that such uses might have lower market value than other FS biochar uses, mainly because of concerns about quality. Also, the authors found, in a study for Kampala, Uganda, that demand was being met by conventional materials and there was no need for organic-based alternatives like biochar in the local market (Diener et al., 2014). Therefore, a case-specific market analysis would be essential before pursuing alternative resource recovery routes.

3.4 Summary and research gaps

The critical review of existing literature highlighted the opportunities for resource recovery via pyrolysis of human excreta and FS. The development of these opportunities is currently constrained by a lack of understanding of product properties, and how these are influenced by potential source interventions aiming to maximise recovery potential. The following gaps in knowledge were identified which need to be addressed in order to improve the circularity of human excreta management via pyrolysis treatment:

- The investigation of how a specific parameter (e.g. urine diversion, cover material addition) affects FS and biochar characteristics cannot be quantified based on existing knowledge, as there are **many influencing parameters that are challenging to monitor and control** for on-site sanitation systems (e.g. type of containment facility, environmental conditions, user behaviour, etc.).
 - **Pure human excreta samples** are therefore a suitable feedstock for fundamental research on FS treatment and resource recovery, particularly for emerging sanitation technologies, such as CBS.
 - For this, the development of a **specified protocol for human excreta sampling** in the context of sanitation research is needed to ensure consistency among experimental studies.

- Existing literature on the investigation of **urine diversion** as a source control strategy has focused on nutrient recovery. Nevertheless, in the context of pyrolysis, there is still a **lack of fundamental research that establishes quantifiable relationships between source conditions and biochar agricultural properties**. More research is needed on **circular approaches of managing faecal biochars and urine-derived nutrients** to produce complete soil improving and fertilising products.
- There is **very limited knowledge** on how separation of human excreta affects **energy recovery** (including solid fuel production) **and carbon dynamics** during pyrolysis.
- The addition of biomass **cover materials** evidently alters the material balance and carbon behaviour during FS pyrolysis, but there is currently **limited understanding of the effects on carbon flows and carbon sequestration** potential – including on potential synergistic benefits during excreta-wood co-pyrolysis.
- Finally, there is a **very limited number of case studies** for full-scale pyrolysis treatment plants. These will be essential as resource recovery applications from FS pyrolysis are upscaled, and to **allow the statistical interpretation** of findings, which is currently only feasible for laboratory-scale studies.

4 Materials and methods

4.1 Human excreta sampling protocol

This research required the collection of faeces and urine samples from adult volunteers. Samples of source-separated faeces and urine were collected from a total of 24 volunteers (in London, United Kingdom) over three periods, as described in **Table 4.1**. Ethical approval was received from the Research Governance and Integrity Team of the College (ref. 21IC6817) and samples were obtained according to the Imperial College Healthcare Tissue Bank process (licence 12275) (formal approval letters included in **Appendix C**). A sampling protocol was developed to ensure the process was easy and safe, and in line with ethical approval requirements and Human Tissue Bank regulations. Volunteers were given appropriate containers and materials for collection (see **Figure 4.1** and **Figure 4.2**), as well as a detailed information sheet and sampling instructions (refer to **Appendix C**). All samples were anonymised and participants were asked to voluntarily disclose their diet type (vegetarian/vegan or non-vegetarian).

Table 4.1: Description of the sampling protocol followed in this research for human excreta collection.

Period	No. volunteers	Type of samples	Relevant chapter	Diet type
September-December 2021	12 (*A)	Source-separated human faeces (SSF) and urine (SSU)	Chapter 5	Ten of the volunteers were non-vegetarian and two followed a vegetarian diet
June 2022	10 (*A)	Source-separated human urine (SSU)	Chapter 6	Ten of the volunteers were non-vegetarian and two followed a vegetarian diet
July 2022	12 (*B)	Source-separated human faeces (HF)	Chapter 7	All volunteers were non-vegetarian

*Groups A and B are noted to highlight the separate sampling campaigns that took place during September-December 2021 and June 2022 (Group A participants); and July 2022 (Group B participants). The different naming conventions used in **Chapters 5** and **7** for faeces samples (SSF and HF respectively) were also chosen to highlight the difference in sample origin.

Participants were asked not to collect samples or to self-exclude from the study if they had any condition that can be transmitted through human excreta (including COVID-19) (refer to **Appendix C** for more details). Female participants were asked not to collect samples during menstruation. These exclusion criteria were put in place to ensure the safety of the researcher handling the samples and to avoid causing stress to participants. Participation was strictly voluntary, and participants did not have to provide a

reason for choosing not to collect samples or withdrawing from the study. All samples were delivered to a designated laboratory refrigerator at Imperial College London (during pre-arranged times) so that participants could drop-off samples in privacy, and pick-up the next sampling kit. Samples were prepared according to the requirements of each set of experiments presented in the following chapters, and preparation methods are described in the relevant “Materials and Methods” sections.



Figure 4.1: Content of sampling kits for faeces collection (a) and recommended set up for faeces collection with synthetic sample (green colour) for reference (b).



Figure 4.2: Bottles used for urine collection (500 mL centrifuge bottles, autoclavable, plastic).

4.2 Pyrolysis experiments

The experimental configuration for pyrolysis experiments is illustrated in **Figure 4.3** and **Figure 4.4**. Slow pyrolysis was carried out using a Carbolite HTR 11/75 rotary furnace fitted with a 500mL quartz reactor connected to an oil trap and condenser. The sample mass and operational parameters chosen for each set of experiments (retention time, highest heating temperature (HHT), heating rate and inert gas (N_2) flow rate) are described in the relevant chapters. All connecting interfaces were sealed with heat-proof grease to ensure that all evolved gases would go through the condensing and cooling system before being evacuated from the top of the condenser. The heating programme was set using the in-built temperature controller and the accuracy of temperature readings within the furnace was validated via blank runs (using a thermocouple placed at the centre of the quartz reactor), which showed minimal deviation (<5%) between the set and actual temperature in the furnace (see **Appendix D** “Quality control assurance”).

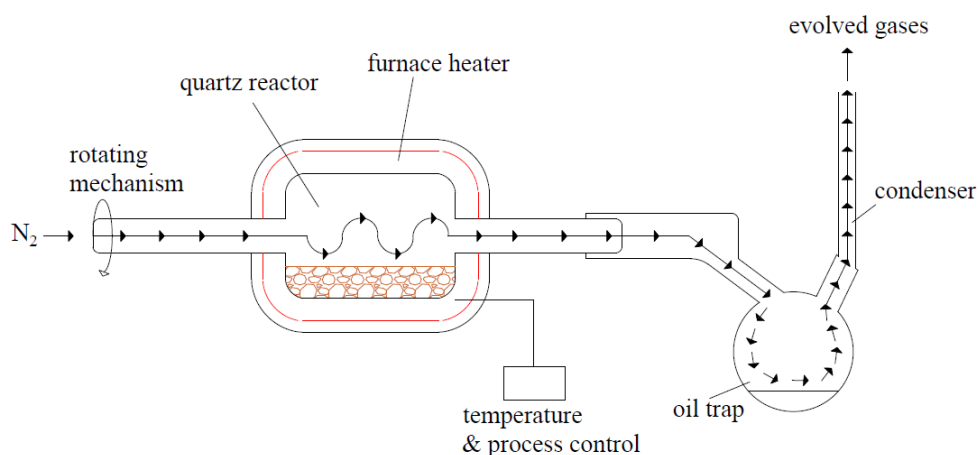


Figure 4.3: Schematic of the experimental pyrolysis set up at the Roger Perry Laboratory (Imperial College London) showing a Carbolite HTR 11/75 rotary furnace fitted with a quartz reactor connected to an oil trap and condenser.

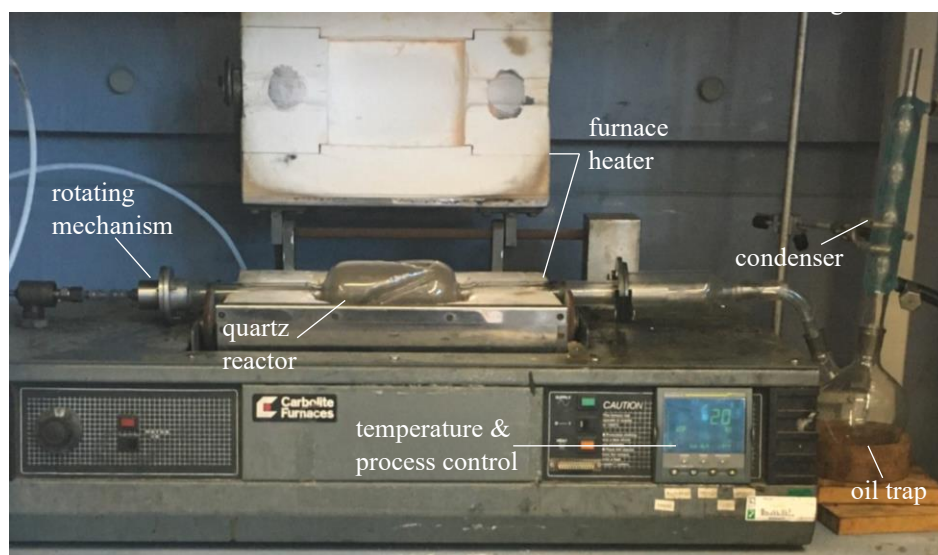


Figure 4.4: Photograph of the experimental pyrolysis set up at the Roger Perry Laboratory (Imperial College London) showing a Carbolite HTR 11/75 rotary furnace fitted with a quartz reactor connected to an oil trap and condenser.

4.3 Analytical methods

General analytical methods used for multiple chapters presented in this thesis are described here. Further analytical methods, equations and calculations are described in the “Materials and Methods” sections of individual chapters. Experiments took place in the Roger Perry Laboratory and the Centre for Infrastructure Materials at the Department of Civil and Environmental Engineering, Imperial College London, unless stated otherwise. All analyses were performed in triplicate and results are presented as mean values, unless it is stated that median values are presented instead. To assess uncertainty in the measured values, standard deviation values are presented in brackets, where available. Samples were manually ground to a particle size of <2 mm for pH, electrical conductivity (EC),

CaCO₃-equivalence and cation exchange capacity (CEC) determination and <500 µm for all other analytical procedures (<125 µm for X-ray diffraction analysis).

4.3.1 Analyses of feedstock and biochar samples

4.3.1.1 Thermogravimetric analysis (TGA) and proximate analysis via TGA

Thermal analysis of samples was conducted on a simultaneous thermal analyser (STA) 449 F5 Jupiter (NETZSCH, Germany) to obtain the thermogravimetric analysis (TGA) and derivative thermogravimetry (DTG) curves under 50 mL/min N₂ flow (10 °C/min heating rate). Proximate analysis was conducted for the determination of volatile matter, fixed carbon and ash content (for dried samples) (Eq. 4.1-4.3), as well as moisture content (for samples analysed on an as-received basis) (Eq. 4.4-4.7), following standard method ASTM D7582-15 (ASTM, 2015) as adapted by Krueger et al. (2021a) for implementation by TGA, using 15±2 mg of sample. The automated procedure programmed on the STA consisted of a drying phase at 105 °C (10 min hold time), followed by continuous heating to 950 °C in an inert N₂ atmosphere (7 min hold time), and then cooling and combustion in air at 600 °C (30 min hold time) (refer to **Figure A5** – Appendix D – for an indicative TGA plot used for proximate analysis interpretation). The chosen heating rate was 20 °C/min for the drying phase and 25 °C/min for the following segments of the analysis.

$$\text{Volatile matter} \left(\text{dry} \frac{w}{w} \% \right) = \frac{m_1 - m_2}{m_1} \times 100 \quad (\text{Eq. 4.1})$$

$$\text{Ash} \left(\text{dry} \frac{w}{w} \% \right) = \frac{m_3}{m_1} \times 100 \quad (\text{Eq. 4.2})$$

$$\text{Fixed carbon} \left(\text{dry} \frac{w}{w} \% \right) = 100 - \text{Volatile matter} \left(\text{dry} \frac{w}{w} \% \right) - \text{Ash} \left(\text{dry} \frac{w}{w} \% \right) \quad (\text{Eq. 4.3})$$

$$\text{Moisture} \left(\text{as received} \frac{w}{w} \% \right) = \frac{m_0 - m_1}{m_0} \times 100 \quad (\text{Eq. 4.4})$$

$$\text{Volatile matter} \left(\text{as received} \frac{w}{w} \% \right) = \frac{m_1 - m_2}{m_0} \times 100 \quad (\text{Eq. 4.5})$$

$$\text{Ash} \left(\text{as received} \frac{w}{w} \% \right) = \frac{m_3}{m_0} \times 100 \quad (\text{Eq. 4.6})$$

$$\begin{aligned} \text{Fixed carbon} \left(\text{as received} \frac{w}{w} \% \right) = 100 - \text{Moisture} \left(\text{as received} \frac{w}{w} \% \right) - \\ \text{Volatile matter} \left(\text{as received} \frac{w}{w} \% \right) - \text{Ash} \left(\text{as received} \frac{w}{w} \% \right) \end{aligned} \quad (\text{Eq. 4.7})$$

where m₀ is the initial (as received) weight of the sample, m₁ is the dry weight of the sample (after drying at 105 °C to constant weight), m₂ is the stable sample weight recorded at 950 °C (7 min hold time) and m₃ is the weight recorded after combustion at 600 °C (30 min hold time).

4.3.1.2 Calorific value (higher heating value)

The higher heating value (HHV) was determined by bomb calorimetry, following standard method ASTM D5865/D5865M-19 (ASTM, 2019) on a 6100 Calorimeter (Parr, USA). An aliquot of 0.5 ± 0.05 g (oven dried) sample was placed into a crucible, the accurate sample weight was recorded and a fuse wire was placed closely above the surface of the sample. The crucible was placed inside the bomb which was sealed and pressure-filled with oxygen, before being placed in a bucket filled with a pre-weighed quantity of room temperature distilled water. After the sample was ignited, the temperature change of the water was automatically calculated by in-built software and the HHV was reported (in MJ/kg) based on internal calibration data. The calibration was performed using standardised benzoic acid ($\text{C}_6\text{H}_5\text{COOH}$) pellets of known HHV (26.454 MJ/kg).

4.3.1.3 CHNS(O) analysis

CHNS analysis was performed on a ThermoScientific Flash Smart Elemental Analyzer according to BS EN ISO 16948:2015 (ISO, 2015). Briefly, the (oven dried) sample was weighed and combusted in a temporarily enriched oxygen atmosphere undergoing a very exothermic reaction (up to 1800 °C). The combustion process generates CO_2 , H_2O (g), nitrogen oxides (reduced to N_2) and sulphur trioxide (reduced to SO_2). Gases were automatically analysed through gas chromatography, using a thermal conductivity detector (TCD), and results were converted to % (w/w) values by internal software based on calibration data. The same method was followed for C analysis of oil samples.

Oxygen analysis was performed on the same analyser, but using a separate reactor that performs pyrolysis instead of combustion (at 1060 °C) producing N_2 , H_2O and CO. The CO was automatically separated and detected by the TCD, and results were calculated by internal software as % values, similar to CHNS data. The quality assurance protocol followed for this analysis is described in detail in **Appendix D**. The calibration was performed using standards of cystine, methionine, sulphanilamide and/or BBOT (2,5-Bis [5-tert-butyl-benzoxazol-2-yl] thiophene) of known CHNSO composition.

4.3.1.4 Fourier-transform infrared spectroscopy (FTIR)

The structure of the biochar and feedstock materials was investigated by Fourier-transform infrared spectroscopy (FTIR). The FTIR spectra were collected based on ASTM E1252-98 (ASTM, 2021), on a Nicolet 6700 FTIR Spectrometer (Thermo Fisher Scientific USA) over the wavenumbers range 4000-500 cm^{-1} , using a diamond ATR-crystal, with internal correction for background signal.

4.3.1.5 Inductively Coupled Plasma Optical Emission Spectroscopy (ICP-OES)

Analysis of the inorganic constituents of samples was performed by Inductively Coupled Plasma Optical Emission Spectroscopy (ICP-OES) on an Avio 500 (Perkin Elmer, USA). The samples were first dry-ashed at $490 \pm 5^\circ\text{C}$ for 4 h (APHA, 1992; Hseu, 2004) and then digested with aqua regia for 2 h at 95°C , based on Standard Method 3050B (US EPA, 1996) as adapted by Krueger et al. (2020). Before analysis, all solutions were diluted with 1 M HNO_3 (within the calibration concentration range) and filtered using a $0.45\ \mu\text{m}$ pore size WCN Whatman membrane. All glassware used during analysis and solution preparation were acid-washed overnight with 10% HNO_3 , to ensure no trace elements were present that could interfere with the analysis. A rigid quality assurance protocol, including spike testing and validation via X-ray fluorescence (XRF) analysis, was followed (described in detail in Appendix D).

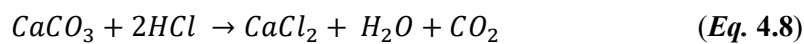
4.3.2 Analyses of biochar samples

4.3.2.1 pH and electrical conductivity (EC)

The pH and electrical conductivity (EC) of biochars were measured at a 1:20 ratio (biochar: H_2O [g:mL]) after mechanical shaking for 1 h (Singh et al., 2017), using a Jenway 3540 combined pH and conductivity meter. The quality assurance protocol followed for this analysis is described in detail in Appendix D.

4.3.2.2 Liming potential (CaCO_3 -equivalence)

The biochar liming potential was determined as CaCO_3 -equivalence by adding 10 mL of standardised 1 M HCl to 0.5 g of (oven dried) sample, shaking for 2 h and leaving the mixture to stand overnight. The solution was then titrated with standardised 0.5 M NaOH until pH 7 was reached (Singh et al., 2017). A blank titration was performed using 10 mL of standardised 1 M HCl and the volume of NaOH consumed (V_b) was recorded. A reference titration using a (dried) CaCO_3 powder instead of the biochar samples was also performed for quality control. The liming potential (% CaCO_3 -equivalence) was calculated based on Eq. 4.8-4.9.



$$\text{CaCO}_3 (\%) = \frac{M \times (V_a - V_b) \times 10^{-3} \times 100.09 \times 100}{2 \times W} \quad (\text{Eq. 4.9})$$

where M is the (standardised) molarity of NaOH used (mol/L), V_a and V_b are the values of the NaOH volume required to neutralise the HCl after contact with the biochar sample or blank respectively (mL), 100.09 is the molar mass of CaCO_3 (g/mol) and W is the mass of the dried biochar sample (g). The 2 in the denominator of Eq. 4.9 is to account for the 2 moles of HCl consumed by 1 mole of CaCO_3 (see Eq. 4.8).

4.3.2.3 Cation exchange capacity (CEC)

Cation exchange capacity (CEC) was determined by saturating 1 g of (oven dried) biochar sample with a 1 M ammonium acetate ($\text{NH}_4\text{CH}_3\text{CO}_2$) solution (40 mL) (at pH 7) overnight, followed by washing with 60% ethanol (4 x 15mL aliquots) and discarding the leachate. Then, a 40 mL solution of 1 M KCl was added to displace the NH_4^+ exchangeable cations overnight (Chapman, 1965). The final solution was filtered through a Whatman no.1 filter under vacuum, adding another 60mL of 1 M KCl solution to slowly wash the remaining solids.

The NH_4^+ concentration in the final extract was analysed using a Skalar auto-analyser (Skalar Analytical BV) based on the modified Berthelot reaction and with the use of specified reagents (buffer solution with potassium sodium tartrate and tri-sodium citrate, stock solution of NH_4Cl used for standard preparation and calibration, and solutions of sodium salicylate, sodium dichloroisocyanurate and sodium nitroprusside). Briefly, according to the Berthelot reaction, ammonia is chlorinated to monochloramine which reacts with salicylate to 5-aminosalicylate. After oxidation and oxidative coupling, a green coloured complex is formed when ammonia reacts with the Berthelot reagent in the presence of sodium hydroxide. The absorption of the formed complex is measured at a wavelength of 660 nm. The quality assurance protocol followed for this method is described in **Appendix D**.

The CEC value was calculated according to **Eq. 4.10**.

$$CEC \left(\frac{\text{mmol}}{\text{kg}} \right) = \frac{NH_4^+ \text{ concentration } \left(\frac{\text{mg}}{\text{L}} \right) \times V \text{ (mL)}}{NH_4^+ \text{ molecular mass } \left(\frac{\text{g}}{\text{mol}} \right) \times W \text{ (g)}} \quad (\text{Eq. 4. 10})$$

where V is the volume of the analysed extract (in this case, 100mL) and W is the mass of the dried biochar sample (g).

4.3.2.4 Plant-available phosphorus (formic acid extraction and Olsen-P methods)

Plant availability of phosphorus (P) was determined after overnight extraction by 2% formic acid (0.35 g oven dried biochar mixed with 35 mL formic acid solution) followed by ICP-OES analysis to measure the P concentration in the solution (Wang et al., 2012; IBI, 2015). The formic acid extraction method has been established as a standard method for P availability in biochar samples (Singh et al., 2017). Readily available P was also determined, using the Olsen method, by shaking 1 g of (dried) biochar with 20 mL NaHCO_3 (pH 8.5) for 30 min, followed by ICP-OES analysis of the solution (Olsen, 1954). Before analysis, all solutions were diluted with 1 M HNO_3 (within the calibration concentration range) and filtered using a 0.45 μm pore size WCN Whatman membrane.

4.3.2.5 X-ray diffraction (XRD)

The crystalline phases of biochar samples (oven dried) were analysed by X-ray diffraction (XRD) using a Brucker AXS D8 Advance system with a non-monochromated Cu K α radiation source, with an average $\lambda=1.504$ Å over a scanning range of 10–90 ° (2 θ range). Background subtraction and peak identification were performed using in-built software on the XRD analyser. The phase determination was performed using in-built High Score Plus X'Pert software methods (Malvern Panalytical) but restricted to elements identified via previously conducted quantitative analyses (ICP-OES, CHNS, XRF).

4.3.2.6 Scanning electron microscopy (SEM) and Energy-dispersive X-ray spectroscopy (EDX)

The surface morphology of the biochars was analysed under field-emission scanning electron microscopy (SEM), combined with Energy-dispersive X-ray spectroscopy (EDX) to identify the elemental composition of samples. Analysis was performed on a TM4000 Tabletop Microscope (Hitachi, Japan) fitted with an Oxford Instruments Aztec energy dispersive spectroscopy system, with an accelerating voltage of 10 kV.

4.3.2.7 X-ray fluorescence (XRF)

X-ray fluorescence (XRF) analysis was performed on a Malvern Panalytical Zetium XRF analyser, using the fused bead method as described in Waqas et al. (2018), to determine the metals present in biochar samples as oxides. Pre-weighed (oven dried) biochar samples were heated to 550 °C for 2 h and then 850 °C for 4 h to remove volatiles and convert all metals to metal oxides. The sample was then prepared by weighing 0.9 g of sample and 9 g of flux (lithium metaborate, LiBO₂) and melting the mixture at 1000 °C to form a glass disk, which was analysed by XRF. The obtained peaks were quantified using in-built software of the XRF analyser, to obtain the oxide composition normalised to 100%.

4.3.2.8 Brunauer, Emmett, and Teller (BET) surface area

The surface area of the biochars was measured using the Brunauer, Emmett, and Teller (BET) method on a Quantachrome Corp Autosorb iQ3 Gas Sorption Analyser (Anton Paar Ltd, Switzerland) (ISO, 2022). First, 0.4 g of (dried) biochar sample was placed in a glass sample cell and degassed under vacuum at 300 °C for 90 min to remove moisture and adsorbed gases from the sample surface. The sample was then re-weighed and loaded onto the analysis station, submerged in a bath of liquid nitrogen. The tube dead-space was measured by helium followed by nitrogen adsorption at the temperature of liquid nitrogen (~ -196 °C) over the pressure range of $P/P_0=0.05-0.3$, where P is the pressure of the adsorbate gas and P_0 is the saturation pressure of the gas at the analysis temperature. The specific surface area of the sample was calculated according to the BET method, using in-built software of the Gas Sorption Analyser (ISO, 2022).

4.3.3 Analyses of fresh urine samples

Fresh urine samples collected from volunteers were acidified with concentrated HNO_3 and stored at 4 °C before analysis. Where daily excretion quantities of elements are presented, these are based on the estimated daily urine production of 1.4 L/person/day reported by Rose et al. (2015).

P, K, Ca and Mg concentrations were analysed using ICP-OES (refer to **Section 4.3.1.5**) after dilution with 1 M HNO_3 (within the calibration range). The pH was measured with a Jenway 3540 combined pH and conductivity meter. $\text{NH}_4\text{-N}$ analysis was performed on a Skalar auto-analyser (Skalar Analytical BV) as described in **Section 4.3.2.3**. Total N (TN) content was measured as described in **Section 4.3.1.3** (via CHNS analysis) using samples of freeze-dried fresh urine.

4.3.4 Statistical analyses

All statistical analyses were conducted using in-built functions in MATLAB (MathWorks, 2023). Error ranges are reported as standard deviation values and, for linear regression models, as 95% confidence intervals. While previous research has reported that normality assumptions are violated for FS samples (Englund et al., 2020; Krueger et al., 2021a), this research investigates the characteristics of biochars prepared from pyrolysis of homogenised composite samples of human excreta (see **Sections 4.1** and **5.2.1**) or FS (**Section 8.2.4**), and hence the heterogeneity of individual samples does not participate in the assessment. The homogenisation process was validated by repeated measurements of homogenised samples to ensure a good repeatability of measurements. Experimental error was assumed to follow a normal distribution according to the central limit theorem (Montgomery, 2017). Where extreme outliers were observed, or for values presented for individual rather than composite samples (e.g. moisture content – **Figure 5.2**), the median is used as the best estimator rather than the arithmetic mean.

All statistical tests assume a normal distribution of random uncertainty, unless otherwise stated³. Analysis of variance (ANOVA) was used for the statistical comparison of the variance between and within groups of data, according to the assumptions of normality, homogeneity and independence. One-way ANOVA was used when one factor (or independent variable) was assessed, with two or more levels. Two-way ANOVA was used when two factors were assessed, with two or more levels each. The specific ANOVA parameters and objectives for each experiment are discussed in the “Materials and methods” sections of relevant chapters. For the analysis of individual FS samples in **Section 8.3.1**, the non-parametric Mann-Whitney U test (5% significance level) was used to assess significant differences between medians, as the normality assumption could not be confirmed³ (Krueger et al., 2021a).

4.3.5 Quality control

The detailed quality control procedure for each (quantitative) analytical method used in this thesis is described in **Appendix D**.

³ The normality assumption was tested using Q-Q plots and hypothesis testing (Lilliefors test).

5 Source separation of human excreta for improved resource recovery via pyrolysis⁴

5.1 Introduction

The investigation of literature knowledge highlighted the necessity to consider how source control can be better deployed in future sanitation systems to enable improved treatment and resource recovery efficiency, without assuming that current designs should be replicated. In fact, the impact of such work can be significant, as currently approximately 1.7 billion people have access to on-site sanitation that is not safely managed (WHO/UNICEF, 2023). In order to meet SDG 6 on “clean water and sanitation”, FSM services to accommodate these needs must be secured by 2030, creating an opportunity to inform the design of new sanitation systems to be focused on resource recovery. For this, the connections between source conditions, treatment efficiency and resource recovery should be understood, and source control strategies deployed systematically. Source separation of faeces and urine was identified as a promising strategy to maximise resource recovery via pyrolysis of human excreta.

This chapter presents a bench-scale experimental study to quantify the effect of source separation of human excreta on resource recovery via pyrolysis. First, the two excreta components (faeces and urine) were characterised, both separately and combined, while maintaining other influencing parameters of composition, apart from source separation, constant. This controlled experimental design differs from existing studies on FS pyrolysis, that use samples from on-site sanitation facilities (e.g. VIPs, UDDTs) with different toilet users, and hence are influenced by several intervening variables (see **Section 3.1.2**). As a second step, biochars produced from source-separated faeces (SSF) and mixed urine and faeces (MUF) at different pyrolysis temperatures (450, 550, 650 °C) were assessed for their resource recovery potential. This encompassed the characterisation of biochar samples for their thermal properties and elemental composition, and the assessment of the effect of pyrolysis temperature on biochar stability. The produced biochars were fully characterised for properties related to agricultural and solid fuel applications, including the examination of their crystalline composition and surface morphology.

While this research deployed pyrolysis as the treatment technology under investigation, presented findings can be used to inform other thermal treatment technologies that may benefit from urine diversion. At a higher level, this work can inform the design of new circular sanitation systems, taking all FSM components under consideration, from the source (i.e. toilet design) to the envisioned end-products.

⁴ This chapter contains work published by Koulouri et al. (2023), see **Section 1.5**.

5.2 Materials and methods

5.2.1 Human excreta sample preparation

Samples of source-separated faeces (SSF) and undiluted urine (SSU) were collected from volunteers according to the protocol described in **Section 4.1**. The SSF samples were dried to constant weight at 105 °C, and dry-sterilised at 150 °C for 150 min as suggested by Krueger et al. (2021a). Dry samples were mechanically mixed, homogenised and ground to <10 mm using pestle and mortar (see **Figure 5.1a**). Samples were combined into a composite SSF sample before pyrolysis experiments, both to increase sample representativeness (US EPA, 1995) and to ensure confidentiality of volunteers.

To prepare mixed samples of urine and faeces (MUF), urine samples were collected and stored at 4 °C for up to 1 week before being blended with raw faeces at a ratio of 1g:10mL (faeces:urine). This ratio is considered representative of the ratio of daily excretion (Rose et al., 2015) and that reported for on-site sanitation systems (Krounbi et al., 2019). Mixed samples were then dried, dry-sterilised, ground and combined into a homogenised composite MUF sample, following the same procedure as for SSF samples. Before analysis, all samples were manually ground and sieved to <2 mm (or smaller particle sizes) as described in **Section 4.3** (see **Figure 5.1b**).



Figure 5.1: Photographs of dried, dry-sterilised, homogenised samples of (a) source-separated faeces (SSF) (here the particle size is <10 mm, for pyrolysis experiments); and (b) mixed urine and faeces (MUF) (here the particle size is <2 mm, for analytical procedures).

5.2.2 Pyrolysis experiments

A 30 g sample aliquot was placed in the rotary laboratory-scale pyrolysis reactor (see **Section 4.2**) and N₂ was purged at a flowrate of 1.5 L/min to ensure an inert atmosphere is maintained. A preliminary assessment showed that a high gas flow rate is optimum for biochar production using this type of laboratory-scale reactor, to ensure that evolving gases are evacuated and do not condense on the inside of the furnace. The experiments were conducted for the two feedstocks (SSF, MUF) and at three prescribed temperatures (HHTs) (450, 550, 650 °C), using a heating rate of 10 °C/min and holding for 30 min at the required HHT.

These operational parameters were based on previous research for FS (Liu et al., 2014; Ward et al., 2014; Woldetsadik et al., 2017) and a preliminary thermogravimetric assessment for human faeces. The preliminary assessment was conducted on an STA 449 F5 Jupiter using ~15mg of sample (see **Section 4.3.1.1**) and was validated for bigger sample sizes (30 g) on the laboratory-scale pyrolysis reactor. Results showed no significant change in sample weight loss after 30 min retention time for all the HHTs tested (450, 550, 650 °C). Detailed results are included in **Appendix E**.

All experiments were performed in triplicate and average values are reported. The produced biochars are referred below as SSF450, SSF550, SSF650 and MUF450, MUF550, MUF650, based on the combination of feedstock type and pyrolysis temperature used.

5.2.3 Analytical methods and statistical analyses

For this chapter, the analytical methods presented in **Section 4.3** were used. Results were statistically analysed by two-way ANOVA (using MATLAB) to investigate the influence of source separation and pyrolysis temperature on biochar characteristics, at the 0.05 significance level. Multiple comparison tests were used to localise the impact of pyrolysis temperature, where a significant influence was detected.

5.3 Results and discussion

5.3.1 Human excreta characterisation

Feedstock characteristics are summarised in **Table 5.2** (on a dry weight basis). Proximate analysis showed higher content of fixed carbon for SSF compared to MUF (15.1% and 11.2% respectively) and the opposite trend for ash content (12.5% and 18.9% respectively). The higher ash content of MUF can be attributed to the presence of inorganic salts in urine (Rose et al., 2015). The measured calorific value is also higher for SSF compared to MUF (21.8 MJ/kg compared to 17.8 MJ/kg) showing that source-separated faeces are a more energy-dense feedstock. MUF are higher in N (6.8% compared to 4.9% for SSF) as well as P and K, which is expected, as most of the nutrients excreted from the human body are found in urine (Larsen et al., 2013). As these results are expressed on a dry weight basis, N volatilisation during thermal drying is expected for both feedstocks. The Ca content is relatively higher for SSF, and Mg is almost equally distributed between the two excreta streams. Nutrient concentrations in fresh urine are included in **Table 5.1**.

The urine fraction also increases the volume and moisture of mixed excreta samples, often to >95% initial moisture content (MC), which has been previously defined as the threshold to avoid solid-liquid separation requirements before dewatering (Tayler, 2018; Gold et al., 2018b). In contrast, all SSF samples had MC<90% (median value 78.2%) (**Figure 5.2**) and more than a quarter of the fresh faeces

samples had MC values below 75%, which is the mechanical dewatering limit (Tayler, 2018; Krueger et al., 2021a). The evaporation during short-term storage (for example in CBS toilets) and the addition of cover materials such as wood, would also further reduce the MC, suggesting that achieving values below 75% systematically is feasible for urine-diverting dry toilets. Source separation can therefore be a key strategy to reduce the need for mechanical dewatering before pyrolysis, which largely influences the energy efficiency of FS pyrolysis systems (Bond et al., 2018). Solar drying can be a feasible option for SSF feedstocks, although the process efficiency is case-specific and can be dependent on the geographical location and local climate conditions (Liu et al., 2014).

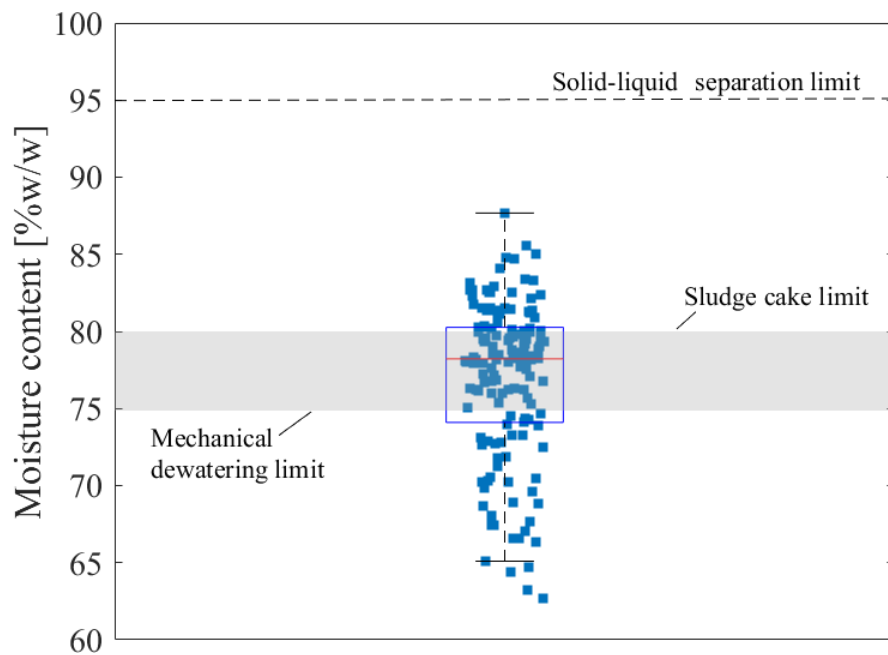


Figure 5.2: Moisture content (%w/w) of fresh source-separated faeces (SSF) samples (n=150) collected for this research project from 12 volunteers in the UK (see **Section 4.1**). The figure includes the solid-liquid separation limit (95%), sludge cake limit (80%) and mechanical dewatering limit (75%) as defined in Tayler (2018) and Krueger et al. (2021a).

Table 5.1: Nutrient concentrations in fresh urine expressed as average values (g/L), range of values ([min-max] g/L) and daily excretion quantities (g/person/day) based on the estimated daily urine production of 1.4 L/person/day reported by Rose et al. (2015).

	TN	P	K	Ca	Mg
g/L	8.13	0.86	1.01	0.10	0.10
[min-max] g/L	[5-11]	[0.6-1.0]	[0.9-1.1]	[0.08-0.14]	[0.09-0.12]
g/person/day	11.38	1.21	1.42	0.15	0.14

Table 5.2: Physical and chemical characterisation (on a dry weight basis) of biochars produced from source-separated faeces (SSF) and mixed human excreta (MUF) at different pyrolysis temperatures (450, 550, 650 °C), and their corresponding feedstocks. Mean values presented from analyses performed in triplicate (standard deviation in brackets).

Parameter	Unit	Feedstocks		SSF biochars			MUF biochars			Guidelines (max. allowed values)		
		SSF	MUF	450°C	550°C	650°C	450°C	550°C	650°C	(EBC, 2012)	(IBI, 2015)	(WBC, 2023)
Char yield	[%]			34.4 (±0.5)	31.1 (±0.5)	29.4 (±0.2)	45.5 (±0.9)	41.7 (±0.9)	38.5 (±0.6)			
VM	[%]	72.5 ±0.6	70 (±0.2)	24.4 (±1.7)	14.8 (±0.3)	12.9 (±1.5)	27.0 (±0.1)	18.8 (±1.2)	15.0 (±0.3)			
FC	[%]	15.1 ±0.3	11.2 (±0.3)	43.0 (±0.7)	50.5 (±0.6)	50.1 (±0.7)	22.9 (±0.6)	26.4 (±0.7)	28.7 (±0.3)			
Ash	[%]	12.5(±0.4)	18.9 (±0.5)	32.6 (±0.7)	34.7 (±0.6)	37.0 (±0.3)	50.1 (±0.6)	54.8 (±0.5)	56.3 (±0.2)			
HHV	[MJ/kg]	21.8(±0.1)	17.8 (±0.0)	20.3 (±0.1)	17.9 (±0.4)	17.7 (±0.3)	13.3 (±0.3)	12.3 (±0.5)	12.3 (±0.1)			
C	[%]	50 (±0.4)	41 (±0.8)	50.9 (±0.6)	50.4 (±0.3)	48.8 (±0.6)	33.1 (±0.6)	32.2 (±0.9)	32.2 (±0.4)			
H	[%]	6.8 (±0.2)	5.3 (±0.2)	3.3 (±0.0)	1.8 (±0.0)	1.3 (±0.0)	1.9 (±0.1)	1.1 (±0.0)	0.8 (±0.0)			
N	[%]	4.9 (±0.2)	6.8 (±0.3)	4.7 (±0.0)	4.5 (±0.1)	4.3 (±0.1)	4.9 (±0.0)	4.4 (±0.1)	4.0 (±0.1)			
S	[%]	1.3 (±0.1)	2.1 (±0.4)	1.2 (±0.0)	<0.1	<0.1	2.0 (±0.1)	1.4 (±0.3)	0.8 (±0.0)			
H/C ratio	-			0.77	0.43	0.32	0.68	0.41	0.30			
pH	-			9.6 (±0.0)	10.4 (±0.1)	11.2 (±0.1)	9.2 (±0.1)	10.4 (±0.0)	10.6 (±0.1)			
EC	[mS/cm]			2.7 (±0.2)	3.5 (±0.1)	4.9 (±0.2)	28.8 (±0.8)	29.2 (±0.4)	28.8 (±0.4)			
CaCO ₃ -eq.	[%]			12.2 (±0.3)	13.2 (±0.4)	13.0 (±0.3)	5.3 (±0.2)	8.2 (±0.4)	11.4 (±0.5)			
CEC	[cmol/kg]			69.6 (±0.7)	40.1 (±1.0)	24.8 (±1.1)	73.8 (±0.6)	47.8 (±1.0)	46.0 (±3.0)			
BET	[m ² /g]			1.3 (±0.1)	2.4 (±0.1)	3.1 (±0.3)	8.2 (±0.2)	10.9 (±0.2)	19.6 (±0.3)			
P	[g/kg]	22.4 (±0.1)	32.2 (±0.5)	50.9 (±0.0)	55.1 (±1.7)	58.3 (±1.4)	58.7 (±0.9)	64.4 (±0.0)	66.8 (±2.7)			
P (2% FA)	[g/kg]			49.1 (±1.1)	53.0 (±1.6)	50.3 (±1.0)	56.0 (±0.3)	61.7 (±0.1)	62.5 (±0.1)			
	[% P]			96.6	96.1	86.3	95.5	95.8	93.6			
Olsen-P	[g/kg]			3.8 (±0.3)	3.8 (±0.4)	5.0 (±0.5)	5.7 (±0.3)	14.4 (±1.4)	16.4 (±2.4)			
	[% P]			7.5	6.9	8.6	9.7	22.4	24.6			
K	[g/kg]	12.9 (±0.1)	28.5 (±0.2)	45.3 (±1.4)	48.6 (±1.5)	51.9 (±1.1)	74.5 (±0.7)	84.8 (±1.9)	84.8 (±0.2)			
Ca	[g/kg]	23.2 (±0.1)	17.7 (±0.1)	53.9 (±1.3)	59.4 (±2.2)	63.9 (±2.0)	40.3 (±1.1)	41.4 (±0.7)	43.2 (±1.4)			
Mg	[g/kg]	8.8 (±0.1)	8.7 (±0.1)	20.6 (±0.3)	22.9 (±0.5)	24.6 (±1.3)	16.8 (±0.1)	18.4 (±1.3)	19.4 (±1.7)			
B	[mg/kg]	9.6 (±2.6)	17.3 (±4.8)	34.6 (±1.6)	34.1 (±0.1)	39.6 (±3.9)	83.2 (±0.1)	83.6 (±3.6)	86.8 (±0.8)			
Zn	[mg/kg]	195.5(±11.3)	145(±11.4)	443.5(±31.5)	449.4(±14.4)	535.0(±41.0)	245.3 (±2.0)	260.5 (±5.6)	263.1 (±3.6)	400	416-7400	420-1000
Mn	[mg/kg]	109 (±5.2)	82.3 (±8.1)	318.9(±10.4)	322.0(±24.7)	353.7(±17.0)	188.9(±5.5)	202.3 (±8.6)	205.7(±18.3)			
Fe	[mg/kg]	236.8(±10.1)	182(±23.4)	739.3(±20.1)	752.8(±23.9)	792.6(±38.2)	492.3(±42.9)	462.7(±12.1)	468.5(±10.2)			
Cu	[mg/kg]	82.4 (±2.4)	69.2 (±3.2)	120.4 (±3.1)	122.3 (±1.1)	135.7 (±6.1)	81.7 (±1.8)	88.7 (±0.4)	90.9 (±0.1)	100	143-6000	140-200
Ba	[mg/kg]	<10	<1	25 (±3.3)	24.7 (±2.0)	27.8 (±2.2)	4.8 (±0.5)	6.2 (±1.7)	3.8 (±1.2)			
Cd	[mg/kg]	<1	<1	<1	<1	<1	<1	<1	<1	1-1.5	1.4-39	1.5-5
Co	[mg/kg]	<1	<1	<1	<1	<1	<1	<1	<1		34-100	
Cr	[mg/kg]	<1	<1	<1	<1	<1	<1	<1	<1	80-90	93-1200	100-200
Pb	[mg/kg]	<10	<1	<10	<10	<10	<1	<1	<1	120-150	121-300	120-300

VM = Volatile matter, FC = Fixed carbon, HHV = Higher heating value, EC = Electrical conductivity, CaCO₃-eq. = Calcium carbonate equivalence, CEC = Cation exchange capacity, P (2% FA) = 2% formic acid extractable P, EBC = European Biochar

Certificate, IBI = International Biochar Initiative, WBC = World Biochar Certificate

Further structural differences between the two sample groups (SSF, MUF) were investigated by FTIR spectroscopy (refer to **Section 4.3.1.4**). FTIR has been suggested as a quick method to measure faecal fat content, mainly by identifying regions of fatty acids (De Koninck et al., 2016). Typical peaks of aliphatic C-H bonds appearing at 2920 cm^{-1} and 2850 cm^{-1} are almost identical for SSF and MUF (**Figure 5.3**), as the fat content in human excreta sludges is attributed to the faecal stream. A distinct shoulder peak also appears for SSF at $\sim 1745\text{ cm}^{-1}$ which can be related to the presence of lipids (C=O stretching). Additional variations observed within the $1750\text{--}1550\text{ cm}^{-1}$ wavenumber range can be attributed to the difference in protein structure when urine is present, as amide and peptide bonds form strong peaks within this region (Haris & Severcan, 1999). Both sample groups show a broad peak at $3700\text{--}3100\text{ cm}^{-1}$ (O-H stretching) and a strong peak at $1200\text{--}950\text{ cm}^{-1}$ (C-O and C-C stretching, linked to carbohydrates) which have previously been observed for FS (Krueger et al., 2021b). The latter peak at $1200\text{--}950\text{ cm}^{-1}$, as well as the peak forming around 500 cm^{-1} have also been attributed to the presence of phosphates (Jastrzębski et al., 2011; Shapaval et al., 2019).

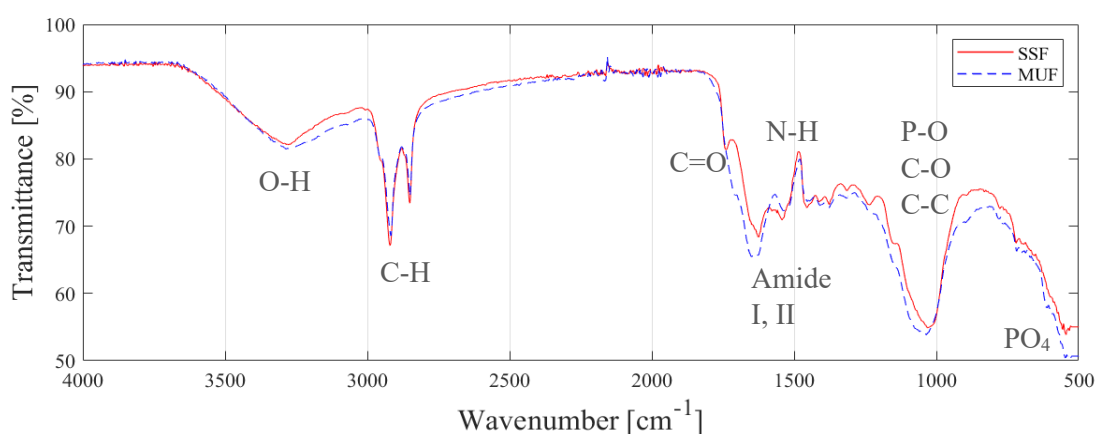


Figure 5.3: Fourier-transform infrared spectroscopy (FTIR) spectra for source-separated faeces (SSF) and mixed human excreta (MUF), in the $4000\text{--}500\text{ cm}^{-1}$ wavenumber range.

Thermal analysis results (**Figure 5.4**) show the differences in decomposition behaviour when urine is present, compared to SSF samples. Three thermal decomposition stages are observed within the typical pyrolysis temperature range ($200\text{--}700\text{ }^{\circ}\text{C}$) for both sample groups. Similar stages have been observed by previous studies for human excreta (Somorin et al., 2020b) and FS (Krueger et al., 2021b). This analysis provides a deeper understanding of how source separation impacts decomposition behaviour.

The three identified stages in **Figure 5.4** are as follows:

1) **200-400 °C**

Rapid weight loss (>50% of initial weight) mainly attributed to the decomposition of protein, hemicellulose, cellulose and other carbohydrates (Krueger et al., 2021b). The maximum weight loss rate peak is observed at 310 °C for SSF, while for MUF it is observed at 320 °C and preceded by a smaller peak between 240-290 °C. This different pattern can be attributed to the decomposition of urea-based nitrogenous compounds in MUF (Jones & Rollinson, 2013; Zhu et al., 2021).

2) **400-550 °C**

Weight loss within this temperature range has been attributed to the completion of lignin decomposition reactions and to the cracking of oil and grease (Krueger et al., 2021b). A distinct shoulder is observed on the DTG curves, occurring at around 450 °C until the completion of main pyrolytic reactions by 550 °C. Similar findings have been reported by Fidalgo et al. (2019) for human faeces. Here, it can be further observed that this shoulder is more distinct for SSF compared to mixed excreta, due to the excretion of dietary fat through the faecal stream.

3) **>550 °C**

During the final stage, and for temperatures >550 °C, gradual weight loss occurs due to continued carbonisation. At temperatures higher than 700 °C further weight loss occurs, which can be attributed to the decomposition of inorganic compounds, including carbonates (Kwon et al., 2018). Notably, this occurs at a higher rate for urine-containing excreta sources, which can be attributed to the high presence of inorganic salts in urine (Rose et al., 2015).

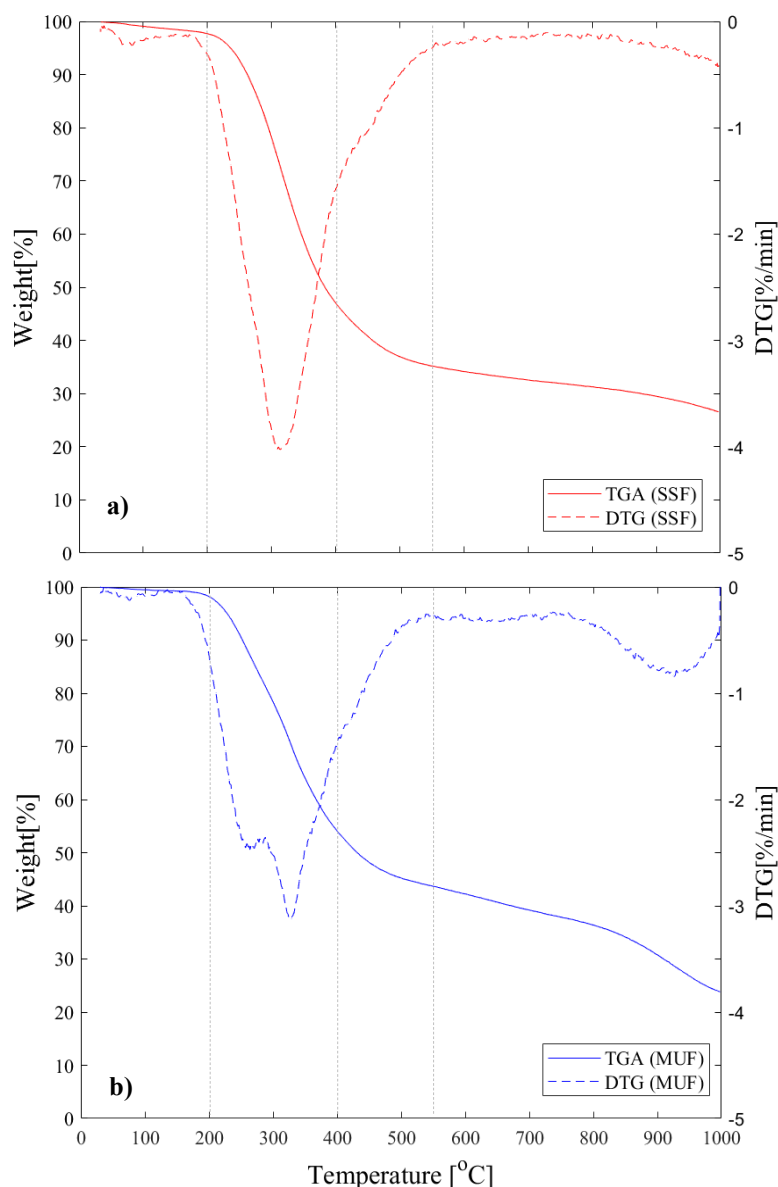


Figure 5.4: Thermogravimetric analysis (TGA) (weight loss expressed as % w/w) and derivative thermogravimetry (DTG) curves for source-separated faeces (SSF) (a) and mixed human excreta (MUF) (b), showing the thermal decomposition behaviour of the samples (up to 1000 °C, 10 °C/min, 50 mL N₂/min).

5.3.2 Biochar stability

According to the International Biochar Initiative (IBI), the World Biochar Certificate (WBC) and the European Biochar Certificate (EBC) guidelines, the H/C molar ratio can be used as an indicator of biochar stability. Biochars with $H/C < 0.7$ are recommended as stable, as this indicates the prevalence of fused aromatic ring structures, while $H/C > 0.7$ indicates that pyrolytic reactions have not been completed (EBC, 2012; IBI, 2015; WBC, 2023). This requirement is met for excreta-derived biochars produced at 550 °C or higher temperatures, while at 450 °C the H/C ratio is 0.8 for SSF and 0.7 for MUF (**Table 5.2**). Previous studies that tested FS-derived chars produced at 500 °C reported $H/C < 0.7$, indicating complete pyrolytic reactions at this temperature (Liu et al., 2014; Krounbi et al., 2018). Therefore, biochar stability during human excreta pyrolysis is expected to be achieved between 450-500 °C.

These findings were confirmed by FTIR analysis (**Figure 5.5**). Notably, some vibrations at 3000-2800 cm^{-1} , which are linked to aliphatic C-H bonds present in the feedstock materials, were still observed for biochars produced at 450 °C, particularly for SSF which had the highest H/C ratio at this temperature. These aliphatic structures have been suggested as an indication of the level of decomposition of organic wastes (Smidt & Schwanninger, 2005). Similar results for sewage sludge-derived chars have shown that these peaks disappear at temperatures ≥ 500 °C (Ren et al., 2018).

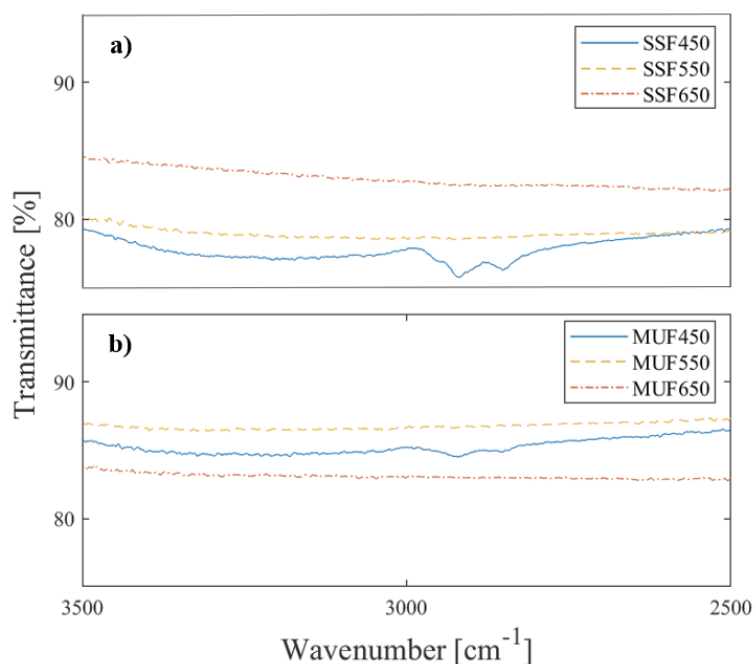


Figure 5.5: Fourier-transform infrared spectroscopy (FTIR) spectra for source-separated faeces (SSF) (a) and mixed urine and faeces (MUF) (b) biochars in the 3500-2500 cm^{-1} wavenumber range, as an indication of the level of thermal decomposition achieved during treatment.

Therefore, temperatures ≥ 500 °C are recommended for human excreta pyrolysis applications, particularly those involving environmental exposure (e.g. use of the biochar in agriculture). Further optimisation should be performed on a case-specific basis, as the optimum temperature would be influenced by several factors, including feedstock mass and furnace size.

5.3.3 Biochar characterisation

Biochar characteristics are summarised in **Table 5.2**. ANOVA results (**Table A7** – Appendix F) showed that source separation has a significant ($p < 0.05$) effect on all tested parameters. The char yield is higher for MUF compared to SSF for all pyrolysis temperatures, although this increase can be attributed to the higher ash content in MUF. Both types of biochar showed a similar volatile matter decrease pattern for the temperatures tested (no significant interaction effects observed between source separation and temperature by ANOVA). The relatively high residual volatile matter content of these biochars ($> 10\%$), even after preparation at high pyrolysis temperatures (≥ 550 °C), can be attributed to the method followed

for volatile matter determination (at 950 °C). As discussed for the presented TGA curves (**Figure 5.4**), a second weight loss peak occurs for both feedstocks at high temperatures (>700 °C) due to the decomposition of inorganic compounds. Therefore, an overestimation of the volatile matter can occur for excreta-based feedstocks when using standard method ASTM D7582-15 for proximate analysis (see **Section 4.3.1.1**) and should be taken into consideration when interpreting results.

5.3.3.1 Value as a soil amendment

The measured pH values (>9) show that all the prepared biochars were alkaline. The pH increased alongside the ash content with higher pyrolysis temperatures, similar to previous findings for FS (Liu et al., 2014; Krounbi et al., 2018). A quantifiable parameter often used in agriculture relating to the ability to treat acidified soils is the biochars' liming potential, expressed as CaCO₃ equivalence (Ippolito et al., 2015). In this aspect, a difference is observed between biochar types, with SSF having significantly higher liming potential (p-values in **Table A7** – Appendix F) despite pH values being similar, particularly at temperatures up to 550 °C. Moreover, the electrical conductivity of MUF biochars is up to 10 times higher than SSF biochars (**Table 5.2**); this is indicative of the large ion concentration present in urine. These ions include saline-causing species, as confirmed by XRD analysis (refer to **Figure 5.7**), and should be taken into consideration particularly for salt sensitive plants that may be negatively affected by high salinity in applied biochars (Bleuler et al., 2020).

Cation exchange capacity (CEC) is also a crucial parameter for soil amendments as it is a measure of a soil's ability to adsorb and retain nutrients in exchangeable forms that are available for plant uptake (Chapman, 1965). CEC was found to decrease with increasing temperature for both sample groups, particularly for SSF, suggesting that lower pyrolysis temperatures (≤500 °C) may be required for improvement of a soil's nutrient-holding capacity from human excreta-derived biochars (**Table 5.2**). The same trend has been reported for CBS sludge (Krounbi et al., 2019), while the opposite trend was observed by Gold et al. (2018) for FS from septic tanks and pit latrines, where additional waste inputs are often added and may deviate from the behaviour of human excreta. Gold et al. (2018) also noted the inconsistent influence of pyrolysis temperature on CEC for different types of chars.

The fact that the method used measures CEC at pH 7 needs to be taken under consideration when interpreting these results, as pH-dependent variable charges can influence CEC measurements (Munera-Echeverri et al., 2018). The ammonium acetate extraction method at pH 7 used in this study is a common standard for comparison between laboratories, however, the high pH and liming potential of the produced biochars might affect their nutrient exchange behaviour when applied to different soils, as generally CEC has been found to increase with increasing pH (Singh et al., 2017).

Further information, on the comparative nutrient recovery potential and heavy metal content of SSF and MUF biochars, is presented in the following sections.

5.3.3.1.1 Nutrient recovery

Generally, P and K are concentrated in biochars, with MUF providing higher PK concentrations (**Table 5.2**), as expected based on the daily excretion ratios of these elements between faeces and urine (Rose et al., 2015). For plant-available P, formic acid extraction is the established method for biochars, due to its ability to represent the acidic microenvironment produced by plant roots that extracts more nutrients from the soil (Singh et al., 2017; Bleuler et al., 2020). The 2% formic acid extractable P appears to be very high for biochars produced at temperatures up to 550 °C but starts declining at 650 °C (**Table 5.2**), suggesting that temperatures around 500-550 °C would be best for P availability in excreta-based biochars. These results are in line with previous studies that suggest temperatures <600 °C when producing biochars for use in agriculture (Glaser & Lehr, 2019).

The Olsen-P method for soil P availability was also tested, as an indication of the readily available inorganic P fraction (FAO, 2021). Results are significantly lower compared to the formic acid extraction method, suggesting that the biochars can be used as slow-release fertilisers and only a small fraction of the P will be readily available (C. Wang et al., 2022). Although slow-release fertilisers may not be sufficient for highly nutrient-deficient soils or plants with very high P demand and short growing cycles, the lower readily available P fraction may be ideal for longer or repeated growing cycles and for environments where nutrient loss through leaching occurs at a higher rate (e.g. wet climates) (Elsworth & Paley, 2009). Therefore, the suitability of biochar fertilisers can only be examined on a case-specific basis. A method to increase the readily available P fraction of SSF biochars via precipitation of P-containing compounds from stored urine is investigated in **Chapter 6**.

Notably, the Olsen-P fraction increased with higher pyrolysis temperatures, in contrast to the formic acid extractable P fraction. This can be expected as the Olsen-P method has been designed primarily to extract readily available inorganic forms of P, which concentrate in the ash fraction during pyrolysis (FAO, 2021). Nevertheless, its suitability for biochar analysis is questionable as it can severely underestimate sorbed or chemically bound forms of P (e.g. calcium phosphates) that may be available in field conditions (e.g. depending on the pH). Using multiple extraction methods to account for varying objectives, soil properties and plant needs is therefore recommended. In any case, environmental factors, such as temperature and soil pH need to be considered to determine the actual availability of nutrients via plant uptake measurements (Krounbi et al., 2018).

In terms of N, expressing the feedstock and biochar N content results shown in **Table 5.1** and **Table 5.2** as g/person/day (based on daily excretion ratios presented in Rose et al. (2015)) allows the estimation of the N daily excretion (**Table 5.3**) and N retention for two scenarios: a) a source-separating system, with SSF550 biochar and separated urine (SSU) as its products (**Table 5.4**), and b) a mixed-streams system with MUF550 biochar as its product (**Table 5.5**). It is calculated that for scenario (a) only 3.2% of the total N excreted is retained in the SSF550 biochar and 80% can be retained in SSU, assuming up

to 10% volatilisation losses during SSU transport in closed containers (refer to **Section 6.3.1**) (Udert et al., 2006). For scenario (b) MUF550 contains only 12.6% of the total N excreted, due to volatilisation losses. Therefore, around 70% more N (% of total) is retained and available for recovery through a source-separating system, compared to a mixed-streams scenario.

As for Ca and Mg, these elements are concentrated in the biochar as the pyrolysis temperature increases, but are higher for SSF than MUF biochars, in line with excretion ratios and their concentrations in the feedstocks (**Table 5.2**).

Table 5.3: Total daily N excretion (g/person/day) via faeces and urine (on a dry and wet basis respectively).

	Faeces (dry)	Urine (wet)
Daily excretion ¹	29 g/person/day	1.4 L/person/day
N	4.90%	8.13 g/L
Daily N excretion	1.42 g/person/day	11.38 g/person/day
Total:	12.8	g/person/day

¹ (Rose et al., 2015).

Table 5.4: N retention (%) for the source separation scenario (source-separated faeces [SSF] and urine [SSU]) with SSF550 biochar and SSU as its products.

	SSF550	SSU
N	4.50%	8.13 g/L
Char yield	31.10%	-
Daily excretion		
Faeces (dry) ¹	29 g/person/day	-
Urine (wet) ¹	-	1.4 L/person/day
N available for recovery		
N	0.41 g/person/day	11.38 g/person/day
Losses ²	-	10%
N available for recovery	0.41 g/person/day	10.24 g/person/day
% of total N excreted:	3.2%	80.0%
	83.2%	

¹ (Rose et al., 2015); ² Assuming up to 10% volatilisation losses during SSU transport in closed containers (refer to **Section 6.3.1**) (Udert et al., 2006).

Table 5.5: N retention (%) for a mixed urine and faeces (MUF) scenario with MUF550 biochar as its product.

	MUF550
N	4.4%
Char yield	41.7%
Daily excretion	
Faeces (dry) ¹	29 g/person/day
Urine (dry) ¹	59 g/person/day
N available for recovery	
N	1.61 g/person/day
% of total N excreted:	12.6%

¹ (Rose et al., 2015).

In order to qualitatively investigate the inorganic compounds present, the ash fractions of both biochar types were analysed via FTIR (after ashing at 600 °C for 4 h). Results (**Figure 5.6**) show a band peak at 1040-1010 cm⁻¹ and another at 650-550 cm⁻¹, which can be attributed to phosphate compounds, including potassium phosphates and calcium phosphates (apatites) (Jastrzębski et al., 2011), as further confirmed by XRD analysis (**Figure 5.7**). For SSF, the bands around 1040 cm⁻¹ are forming a more intense peak and there are additional peaks at 1550-1350 cm⁻¹ and ~870 cm⁻¹, which are characteristic of carbonates (Jones & Jackson, 1993) and carbonated apatites (Rehman & Bonfield, 1997; Stanislavov et al., 2018). Calcium carbonate can be formed spontaneously in biochars when CaO or Ca(OH)₂ (formed when CaO is hydrated) react with CO₂ (also refer to **Section 7.3.2.2**) (Galván-Ruiz et al., 2009). The prevalence of carbonates or carbonated apatite in SSF biochars can explain the higher liming potential observed for these biochars and shows their promising soil improvement potential.

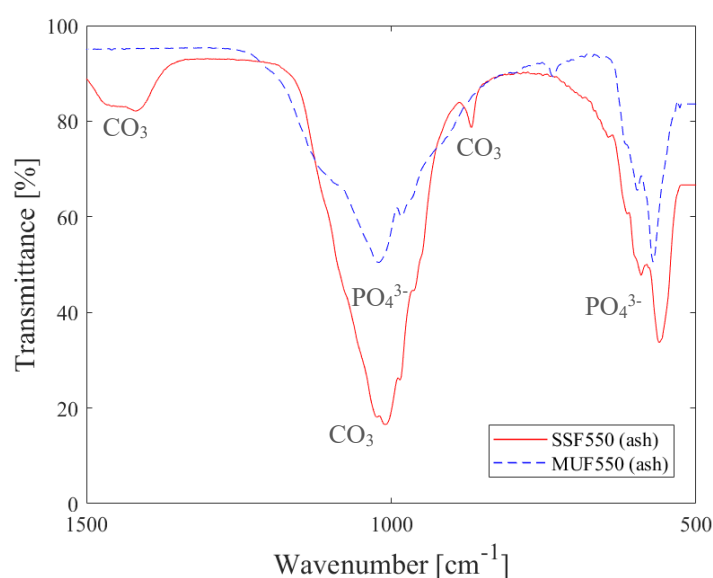


Figure 5.6: Fourier-transform infrared spectroscopy (FTIR) spectra for the inorganic fraction of source-separated faeces (SSF) and mixed urine and faeces (MUF) biochars (produced at 550°C), in the 1500-500 cm⁻¹ wavenumber range.

XRD analysis (refer to **Section 4.3.2.5**) confirmed the heterogeneity of the produced biochars, and the observed peak positions (2θ) were identified as the fingerprint of the crystal phases present (**Figure 5.7**). Both biochar samples are characterised by a broad background diffraction pattern indicative of amorphous silica (Singh et al., 2017), while the broad peaks observed for SSF550 ($2\theta < 40^\circ$) indicate the stacking structure of aromatic layers in the biochar and validate the carbon-rich nature of the sample (Takagi et al., 2004). MUF550 biochar shows mostly distinctive, sharp peaks due to the prevalence of inorganic compounds.

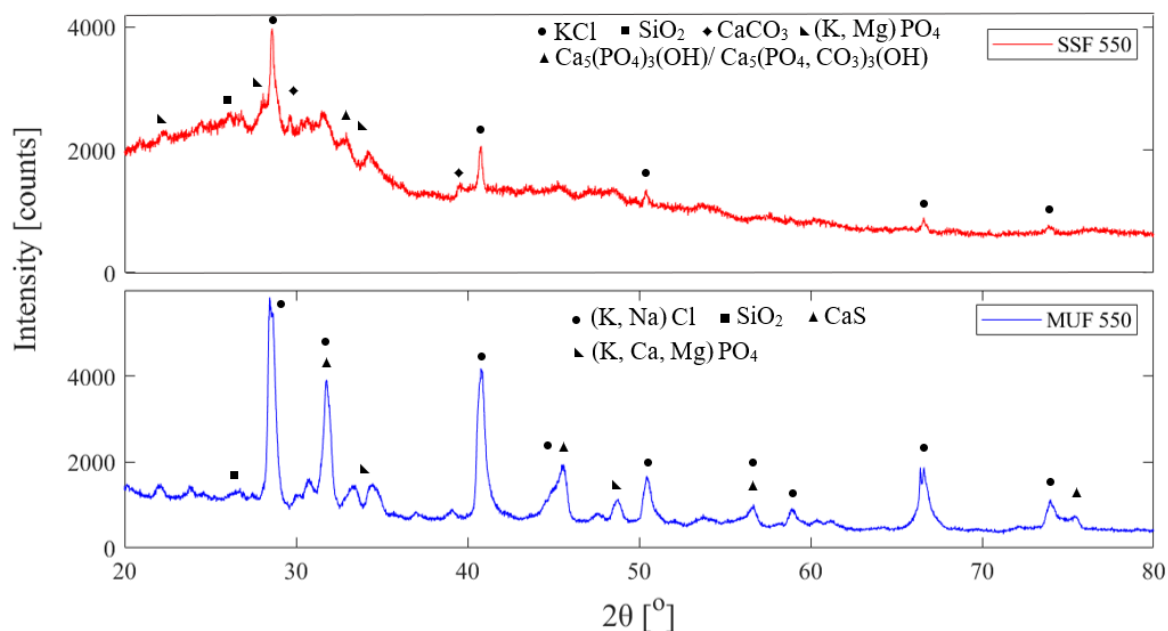


Figure 5.7: X-ray diffraction (XRD) patterns for source-separated faeces (SSF) and mixed urine and faeces (MUF) biochars (produced at 550°C), showing the crystalline structure of the samples ($2\theta = 20\text{--}80^\circ$).

Sharp diffraction peaks observed for both samples match the pattern of KCl salts (Ismail et al., 2022) and are more intense for MUF compared to SSF biochars. The presence of NaCl salts is also observed for MUF550, confirming the stronger salinity of mixed excreta biochars, which may place limitations on their use in agriculture (Bleuler et al., 2020). Similar diffraction peaks have been attributed to the formation of calcium sulphide crystals, suggesting that the S present in MUF biochars may bound Ca ions (Raubach et al., 2014). Both samples included peaks attributed to the presence of potassium, magnesium and calcium phosphates. The high concentration of P in both samples (**Table 5.2**) suggests that amorphous phosphorous compounds are also present in the biochars (Figueiredo et al., 2021).

For SSF550, XRD results confirmed the occurrence of hydroxyapatite (HAP) ($\text{Ca}_5(\text{PO}_4)_3(\text{OH})$) and calcite crystals (CaCO_3), validating FTIR findings on the nature of inorganic compounds present and providing further evidence on the fertiliser value and liming ability of SSF biochars. Overall, FTIR analysis coupled with XRD validation is suggested as a quick and reliable method to confirm the chemical composition, both amorphous and crystalline, of human excreta biochars.

5.3.3.1.2 Presence of heavy metals

As shown in **Table 5.2**, trace elements concentrations meet international standards, while the stricter EBC thresholds are exceeded for zinc (Zn) and copper (Cu) concentrations in SSF biochars (EBC, 2012; IBI, 2015; WBC, 2023). The concentrations of cadmium (Cd), cobalt (Co), chromium (Cr) and lead (Pb) were below the detection limit (the absence of these elements was validated via XRF analysis, see **Table A5** – Appendix D) (Byers et al., 2018).

Multiple comparison test p-values (**Table A7** – Appendix F) showed significant increase in trace elements concentrations at the 550-650 °C temperature range, suggesting that temperatures below 550 °C can limit heavy metal concentration in the final products. Nevertheless, heavy metal bioavailability and mobility are expected to be reduced in biochars compared to feedstocks due to changes in their chemical forms during pyrolysis (e.g. complexation, formation of insoluble oxides, sorption to the biochar surface) (Hossain et al., 2011; Woldetsadik et al., 2017). For full-scale applications, it is expected that human excreta will be mixed with other biomass sources, either as cover materials during containment in toilets or to enhance drying during pre-treatment (e.g. sawdust). Therefore, total heavy metal concentrations are expected to be diluted and should be compared to maximum allowed threshold values in a case-specific context, considering potential country-specific regulations for the intended use(s) of recovered products (see **Section 8.3.1.2**).

5.3.3.2 Value as a solid fuel

The demand for sustainable alternatives to wood-derived solid fuels is intensified by climate change, deforestation and energy challenges, and can be (partially) met through waste biomass valorisation (Lohri et al., 2016). Nevertheless, FS-based solid fuels often have relatively low calorific values and cannot easily sustain the production of marketable briquettes that are competitive against traditional charcoal fuels, particularly for high-ash FS sources (Kiwana & Naluwagga, 2016; Gold et al., 2018a). Source separation may be a method to create more energy-dense solid fuels from human excreta, along with the addition of biomass, such as sawdust or agricultural waste, which has been previously discussed in the literature as a strategy to increase fuel calorific value and dilute the ash, N and S content (Hafford et al., 2018; Hübner et al., 2019). It is evident that SSF biochars have significantly higher calorific values compared to MUF, notably 20.3 MJ/kg for SSF450 compared to 13.3 MJ/kg for MUF450, which corresponds to around 50% increase in calorific value density with source separation. Moreover, source separation allows for the combination of resource recovery objectives, as the urine stream would remain available for nutrient recovery.

For both SSF and MUF biochars, HHVs are lower after pyrolysis and further decrease with an increase of pyrolysis temperature from 450 °C to 550 °C (**Table 5.2**). No significant change in calorific value is noticed between 550 °C and 650 °C. The pattern of calorific value decreasing with increasing temperature has also been noticed for faeces by Ward et al. (2014) and for FS by Gold et al. (2018).

Therefore, within the studied range, temperatures <550 °C are deemed more suitable for the production of excreta-based solid fuels, although this might change with the addition of other biomass sources in the feedstock mix. Lower temperatures (e.g. 350-450 °C) can be considered when the production of solid fuels is the main objective, although char stability is unlikely to be established. In any case, the volatile matter content of char-based solid fuels should be monitored to ensure emissions during combustion are minimised (Falemara et al., 2018).

The lower S concentrations in SSF550 and SSF650 (**Table 5.2**) meet guideline values for the unproblematic combustion of solid fuels in terms of SO_x emissions (<0.2% S content), although these are not met for SSF450 or any of the MUF biochars (Oberberger et al., 2006). Both types of biochars have higher N content than recommended values (<2.5-0.6%) which requires caution over NO_x emissions when burning excreta-based solid fuels (Gold et al., 2017). Therefore, the combination of excreta with other sources of biomass that have lower N and S concentrations compared to FS (e.g. wood), is essential not only to boost the final products' calorific value but also to limit combustion emissions (Hafford et al., 2018; Ippolito et al., 2020). When excreta-based chars are used to produce briquettes, it is recommended that the final products are tested for their composition and fuel characteristics, to ensure emissions and burning efficiency are optimised.

5.3.3.3 Surface morphology

SEM images of SSF and MUF biochars are shown in **Figure 5.8**. A range of formations with different surface morphology can be observed. Notably **Figure 5.8b** captures typical organic structures, as well as crystal-like shapes and porous mineral formations. Some crystal formations appear to be incorporated into the carbon matrix for SSF biochars, which was not observed for MUF (**Figure 5.8a, d**). Interestingly, the surface of MUF biochars was more porous compared to SSF (**Figure 5.8e, f**), as quantitatively validated via BET surface area analysis (**Table 5.2**). MUF650 also presents a more developed pore structure compared to MUF550, which is in line with literature findings of biochar pore volume and surface area increasing with pyrolysis temperature (**Figure 5.8d, e, f**) (Ma et al., 2016; Nkomo et al., 2021). SSF biochars present a more sheet-like texture, with macroporous cavities which can be attributed to the decomposition of the faeces' organic structure (**Figure 5.8c**) (Wildman & Derbyshire, 1991). Both types of biochar appear to have minerals present both on the surface as well as embedded in the carbon matrix, as indicated by the compositional contrast of the images, whereby minerals that have a higher atomic number than carbon appear brighter (Singh et al., 2017).

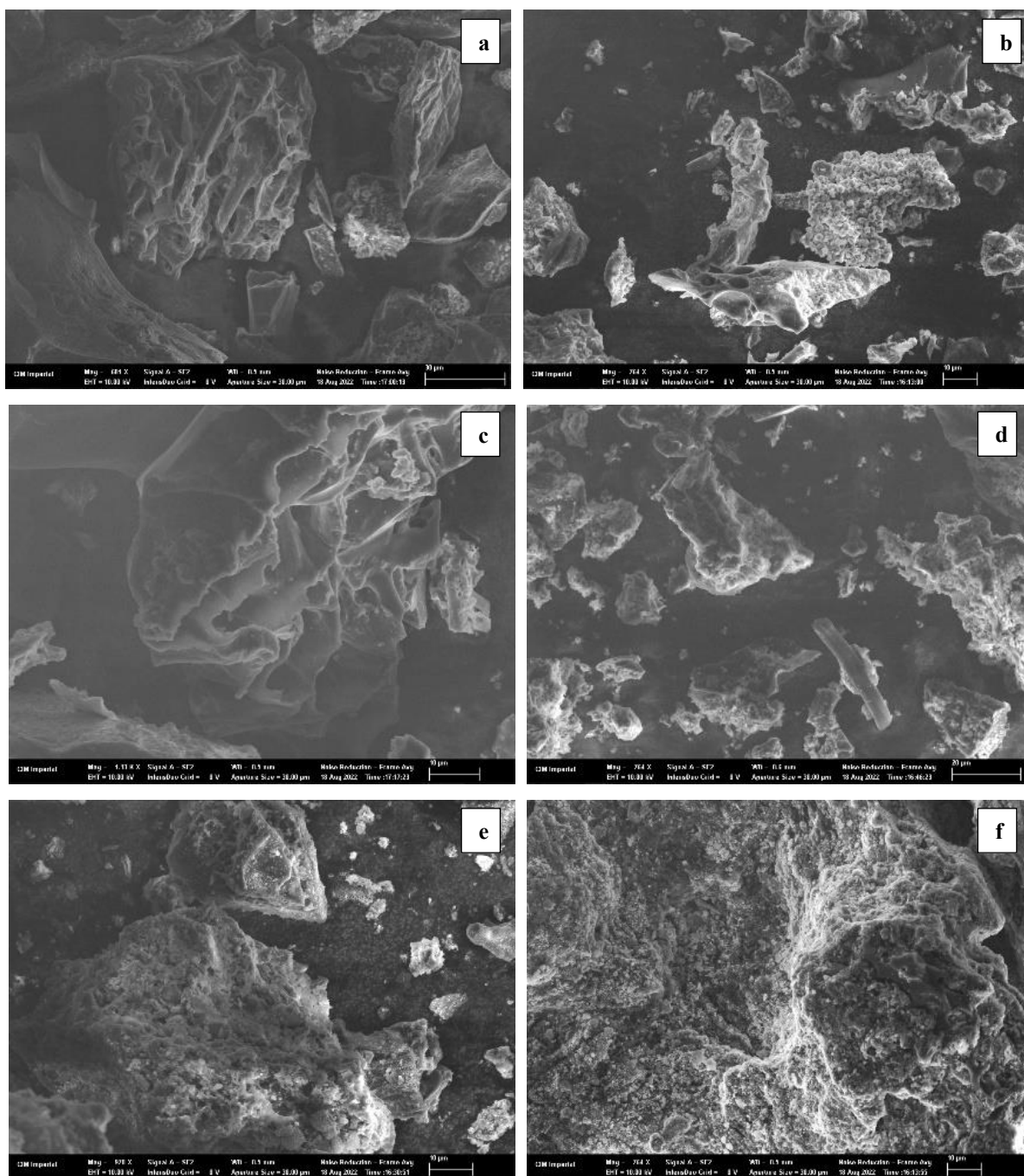


Figure 5.8: Scanning Electron Microscopy (SEM) images for source-separated faeces (SSF) biochars: SSF550 (a), SSF650 (b, c); and mixed urine and faeces (MUF) biochars: MUF550 (d) and MUF650 (e, f), showing the surface morphology of the samples.

The images captured suggest that the addition of urine can alter the surface morphology of produced biochars, which can partly be attributed to the introduction of free moisture on the feedstock surface. Dehydration reactions – occurring either during initial drying at high temperatures, like in this study, or at the onset of pyrolysis in cases of incomplete pre-drying – can cause the formation of richer pore canals and alter the surface structure of resulting biochars (Chen et al., 2022). It has previously been observed that the presence of moisture during sewage sludge pyrolysis can intensify devolatilisation

processes and increase porosity (Xiong et al., 2013). The urine fraction is also rich in $\text{NH}_4\text{-N}$ and K salts, which have been used as activation agents for biomass-derived chars (Moussavi et al., 2013; Zhu et al., 2018). Further investigation of the surface chemistry of source-separated and mixed-stream excreta-based biochars could offer valuable insights on their comparative suitability as adsorbents and other related parameters, including their water holding capacity (Das & Ghosh, 2022). Nevertheless, the increased energy intensity and significant N losses (and emissions) associated with urine-containing sludge pyrolysis, should be balanced against any adsorption reuse benefits.

5.4 Summary

The objective of this chapter was to establish a quantifiable relationship between source separation of human excreta and biochar properties, in the context of resource recovery. The effects of source separation were investigated for different pyrolysis temperatures (450, 550, 650 °C) and this information was used to assess biochars based on their nutrient recovery potential, solid fuel properties and surface morphology.

Results quantified the benefits of urine diversion for nitrogen recovery (70% of total N losses during thermal treatment avoided) and showed an increase in the liming potential of the produced faecal-based biochars. Mixed excreta biochars were more PK-rich and surface morphology investigation showed higher porosity compared to SSF biochars, but their high salinity may place limitations on their use in agriculture, especially for application to salt sensitive plants. For both biochar types (SSF, MUF), the presence of phosphate compounds of high fertiliser value was confirmed by FTIR and XRD analyses, and temperatures between 450-550 °C are recommended to optimise nutrient and carbon behaviour when pyrolysing human excreta. The quality of produced solid fuels was also improved when SSF was the feedstock for pyrolysis, including a 50% increase in char calorific value.

Overall, the data presented in this chapter validate source separation as a promising strategy to increase nutrient and energy recovery from human excreta pyrolysis, as well as to enable the combination of these objectives by treating each excreta stream separately. An additional finding was that mixed excreta may be better suited for adsorption applications, due to the increased surface area of the produced biochars. Further research is recommended to investigate practical impacts on different resource recovery options, depending on the local context and demand for different products.

The findings presented in this chapter can be used to inform the design of circular sanitation systems, linking resource recovery objectives to source conditions, and vice-versa.

6 Enhancing the nutrient content of faecal biochar via adsorption and precipitation from human urine: a circular approach⁵

6.1 Introduction

The findings presented in **Chapter 5** quantified the benefits of separating urine and faeces at source, to enable treatment of each stream based on its inherent properties. A key finding was the potential to avoid N volatilisation losses from urine during thermal treatment, hence maximising the nutrient recovery potential of the urine stream, while the soil improving properties of faecal-derived biochars were also established. Therefore, while separate collection and treatment of human excreta is beneficial, the systemic management of the two excreta streams can maximise the value of produced agricultural products by combining nutrient supply and soil enhancing properties from urine and faecal biochars, respectively.

To this end, this chapter presents a comprehensive method to enrich faecal-derived biochars with N and P recovered from stored, fully hydrolysed human urine via adsorption and precipitation. First, the mass balance of key nutrients ($\text{NH}_4\text{-N}$ ⁶, P, K, Ca, Mg) was observed during the long-term storage of undiluted human urine (up to 6 months, as suggested by the World Health Organization (WHO, 2006) for safe application). Then, the mechanisms of NH_4^+ adsorption onto faecal-derived biochars were investigated under varying biochar doses and $\text{NH}_4\text{-N}$ concentrations. Finally, the simultaneous recovery of N and P from fully hydrolysed urine was optimised via combined adsorption and precipitation induced by MgO addition. The produced nutrient-enriched biochars were characterised via XRD, FTIR, SEM/EDX and ICP-OES analyses, to comprehensively assess their composition and agricultural value. The findings presented in this chapter can contribute to the advancement of resource-oriented sanitation systems that encompass the entire sanitation service chain.

6.1.1 Background

The regulatory framework around the reuse of human urine in agriculture is still unclear, with most countries having limited or no formal guidelines in place. According to WHO (2006), urine stored for 1 month can be safely applied to fodder crops or crops intended for further processing, and urine stored for 6 months can be freely applied to any type of crops. In any case, the large volume produced daily (~1.4 L/person/day) and the unstable, smelly nature of the solution makes the direct application of stored urine in agriculture challenging (Rose et al., 2015; Pathy et al., 2021). Therefore, stabilisation and

⁵ This chapter contains work published by Koulouri et al., (2024a), see **Section 1.5**.

⁶ Ammoniacal nitrogen (referring to both NH_4^+ and NH_3 forms) expressed as N.

nutrient recovery technologies are often deployed, with the objective of indirectly applying urine-derived nutrients in agriculture.

There are various methods for the treatment of urine that can be combined with nutrient recovery, including precipitation, adsorption, volume reduction technologies (e.g. evaporation, freeze-drying, dehydration), stabilisation methods (e.g. pH modification, nitrification) and bio-electrochemical technologies (e.g. electrodialysis) (Maurer et al., 2006; Pronk & Koné, 2009; Udert et al., 2015; Patel et al., 2020). Of these, precipitation and adsorption are particularly attractive due to their low energy consumption, straightforward process-control and satisfactory nutrient recovery rates (Ronteltap et al., 2007; Begum et al., 2020). Precipitation reactions take place spontaneously during urine storage but can be supplemented with the addition of Ca or Mg (Udert et al., 2006). Previous research has identified the successful precipitation of nutrient-rich compounds such as struvite ($\text{NH}_4\text{MgPO}_4 \cdot 6\text{H}_2\text{O}$) and hydroxyapatite (HAP) ($\text{Ca}_5(\text{PO}_4)_3(\text{OH})$) (Udert et al., 2003b). Adsorption applications often focus on the removal of ammonium (NH_4^+) using porous carriers such as activated carbons, zeolites, or biochars that are characterised by high surface area and/or ion exchange capacity (Pathy et al., 2021). Waste-derived biochars can be a sustainable adsorbent choice, with previous research showing promising results for various waste biomass sources, such as wood, spent coffee grounds and waste corn (Begum et al., 2020; Nguyen et al., 2021).

Faecal-derived biochars are an interesting material to consider for nutrient adsorption from human urine, as they are also naturally rich in P and K and exhibit several soil improving characteristics, such as water and nutrient holding properties (Nicholas et al., 2023a). Therefore, faecal biochar enrichment with urine-derived nutrients may be a promising approach to optimise the composition and marketability of fertilisers produced from human excreta, while establishing an important circularity concept within human waste treatment systems. Apart from waste management and eutrophication prevention benefits, this approach provides an alternative route for the outputs of urine-diverting toilets that is designed around resource recovery, as part of a “human excreta-agriculture-development nexus” (Gwara et al., 2020).

The idea of using faecal biochars to recover nutrients from urine was briefly suggested by Tarpeh et al. (2017) and later applied by Bai et al. (2018) and Yu (2022) for biochars derived from septic tank sludge and wastewater sludge respectively, used as carriers for NH_4^+ adsorption from synthetic NH_4Cl solutions ($\leq 1000 \text{ mg NH}_4^+/\text{L}$) or fresh urine ($< 500 \text{ mg NH}_4\text{-N}/\text{L}$). A recent study by Jiang et al. (2023) observed the effect of faecal biochar addition to the stabilisation of partially hydrolysed urine diluted with flush water ($< 200 \text{ mg NH}_4\text{-N}/\text{L}$), suggesting a model for a “pyrolytic eco-toilet” that uses faecal biochars as a urine pre-treatment method. Nevertheless, septic/wastewater sludges are not representative of the purely faecal waste output of urine-diverting toilets, and synthetic/fresh urine do not have the composition and high NH_4^+ concentration of undiluted stored urine (up to $8000 \text{ mg NH}_4\text{-N}/\text{L}$) (Udert et al., 2006). Therefore, while the high NH_4^+ recovery rates reported by previous

studies are promising, they only correspond to a small proportion of the total N (TN) content in a dynamic solution of urine and may not be representative of on-site containment systems. There are currently no studies using faecal-derived biochars and fully hydrolysed, undiluted stored human urine to produce and comprehensively characterise enriched fertilisers. Improving our understanding of the behaviour of these fundamental waste materials (i.e. faeces and urine) during storage and treatment, both separately and combined, is an essential step to advance safe and sustainable nutrient recovery from on-site sanitation systems.

6.2 Materials and methods

6.2.1 Sample collection and biochar preparation

Samples of source-separated faeces and undiluted urine were collected according to the protocol described in **Section 4.1** (and **Appendix C**). Samples of faeces were dried, dry-sterilised, homogenised and combined into a composite sample (<10 mm particle size) (see **Section 5.2.1**), and slow pyrolysis was carried out on the rotary laboratory-scale reactor (see **Section 4.2**) purged with 1.5 L N₂/min. The HHT was set to 500 °C (heating rate 10 °C/min) and maintained for 30 min, according to results presented in **Chapter 5** on optimal pyrolysis conditions for source-separated faeces. The characteristics of the produced faecal-derived biochar are shown in **Table 6.1**

Table 6.1: Characteristics of the faecal-derived biochar in this study (prepared at 500 °C) (on a dry basis).

pH (-)	C (%)	H (%)	N (%)	S (%)	P (g/kg)	P (2%FA) (g/kg)	Olsen -P (g/kg)	K (g/kg)	Ca (g/kg)	Mg (g/kg)	CEC (cmol/kg)	BET (m ² /g)
11.4	50.3	2.4	4.9	<0.	53.9	51.6	3.8	43.0	64.0	20.8	57.6	2.1
±0.1	±0.9	±0.1	±0.3	2	±1.3	±1.6	±0.4	±0.6	±1.4	±0.4	±0.8	±0.1

P (2% FA) = 2% formic acid extractable P, CEC = Cation exchange capacity, BET = Brunauer-Emmett-Teller surface area

Urine sub-samples from individual volunteers were collected in plastic 500 mL containers at various times of day (see **Section 4.1**) and aliquots of each sub-sample were combined into replicates of composite samples (n=5) which were stored in containers with tightly closed lids at room temperature (RTP) for 180 days. The containers were not mixed, to model conditions on-site or at low-cost decentralised storage facilities. Urine samples were analysed at different storage periods for their pH and nutrient concentrations (NH₄-N, P, K, Ca, Mg) (see **Section 6.3.1**).

6.2.2 Batch adsorption and precipitation experiments

Batch experiments were performed to investigate NH₄-N adsorption from stored human urine onto faecal-derived biochars, as well as combined N and P recovery via precipitation. The effect of the

biochar dose (25, 50 and 100 g/L) was investigated by adding 0.5-2 g of biochar to 20 mL urine (n=5) in 50 mL Oakridge centrifuge tubes, which were shaken on a reciprocal shaker at 150 rpm for 72 h in order to reach equilibrium. The range of biochar doses was chosen based on preliminary tests and previously published results (Zheng et al., 2018). The effect of MgO addition on combined N and P recovery was investigated for a range of Mg:P molar ratios between 0.5-4 (refer to **Figure 6.3**), both without biochar addition and in combination with different biochar doses tested.

The biochar particle size was between 0.5-2 mm based on preliminary results that showed no significant increase in NH₄-N adsorption and challenging solid-liquid separation for smaller particle sizes. After the adsorption experiments, the samples were centrifuged and filtered using a 0.45 µm pore size WCN Whatman membrane. The urine-treated biochars were washed with deionised water prior to FTIR, XRD, ICP-OES and SEM/EDX analyses. All the adsorption experiments were performed at RTP (~21.5 °C). Previous studies have reported that room temperature is appropriate for faecal biochar NH₄⁺ adsorption studies, particularly for relevance to low-cost applications (Bai et al., 2018). Quality control samples were analysed to investigate the possible N loss during the experiment due to NH₃ volatilisation. Analysis for other main nutrients (K, Ca, Mg) was also conducted along with N and P, to investigate potential competition between elements and ion exchange reactions.

6.2.3 Adsorption kinetics and isotherms

6.2.3.1 Adsorption kinetics models

Kinetics experiments were carried out to determine the time required to reach equilibrium, both for stored human urine [5-7 g NH₄-N/L (n=5)] and a synthetic NH₄Cl solution with initial NH₄-N concentration of 6 g/L, which is within the range of the concentrations observed for stored human urine in this study. Collected data were modelled based on the pseudo-first (**Eq. 6.1**) and pseudo-second order (**Eq. 6.2**) empirical equations shown below, using MATLAB and Excel spreadsheets available from (Bullen, 2021b). Despite the limited theoretical background of these models, they are considered well-suited to macroporous adsorbents where slow surface reactions (e.g. chemisorption) are significant to the process and have widely been used for biochar and activated carbon adsorption studies (Worch, 2021). Previous research on biochar NH₄⁺ adsorption from aqueous solutions has established that the process is not well-described by diffusion models, so these have been excluded from this analysis (Cui et al., 2016; Zheng et al., 2018).

$$\ln \frac{(q_e - \bar{q})}{q_e} = -k_1 t \quad (\text{Eq. 6.1})$$

$$\frac{t}{\bar{q}} = \frac{1}{k_2 q_e^2} + \frac{t}{q_e} \quad (\text{Eq. 6.2})$$

where q_e is the amount of $\text{NH}_4\text{-N}$ adsorbed at equilibrium (mg/g), t is time (h), $\bar{q}$ is the amount of $\text{NH}_4\text{-N}$ adsorbed at time t (mg/g), k_1 (h^{-1}) is the first-order rate constant and k_2 (g/mg/h) is the second-order rate constant.

For the kinetics experiments, biochar (2.5 g) and 100 mL of stored human urine or NH_4Cl solution were added to 250 mL bottles and shaken at 150 rpm for 72 h. The pH of the NH_4Cl solutions was adjusted to $\text{pH } 9.2 \pm 0.1$ using NaOH, to match that observed for real stored urine, and the actual concentration of the solutions was noted after the pH adjustment. A small aliquot of sample (0.5 mL) was collected at several time intervals and separated by filtration before $\text{NH}_4\text{-N}$ analysis. The effect of volume change due to sampling was considered negligible ($\leq 5\%$ volume reduction over the duration of the experiment).

6.2.3.2 Adsorption isotherm models

Adsorption isotherms were subsequently determined and modelled for both stored human urine and for the synthetic NH_4Cl solution using the Langmuir (Eq. 6.3) and Freundlich (Eq. 6.4) two-parameter equations shown below. The Langmuir model is a theoretical model that represents the adsorption process through a monolayer across a homogenous solid surface, while the Freundlich model is an empirical model that represents multilayer adsorption onto a solid surface (Worch, 2021).

$$q_e = \frac{q_m k_L c_e}{1 + k_L c_e} \quad (\text{Eq. 6.3})$$

$$q_e = k_F c_e^{1/n} \quad (\text{Eq. 6.4})$$

where q_e is the amount of $\text{NH}_4\text{-N}$ adsorbed at equilibrium (mg/g); and c_e (mg/L) is the $\text{NH}_4\text{-N}$ concentration in the solution after equilibrium is reached. The Langmuir isotherm parameters are q_m (mg/g) which is the maximum monolayer adsorption capacity; and k_L (L/mg) which is the Langmuir constant. The Freundlich isotherm parameters are n (dimensionless) and k_F ($\text{mg g}^{-1} (\text{mg L}^{-1})^{-1/n}$).

The models were fitted using MATLAB and Excel spreadsheets available from Bullen (2021b). The Dubinin-Radushkevich (D-R) model (Appendix G) was used to further investigate adsorption mechanisms (see Section 6.3.3), but was only applied for the synthetic NH_4Cl solution, as it is dependent on the solubility of the specific adsorbate (Hu & Zhang, 2019).

For the isotherm experiments, biochar (0.5 g) and 20 mL of stored human urine or pH-adjusted NH_4Cl solution were added to 50 mL Oakridge centrifuge tubes and shaken at 150 rpm for 72 h. All isothermal adsorption experiments were conducted at RTP ($\sim 21.5^\circ\text{C}$), for $\text{NH}_4\text{-N}$ concentrations ranging between 1-7 g/L (concentrations achieved by diluting the stored human urine samples [5-7 g $\text{NH}_4\text{-N/L}$ ($n=5$)] and a stock synthetic solution [8 g $\text{NH}_4\text{-N/L}$] with deionised water). The pH of all solutions was adjusted to 9.2 ± 0.1 with NaOH.

6.2.4 Analytical methods and statistical analyses

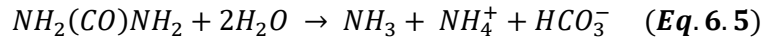
For this chapter, the analytical methods presented in **Section 4.3** were used. Results were statistically analysed by one-way ANOVA to assess differences of $\text{NH}_4\text{-N}$ content during urine storage and adsorption kinetic rates. Two-way ANOVA tests were performed to investigate the influence of biochar dose and MgO addition on N and P adsorption and precipitation from urine (0.05 significance level).

6.3 Results and discussion

6.3.1 Long term urine storage

6.3.1.1 Urea hydrolysis and $\text{NH}_4\text{-N}$ content

The urine characteristics of interest are summarised in **Figure 6.1** and **Table 6.2**. The urea present in fresh urine quickly starts to hydrolyse during storage forming ammoniacal nitrogen (NH_3 and NH_4^+ ; expressed in this thesis as $\text{NH}_4\text{-N}$) and bicarbonate according to **Eq. 6.5** (Mobley & Hausinger, 1989).



The evolution of the $\text{NH}_4\text{-N}$ concentration in urine during a 6-month storage period is shown in **Figure 6.1**. Results have been normalised based on the TN content of each composite urine sample ($n=5$) to account for the high variability of nutrient concentrations in urine (median TN 6.6 g/L [5.2-7.4 g/L]). Interestingly, while absolute values were highly dependent on the initial N concentration, making it impossible to observe statistically significant changes, normalised values showed only small deviations among replicates.

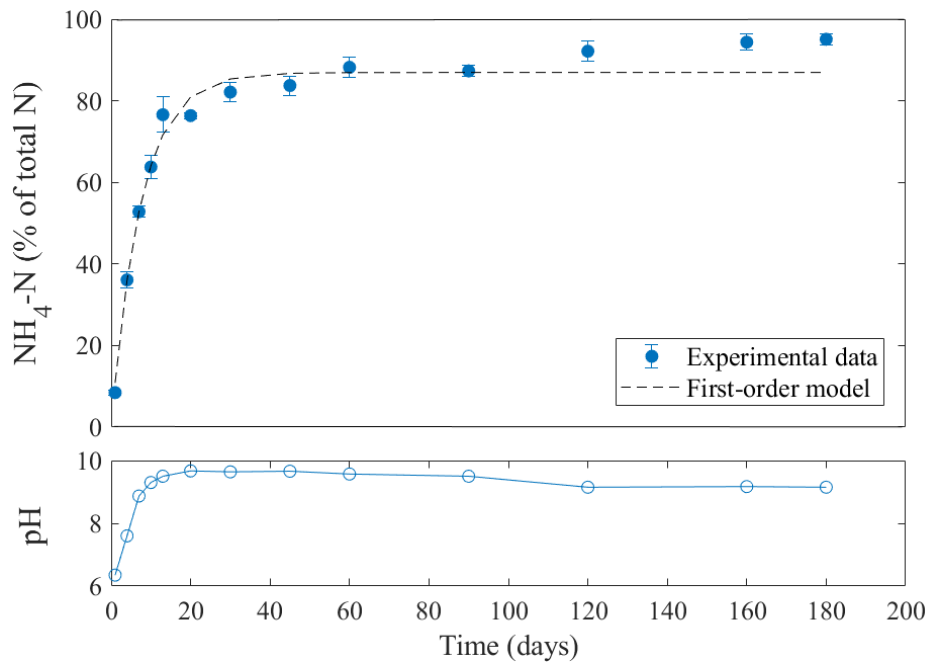


Figure 6.1: $\text{NH}_4\text{-N}$ concentration and pH in undiluted human urine stored for 1-180 days. $\text{NH}_4\text{-N}$ values are normalised based on the total N (TN) content of each composite urine sample (median TN 6.6 g/L [5.2-7.4 g/L]) ($n=5$). Standard deviation values for pH were very small (<0.1) and not visible at this scale.

During the 1st month of storage, a linear increase in NH₄-N concentration associated with urea hydrolysis was observed (reaching 80% of TN), accompanied by a linear increase in pH up to 9.7. The NH₄-N increase followed a first-order reaction model for the first 3 months of storage ($R^2=0.988$), but another statistically significant increase in NH₄-N concentration was observed during the 4th month of storage (up to 90% of TN) (ANOVA results in **Table A8** – Appendix F). This change can be partly attributed to gaseous NH₃ re-dissolution in the liquid phase, as the simultaneous observation of pH buffering towards the NH₄⁺ pK_a value (9.2 (25 °C) - 9.4 (20 °C)) was consistent for all replicates and suggests a dynamic equilibrium between NH₃ and NH₄⁺ gas/liquid phases in the system (Perrin, 1982; Lusk et al., 2017). This experimental study was carried out in tightly sealed containers where gaseous NH₃ can accumulate, exerting a partial pressure that opposes further evaporation and promoting re-dissolution into the liquid phase. As a result, the N losses from the system during storage were minimal (<5% of TN over the 6 months of storage) and most likely occurred during sampling, which required opening the storage containers for small periods of time. Different behaviour and increased N losses may be expected for open or loosely closed storage containers (Udert et al., 2006). No statistically significant changes in NH₄-N concentration or pH were observed after 4 months of storage.

A linear correlation was observed between pH and NH₄-N increase during the first month of storage. Further changes in NH₄-N concentration could be well-predicted by changes in pH values, however, pH should only be used as a quick indicator of hydrolysis completion and NH₄-N increase rate, and not as an indication of absolute concentration values, as these can vary significantly with diet, time of excretion and other factors (Rose et al., 2015).

Previous short-term storage studies have reported much quicker hydrolysis rates, in the scale of hours or days (Udert et al., 2003a; Monetti et al., 2022). The slower hydrolysis rate observed in this study can be partly attributed to different experimental conditions during storage (RTP, no mixing) (Ray et al., 2018; Tao et al., 2019), as well as the lack of urease enzymes present in collected samples. Previous research has identified that urease activity is very high within sewer system infrastructure (pipes, storage tanks, etc.), further accelerating urea hydrolysis rates (Udert et al., 2003a). Slower hydrolysis rates should be expected for container-based sanitation systems and experimental studies where urease enzymes are not introduced from pipes and large-scale tanks.

Short-term studies may also fail to observe the slower rate of NH₄-N increase after the first few weeks of storage. For example, the change in NH₄-N concentration was not significant during the 3rd week of storage ($p=0.931$) but continued increasing with statistical significance after that and up to 120 days of storage (ANOVA results in **Table A8** – Appendix F). Therefore, the experimental storage duration not only affects the form(s) of N available for recovery, but also the stability of the solution and hence the reliability of measured recovery rates. Ammonium adsorption studies using fresh or briefly stored urine may overlook changes in NH₄-N concentrations attributed to normal reactions occurring during storage as responses to experimental parameters, such as adsorbent dose or reaction time.

6.3.1.2 Precipitation and nutrient balance

The increase in $\text{NH}_4\text{-N}$ concentration and high pH values achieved shortly after the start of urine storage (**Figure 6.1**) create conditions favourable to precipitation reactions (Krishnamoorthy et al., 2020). Mass balance calculations were performed for P, K, Ca and Mg, along with the development of $\text{NH}_4\text{-N}$ concentration over the 6-month storage period. While the concentration of $\text{NH}_4\text{-N}$ was increased due to simultaneous urea hydrolysis, the change of concentration of other non-volatile elements can be attributed to precipitation reactions. The elimination of major nutrients from the liquid phase was quantified via ICP-OES (**Table 6.2**) confirming that nutrient-rich precipitates were formed during the long-term storage of undiluted human urine. FTIR analysis of the formed precipitates (**Figure 6.2**) identified major peaks associated with NH_4^+ ($1642, 1417\text{ cm}^{-1}$) (Sidorczuk et al., 2020), $[\text{Ca/Mg}]\text{CO}_3$ ($1514, 852\text{ cm}^{-1}$) (Jones & Jackson, 1993) and PO_4^{3-} ($994, 566\text{ cm}^{-1}$) which can include K-, Ca- and Mg-containing phosphates (Jastrzębski et al., 2011).

Table 6.2: Concentration of major nutrients in composite samples of fresh urine (n=5) and their elimination from the liquid phase (%) during storage for 1 month and 6 months.

	P	K	Ca	Mg
Concentration in fresh urine (mg/L)	861 (± 211.3)	1014.4 (± 138.3)	105 (± 29.9)	101.6 (± 15.7)
Elimination after 1 month (%)	16.8 (± 5.8)	0.8 (± 0.3)	11.2 (± 0.3)	78.1 (± 2.1)
Elimination after 6 months (%)	37.2 (± 6.0)	34.8 (± 5.3)	27.7 (± 1.2)	90.8 (± 0.2)

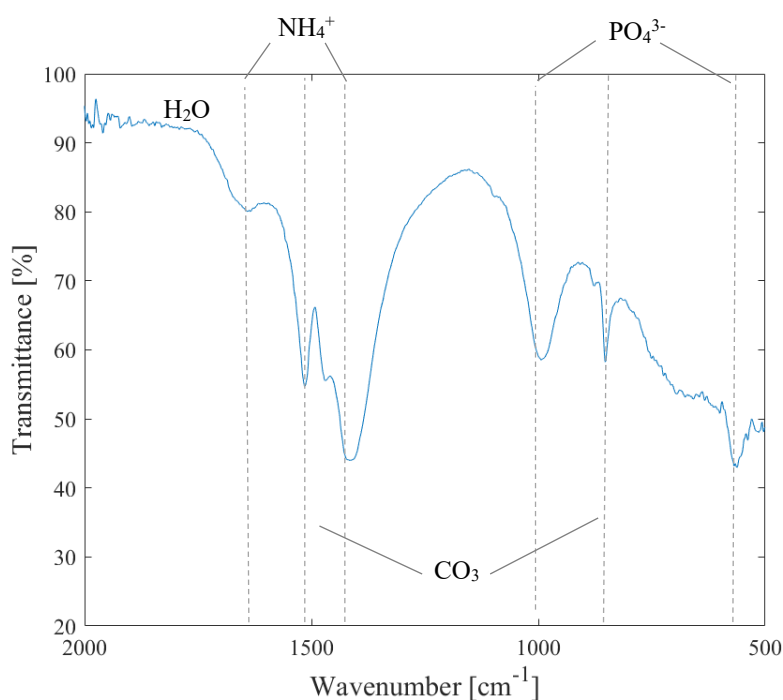


Figure 6.2: Fourier-transform infrared spectroscopy (FTIR) spectrum of the precipitates formed during urine storage, in the $2000\text{-}500\text{ cm}^{-1}$ wavenumber range.

As shown in **Table 6.2**, the high elimination of Mg from the liquid phase during storage (~80% of Mg eliminated after the first month of storage and >90% eliminated after 6 months) suggests that the lack of Mg is a limiting factor for further magnesium phosphates formation (including struvite), causing precipitation reactions to cease soon after urine storage begins. Previous studies on struvite precipitation from source-separated urine have reported MgO as a suitable source of Mg to induce precipitation, due to its chemical properties, including high Mg content and low solubility, as well as cost considerations (Barbosa et al., 2016; Yin et al., 2017). A preliminary assessment was carried out and showed that an MgO dose corresponding to Mg:P (molar ratio) = 1.5 is the optimum for P recovery from fully hydrolysed human urine via precipitation (**Figure 6.3**).

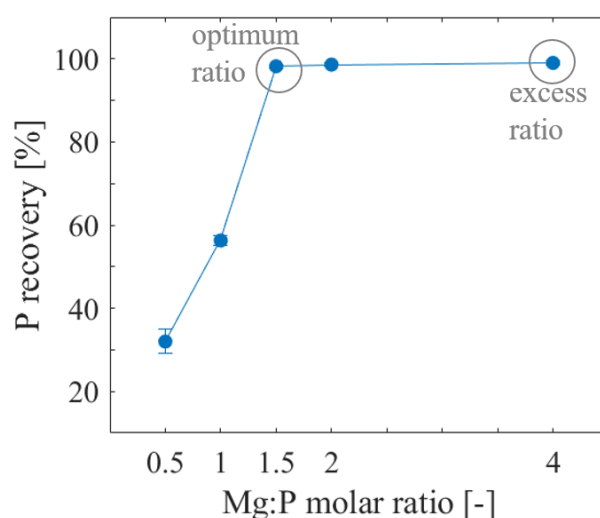


Figure 6.3: P removal from stored human urine (%) after MgO addition to induce precipitation and for different Mg:P molar ratios (without biochar addition). Error bars denote the standard deviation of measured values (n=5).

6.3.2 Combined N and P recovery

6.3.2.1 NH₄-N adsorption kinetics and isotherms

The effect of contact time on NH₄-N adsorption is shown in **Figure 6.4**, for stored human urine (6 months storage time) and for a synthetic NH₄Cl solution (25 g/L biochar dose). For both solutions, the NH₄-N uptake was rapid during the initial 2 hours of the reaction and the adsorption process reached equilibrium after 24 hours, according to ANOVA (**Table A9** – Appendix F). Larger deviation was observed among replicates (n=5) at the onset of adsorption, as the amount of NH₄-N adsorbed was more dependent on the initial concentration of the solution (Begum et al., 2020). Deviation among samples decreased with time as the adsorption became more dependent on biochar dose and reaching equilibrium.

Experimental data have been fitted to pseudo first- and second-order kinetics models and model parameters are shown in **Table 6.3**. The second-order model fits the process very well for both real urine and the synthetic NH₄Cl solution (R²=0.988 and 0.956 respectively). The quick initial adsorption

followed by a continuous decrease in rate until reaching equilibrium is a common finding for favourable adsorption from aqueous solutions and is attributed to the abundance of active adsorption sites and high initial adsorbate concentration at the onset of the process (Nguyen et al., 2021). Interestingly, real urine exhibited a better fit to the model compared to the synthetic solution and the modelled adsorption capacity is almost identical to the experimentally measured capacity for real urine (19.8 mg/g). Therefore, the pseudo second-order model accurately describes the adsorption of $\text{NH}_4\text{-N}$ from real urine on faecal-derived biochars and can be used for the design and optimisation of further, potentially larger-scale, applications. While the synthetic NH_4Cl solution was not a representative alternative to model the adsorption capacity, it did provide useful information on the initial biochar adsorption rate which could be applied to real human urine, for example for continuous adsorption applications (**Figure 6.4**).

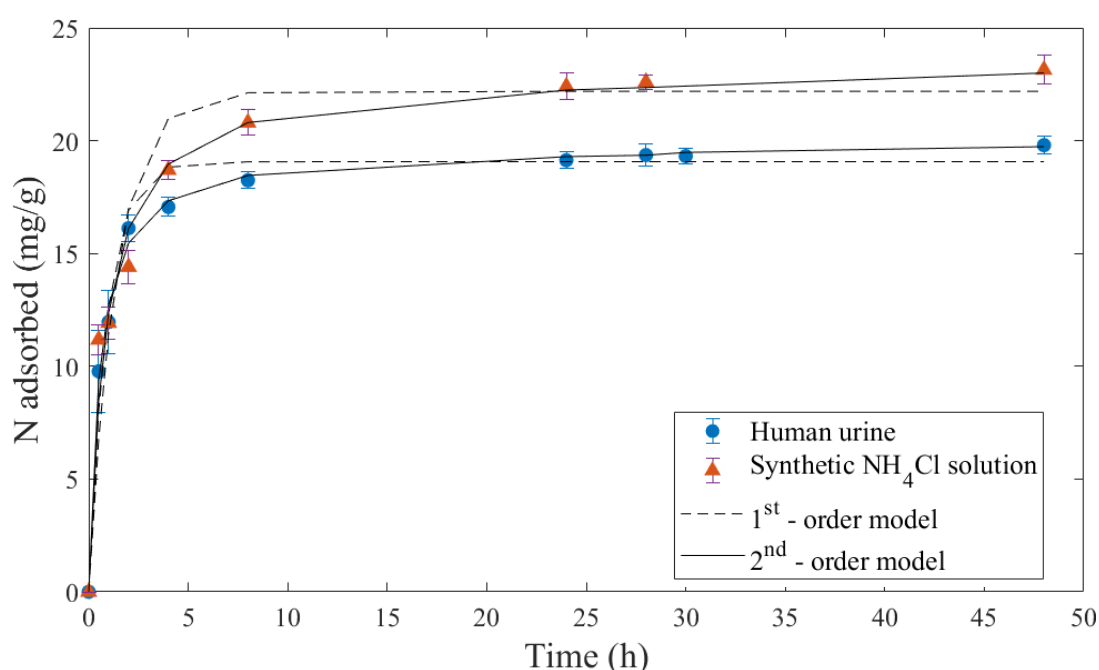


Figure 6.4: Effect of contact time on $\text{NH}_4\text{-N}$ adsorption from stored human urine and a synthetic NH_4Cl solution onto faecal-derived biochar (25 g/L biochar dose) (0–48 h). Results during 48–72 h are not included (no statistically significant changes observed). The system reached equilibrium after 24 hours. Error bars denote the standard deviation of measured values ($n=5$).

Table 6.3: Parameters of the pseudo first- and second-order kinetics models for $\text{NH}_4\text{-N}$ adsorption from stored human urine and a synthetic NH_4Cl solution onto faecal-derived biochars (25 g/L biochar dose).

	Pseudo 1 st -order			Pseudo 2 nd -order		
	q_e (mg/g)	k_1 (h ⁻¹)	R^2	q_e (mg/g)	k_2 (g/mg/h)	R^2
Human urine	19.069	1.09	0.932	19.737	0.092	0.988
NH_4Cl synthetic solution	22.190	0.728	0.855	23.042	0.050	0.956

The parameters of the Langmuir and Freundlich models for $\text{NH}_4\text{-N}$ adsorption onto faecal-derived biochars (dose 25 g/L) are shown in **Table 6.4**, and illustrate the potential of these biochars for adsorption from stored human urine and for a synthetic NH_4Cl solution. The fitted adsorption isotherm curves are included in **Figure 6.5**. Experimental data for human urine exhibit a good fit to the Langmuir and Freundlich models ($R^2 > 0.97$), while the correlation coefficients for the synthetic solution were higher for both cases ($R^2 > 0.99$). This was expected, due to the competition with alternative adsorbates within heterogenous solutions like urine (Bai et al., 2018). Therefore, the theoretical $\text{NH}_4\text{-N}$ adsorption capacity for the synthetic NH_4Cl solution could be an overestimation of that predicted for real urine, although isotherm q_m values were relatively close in this study (23.1 mg/g compared to 22.6 mg/g respectively). For the measured actual $\text{NH}_4\text{-N}$ removal capacity (**Figure 6.5**) a significant difference was observed between synthetic and real samples (23 mg/g for the synthetic solution and 19.8 mg/g for real urine), confirming the hypothesis that competition occurs between NH_4^+ and other species in the (real) urine solution. Moreover, the monolayer adsorption assumption of the Langmuir model may not be met for such a complex adsorption system.

Table 6.4: Parameters of the Langmuir and Freundlich isotherm models for $\text{NH}_4\text{-N}$ adsorption from stored human urine and a synthetic NH_4Cl solution onto faecal-derived biochars (25 g/L biochar dose).

	Langmuir			Freundlich		
	q_m (mg/g)	k_L (L/mg)	R^2	k_F ($\text{mg g}^{-1} (\text{mg L}^{-1})^{-1/n}$)	$1/n$ (-)	R^2
Human urine	22.614	0.0009	0.974	0.695	0.388	0.989
NH_4Cl synthetic solution	23.108	0.0003	0.998	0.118	0.561	0.994

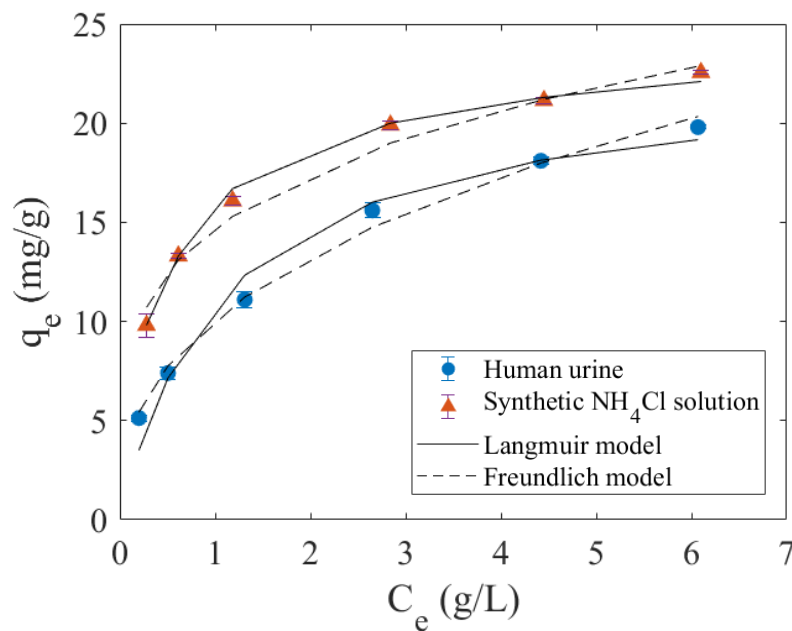


Figure 6.5: Langmuir and Freundlich adsorption isotherms for stored human urine and a synthetic NH_4Cl solution onto faecal-derived biochar (25 g/L biochar dose). Error bars denote the standard deviation of measured values ($n=5$).

Several studies of nutrient recovery from urine have used synthetic NH_4^+ solutions or artificial urine for experimental research, and various urine recipes are available (Ronteltap et al., 2007; Landry & Boyer, 2013; Zheng et al., 2018). Although there are advantages to this approach, including health and safety and ethical protocols that need to be followed during human excreta collection (see **Section 4.1**), there are also several limitations introduced to studies when using a synthetic substrate. Complex hydrolysis and precipitation reactions taking place during storage cannot be realistically observed when using artificial urine. This study used real, fully hydrolysed stored urine to investigate adsorption mechanisms, providing a better understanding of the true $\text{NH}_4\text{-N}$ adsorption behaviour when using faecal-derived biochar as the adsorbent. Successfully fitting isotherm and kinetics models for such a heterogeneous solution is promising for large-scale applications of nutrient recovery from human urine (Wang & Guo, 2020), while the use of source-separated human excreta as the only input materials to the process is particularly relevant to the development of circular sanitation and FSM systems.

6.3.2.2 Effect of biochar dose and MgO addition

The N and P recovery % for different biochar doses (0, 25, 50, 100 g/L) and three MgO addition scenarios (no addition [Mg:P=0]; optimal dose [Mg:P=1.5]; and excess dose [Mg:P=4] – according to **Figure 6.3**) were investigated to assess the suitability of faecal-derived biochars as a carrier for combined adsorption and precipitation from stored human urine (**Figure 6.6**).

When biochar was used without MgO addition, less than 20% of the P in stored urine was removed by the biochar regardless of the dose. The amount of N recovered, however, increased with the increase in biochar dose, from ~14% to >30% of total $\text{NH}_4\text{-N}$, for biochar doses of 25 g/L and 100 g/L respectively. This is attributed to the increase in the biochar-urine contact area with increasing dose (Nguyen et al., 2021). Nevertheless, the increase in N recovery was not linearly correlated to the amount of biochar added, suggesting that complex interactions take place, including parallel precipitation reactions and potential multilayer adsorption mechanisms. Moreover, the addition of the alkaline biochar (pH 11) can alter the pH of the urine solution and hence influence $\text{NH}_4\text{-N}$ adsorption, due to changes in the form of N present ($\text{NH}_3/\text{NH}_4^+$).

The biochar adsorption capacity (19.8 mg $\text{NH}_4\text{-N/g}$) was only reached for low biochar doses (25 g/L), while with higher doses adsorption sites remained unsaturated (Masrura et al., 2022). Therefore, while the higher biochar doses removed more N per litre of urine, the N concentration in the enriched biochar was lower, on a per weight basis. This may have implications on full-scale treatment plants, depending on the objective of the applications. For example, higher biochar doses are favourable when aiming to maximise nutrient removal from urine in order to minimise eutrophication risks downstream. On the contrary, when the main objective is to produce high-value biochar fertilisers, lower biochar doses will lead to more concentrated final products and the remaining urine solution can be further exploited, for example for fertigation.

For the Mg:P=1.5 scenario, >98% of total P (TP) was recovered regardless of the biochar dose. However, when no biochar was added, only 17% of the total $\text{NH}_4\text{-N}$ was recovered via precipitation, and the separation of precipitates from the urine solution was very challenging (**Figure 6.7**). There was no significant increase in N recovery due to MgO dosing when biochar was present in high doses that did not reach adsorption capacity for $\text{NH}_4\text{-N}$ (ANOVA results in **Table A10** – Appendix F). MgO doses higher than Mg:P=1.5 (Mg:P=4 studied as the maximum excess dose) did not have a statistically significant effect on NP recovery.

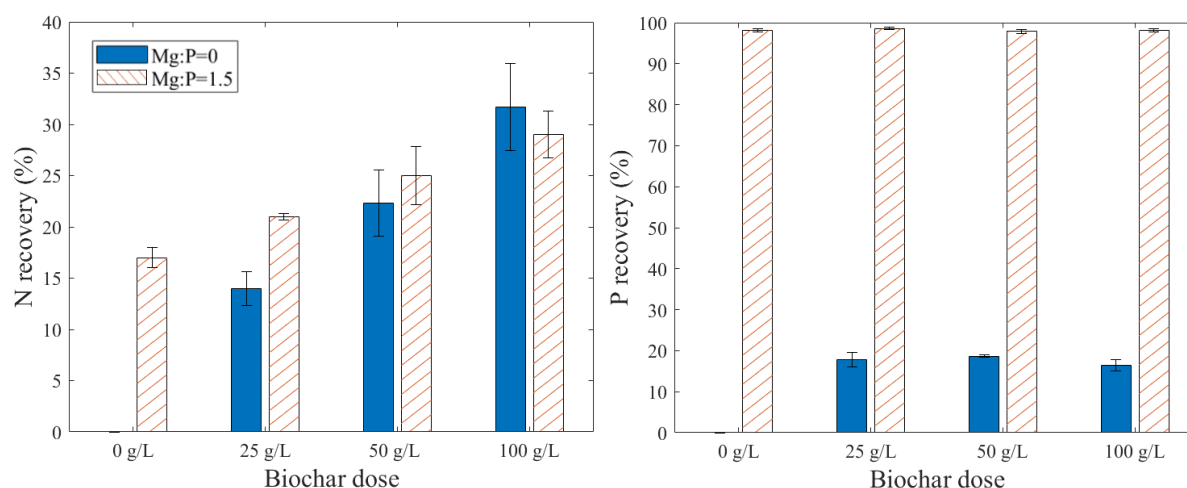


Figure 6.6: Recovery (%) of N and P from stored human urine (6 months) for different doses of faecal-derived biochar (0, 25, 50, 100 g/L) and different MgO doses (Mg:P=0, Mg:P=1.5). Results for Mg:P=4 fall within the statistical range of data shown for Mg:P=1.5 and are not shown. Error bars denote the standard deviation of measured values (n=5).

The combination of MgO (Mg:P=1.5) with the lower biochar dose tested (25 g/L) produced the most NP-rich fertiliser product which was easily separated from the urine solution. The exact NPK ratio of the enriched biochars can vary depending on the composition of urine as well as reaction parameters (temperature, pH, etc.), allowing for further process optimisation to produce fertilisers tailor-made to their intended use. The measured nutrient concentrations for this study are included in **Table 6.5** and show promising NP concentrations for fertiliser use (Moya et al., 2019b). The TN, TP and plant-available P content (as determined via 2% formic acid extraction) of the biochar showed significant increase compared to pre-adsorption values (**Table 6.1**) and the Olsen-P value (as an indicator of readily available P) almost doubled after urine enrichment. While the exact elemental concentrations can vary depending on the composition of human excreta and should not be expected to be replicated for different applications, the ANOVA p-values showing the significance of changes in major nutrients' concentrations before and after adsorption can be used to inform future studies.

Table 6.5: Nutrient concentrations, CHNS analysis and pH value of the faecal-derived biochars after urine adsorption experiments (25 g/L biochar dose, Mg:P=1.5), and ANOVA p-values for the change in parameters before (see **Table 6.1**) and after urine adsorption (**bold** for significant changes).

pH (-)	C (%)	H (%)	N (%)	S (%)	P (total) (g/kg)	P (2% FA) (g/kg)	Olsen-P (g/kg)	K (g/kg)	Ca (g/kg)	Mg (g/kg)
9.7 ±0.1	48.8 ±1.1	2.7 ±0.1	6.5 ±0.6	<0.2	79.6 ±8.4	66.5 ±5.9	7.4 ±0.9	11.7 ±2.0	67.3 ±2.4	30.1 ±4.8
<0.001	0.142	0.021	0.015	-	0.006	0.014	0.003	<0.001	0.108	0.029

Heavy metal concentrations are generally lower for faecal sludge and human excreta compared to sewage sludge (Bleuler et al., 2020), and concentrations for biochars before and after adsorption experiments are shown in **Table 6.6**. Most elements meet the threshold values set by European (EBC, 2012) and international standards (IBI, 2015; WBC, 2023), although Zn and Cu values should be monitored for excreta-derived biochars as they are above the thresholds of EBC and/or WBC, as well as the low-end of the range recommended by the IBI (also refer to **Section 5.3.3.1.2**).

Table 6.6: Trace element concentrations of the faecal-derived biochars before and after urine adsorption experiments (25 g/L biochar dose, Mg:P=1.5), and comparison with max. allowed threshold values from the European Biochar Certificate (EBC), the World Biochar Certificate (WBC) and the International Biochar Initiative (IBI) (**bold** for elements of concern).

Element	Unit	Concentration		Guidelines (max. allowed values)		
		Initial	After adsorption	(EBC, 2012)	(IBI, 2015)	(WBC, 2023)
B	(mg/kg)	35.2 ±3.6	64.16 ±10.18			
Zn	(mg/kg)	479.6 ±53.8	666.62 ±6.13	400	416-7400	420-1000
Pb	(mg/kg)	10.8 ±1.4	18.24 ±3.16	120-150	121-300	120-300
Mn	(mg/kg)	324.6 ±13.36	392.84 ±0.7			
Fe	(mg/kg)	815.37 ±105.8	920.32 ±41.05			
Cd	(mg/kg)	<1	<1	1-1.5	1.4-39	1.5-5
Co	(mg/kg)	<1	<1		34-100	
Cr	(mg/kg)	<1	8.413 ±0.22	80-90	93-1200	100-200
Cu	(mg/kg)	85.52 ±14.87	207.11 ±11.69	100	143-6000	140-200

A note on the feasibility of solid-liquid separation of urine precipitates

During experiments it was observed that, when no biochar was added, the urine-derived precipitates created due to MgO addition formed a sludge and were hard to separate from the liquid urine solution, either by gravity or after centrifugation (**Figure 6.7a**). For full-scale applications this could lead to blockage issues in tanks and pipes, as well as reduce the actual nutrient recovery efficiency due to the incomplete extraction of precipitates. When biochar was added for adsorption, with or without MgO addition (**Figure 6.7b,c**), the solid-liquid separation was easy and quickly achievable by gravity. The formed precipitates due to MgO addition were well combined with the biochar particles (**Figure 6.7c**).

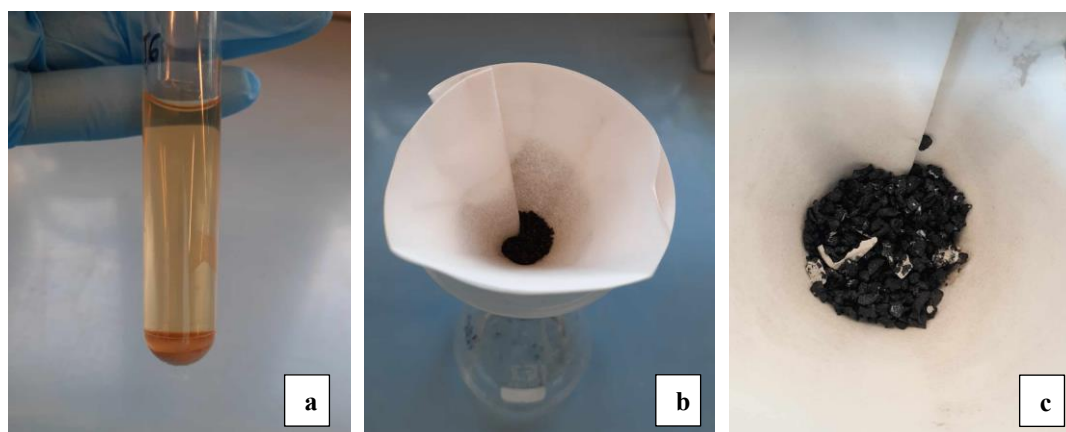


Figure 6.7: Photographs of samples after adsorption studies for different biochar and MgO doses: (a) biochar 0 g/L and Mg:P=1.5 (hard to separate precipitates from urine); (b) biochar 25 g/L and Mg:P=0 (easy separation of biochar from urine); and (c) biochar 25 g/L and Mg:P=1.5 (easy separation of biochar and precipitates from urine).

6.3.3 Adsorption mechanisms

Statistical analysis showed that the biochar dose had a significant influence on N but not P recovery (ANOVA results included in **Table A10** – Appendix F), which can be attributed to the different modes of recovery for each element, i.e. surface adsorption for N and precipitation for P. In contrast, the MgO dose had a significant influence on P but not N recovery. There was however significant interaction between the two variables regarding N recovery, suggesting that the effect of MgO addition depends on the chosen biochar dose. Notably, the influence of MgO addition was only statistically significant for low biochar doses (≤ 25 g/L) that reached adsorption capacity, allowing for the remaining available NH_4^+ to be recovered via struvite precipitation (see **Section 6.3.4**). The lack of significant effects for the higher biochar doses indicates selectivity for direct NH_4^+ adsorption onto the biochar rather than via surface precipitation of struvite, despite both processes being feasible mechanisms for N recovery. Therefore, precipitation was only a secondary mechanism for N recovery when biochar was present.

As shown in **Table 6.1**, the surface area of the faecal-derived biochar was very low ($2.1 \text{ m}^2/\text{g}$) and therefore it is expected that physical adsorption is limited as a mechanism for $\text{NH}_4\text{-N}$ recovery (Nguyen

et al., 2021). In contrast, the cation exchange capacity of the biochar was 57.6 cmol/kg, which is relatively high compared to literature values for other waste-derived biochars (e.g. from agricultural residues) (Gai et al., 2014), suggesting that ion exchange is likely to be the viable adsorption mechanism for NH_4^+ (Ngo et al., 2023). Previous studies on biochar adsorption reported a relationship between NH_4^+ uptake due to ion exchange and the amount of functional groups present on the surface of the biochar (Yin et al., 2017). In this study it was further observed that the characteristic KCl peaks identified via XRD analysis for the untreated faecal-derived biochars (**Figure 6.8**) are not visible for the urine-treated biochars. The supplementary application of the Dubinin-Radushkevich adsorption isotherm model also suggested that ion exchange is the main adsorption mechanism ($E_c=15.4$ kJ/mol) (refer to **Appendix G**). ICP-OES analysis confirmed the elimination of K from the biochar after adsorption (**Table 6.5**), suggesting that an exchange occurs between K^+ and NH_4^+ ions from the biochar and urine respectively. Ion exchange being the main mechanism of biochar adsorption also explains its limited capacity for P recovery, as the usually negatively charged biochar surface cannot exchange anions (Trazzi et al., 2016; Yin et al., 2017). The high amount of P recovered (>98% of total) can be attributed to the precipitates being formed and combined with the biochar, including struvite and hydroxyapatite (see **Section 6.3.4**).

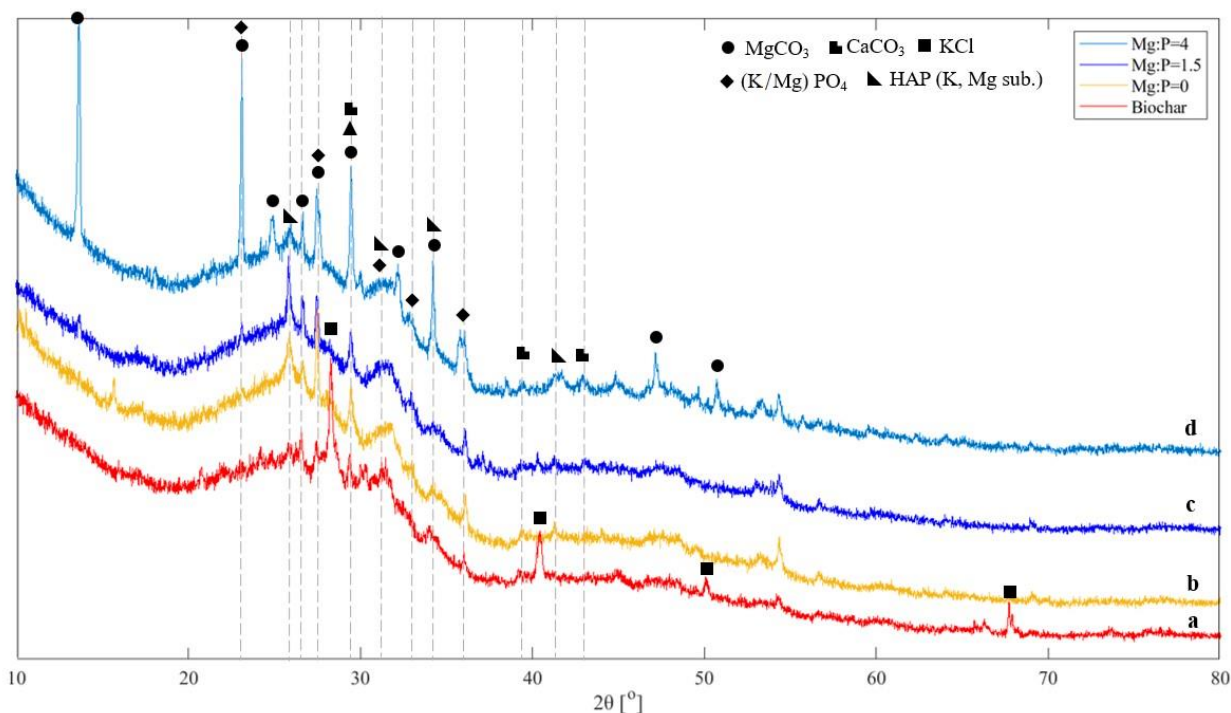


Figure 6.8: XRD patterns for untreated faecal-derived biochar (a) and urine-treated biochars with various MgO addition scenarios: Mg:P=0 (b), Mg:P=1.5 (c) and Mg:P=4 (d). HAP (K, Mg sub.) = hydroxyapatite, including K/Mg substituted and carbonated analogues. (K/ Mg) PO_4 = magnesium and potassium phosphates including struvite and the struvite-K analogue.

Therefore, a trade-off occurs between K and N recovery when using faecal-derived biochars to treat hydrolysed urine. Considering the inherent N deficiency of biochar materials, the benefits of increasing the N content of biochar fertilisers are straightforward (Pathy et al., 2021). From an environmental

protection perspective, reducing the leachable N load from the urine stream is also beneficial (Usman et al., 2022). Nevertheless, due to the very high $\text{NH}_4\text{-N}$ content in hydrolysed urine (up to 7 g/L measured), no more than ~30% of the total $\text{NH}_4\text{-N}$ in urine was removed via biochar adsorption, so a significant amount of N remained available in the urine fraction. The remaining urine solution was rich in both N and K and can be considered for further recovery or fertigation applications. On the contrary, the biochar did not reach capacity for P removal at any of the conditions tested, meaning that it can potentially be reused for further P enrichment. Understanding these synergies and trade-offs is essential to enable the strategic adjustment of the NPK ratio of biochars, which would be an important step towards the production of tailor-made biochar fertilisers from human waste.

While this research aimed to replicate a low-cost decentralised treatment facility with no pH control, further optimisation of N removal can be investigated via pH modification (Yin et al., 2017). The pH of stored urine is quite high (~9.2), at which point around half of the ammoniacal nitrogen is expected to be in the form of NH_3 , which has no charge and therefore does not participate in ion exchange. Future optimisation of this method can focus on maximising the NH_4^+ content over free ammonia. In any case, cost and energy trade-offs should be balanced against N recovery benefits (Chipako & Randall, 2020b).

In terms of P recovery, while the enhancement of the Mg content was found to be necessary to achieve significant recovery efficiencies, it entails a large proportion of the cost required for treatment and recovery (Barbosa et al., 2016). Etter et al. (2011) found that MgO produced from locally available magnesite (MgCO_3) was the most cost-efficient solution for struvite production in Nepal, although CO_2 emissions would be a disadvantage. Natural sources of Mg for the precipitation of struvite or Ca for apatite can also be considered. A previous study by Udert et al. (2003b) indicated that tap water used for flushing can be a source of these elements, although the significant dilution of urine, simultaneous introduction of other competing elements and the potential favourable precipitation of carbonates are a few of the challenges that have been reported. Other natural materials can be considered for the modification of biochars to allow for P recovery, including recent research on eggshell-modified biochars (S. Li et al., 2023).

6.3.4 Characterisation of NP-enriched biochars

The produced enriched biochars were analysed to investigate their chemical composition, both amorphous and crystalline, before and after adsorption. FTIR was used as a preliminary method to quickly observe changes in the chemical structures present in biochars (**Figure 6.9**).

The structure of the material was not significantly altered by urine addition, as the observed spectra were similar for the untreated and urine-treated biochars, including the peak at $\sim 1520\text{ cm}^{-1}$ which has been associated with amine/amide functional groups that could contribute to NH_4^+ adsorption (Begum et al., 2020). When also adding MgO to induce precipitation, there was a change observed in FTIR spectra within the $1600\text{-}1350\text{ cm}^{-1}$ range. The asymmetric bands formed in the region are characteristic

of NH_4^+ bending modes, that have been previously observed for struvite crystals (Cahil et al., 2008). However, the overlap with -OH bending modes makes it challenging to identify the footprint of structural differences, and NH_4^+ stretching modes cannot be analysed due to the strong overlap with O-H stretching at $3600\text{--}2600\text{ cm}^{-1}$ (Dai et al., 2023). Peaks formed at $1550\text{--}1350\text{ cm}^{-1}$ and $\sim 870\text{ cm}^{-1}$ have also been associated with the presence of carbonates and (carbonated) apatites (Stanislavov et al., 2018; Vinoth Kumar et al., 2021). For all biochar samples, there were intense peaks at $1100\text{--}1000\text{ cm}^{-1}$ and $650\text{--}550\text{ cm}^{-1}$ associated with PO_4^{3-} stretching and bending vibrations (Jastrzębski et al., 2011).

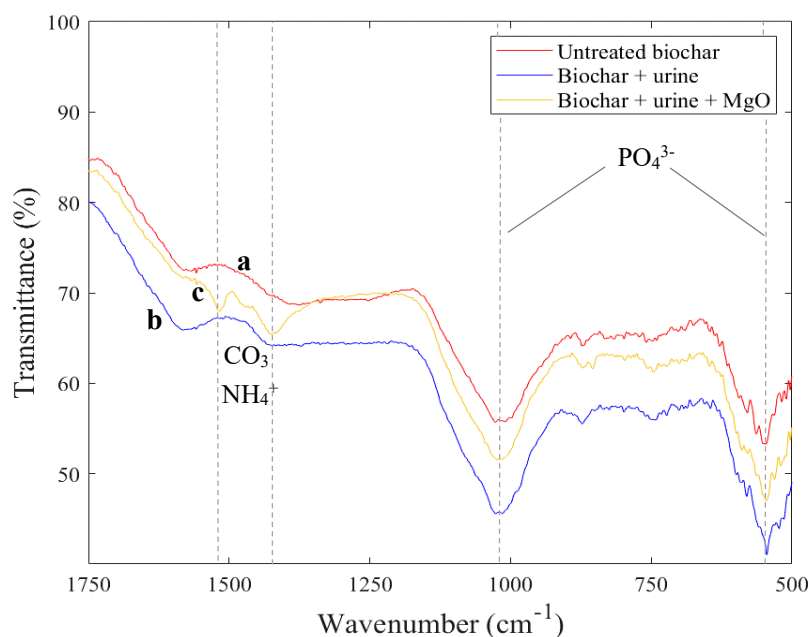


Figure 6.9: Fourier-transform infrared spectroscopy (FTIR) spectra for untreated faecal-derived biochar (a) and urine-treated biochars without MgO (b) or with MgO addition (Mg:P=1.5) (c), in the $1750\text{--}500\text{ cm}^{-1}$ wavenumber range.

The presence of struvite and hydroxyapatite precipitates on the surface of urine-enriched biochars was confirmed by SEM-EDX analysis (**Figure 6.10**). Several types of crystal formations were identified. First, orthorhombic crystals which are typical formations of struvite (Idrus et al., 2018; Kabdaşlı et al., 2022), were observed both as individual larger crystals ($50\text{--}150\text{ }\mu\text{m}$) as well as clusters of smaller crystal formations ($5\text{--}10\text{ }\mu\text{m}$) (**Figure 6.10a,b**). Notably, struvite crystals containing K were also identified, suggesting that the struvite-K analogue ($\text{MgKPO}_4 \cdot 6\text{H}_2\text{O}$) was formed, which can be attributed to the abundance of K^+ ions present in urine after ion exchange occurs (**Figure 6.10c,d**) (Xu et al., 2015). There was a difference in texture observed between the pure struvite (**Figure 6.10a,b**) and the struvite-K crystals, which were less smooth and had noticeable fragments on the surface (**Figure 6.10c,d**). The varying elemental ratio of struvite(-K) crystals (see EDX results in **Figure 6.10**) suggests that the formed precipitates may not be strictly stoichiometric. Due to the heterogenous composition of urine, it is also expected that other magnesium phosphates of low solubility - e.g. $\text{Mg}_3(\text{PO}_4)_2$ ($k_{\text{sp}} = 10^{-24}$ at $25\text{ }^\circ\text{C}$) (Haynes et al., 2013) - may have formed in conjunction with struvite and struvite-K (k_{sp} values 10^{-13} and $10^{-12.2}$ respectively at $25\text{ }^\circ\text{C}$) (Xu et al., 2015; SenGupta, 2017).

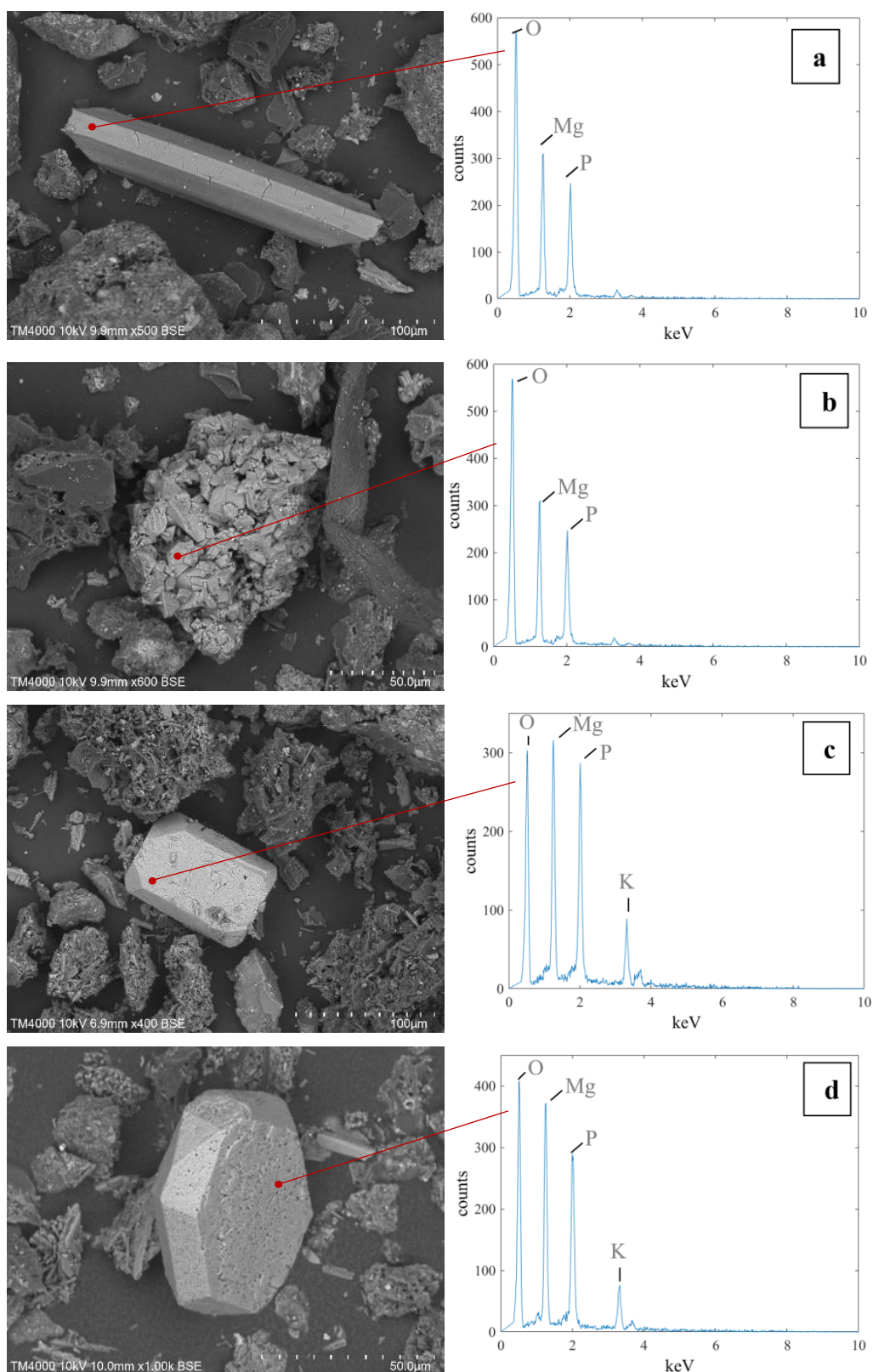


Figure 6.10: Scanning Electron Microscopy (SEM) with Energy Dispersive X-Ray (EDX) analysis for faecal-derived biochars after treatment with stored human urine and MgO (Mg:P=1.5), showing crystals identified as struvite (a, b) and struvite-K (c, d). EDX signal noise peaks at low energy levels (keV<0.3), including for C, have been excluded using default instrumental settings for clarity.

Further observations during analysis showed that the quantity and size of struvite crystals increased as a result of MgO addition (Mg:P=1.5), but no further increases were observed when excess Mg was present (Mg:P>1.5). For excess Mg scenarios, the most prevalent crystal structures were sharp needle-like formations, which were identified by EDX analysis as MgCO_3 (**Figure 6.11**). These needle-like crystals were often observed on the surface of struvite crystals, suggesting that the purity of P-containing crystals was compromised by the presence of excess Mg. The main peaks identified via XRD analysis (**Figure 6.8**) for the Mg:P=4 scenario are in accordance with these findings, confirming the prevalence of MgCO_3 over phosphate compounds. The total NPK concentration of the produced biochars was diluted due to the additional mass of Mg-containing crystals and excess Mg addition was found to have a negative effect on the nutrient value of the enriched biochars. Therefore, the optimum Mg:P ratio (in this case, 1.5) needs to be identified before large-scale applications.

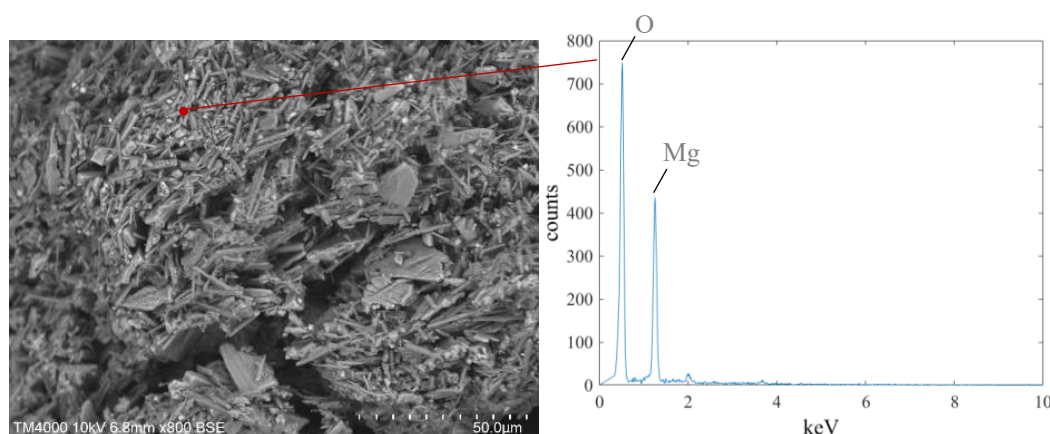


Figure 6.11: Scanning Electron Microscopy (SEM) with Energy Dispersive X-Ray (EDX) analysis for faecal-derived biochar after urine adsorption and excess MgO addition (Mg:P=4), showing the formation of MgCO_3 crystals. EDX signal noise peaks and overlapping peaks at low energy levels (keV<0.3), including for C, have been excluded using default instrumental settings for clarity.

Another common morphological observation for urine-treated biochars was clusters of small spherical particles (1-2 μm) which were identified as Mg/K(CO_3)-substituted hydroxyapatite (**Figure 6.12**) (Huang et al., 2016; Nowicki et al., 2022). The substitution of Ca ions with Mg and K ions can be attributed to the abundance of these elements in the urine solution and is expected to increase the formation of hydroxyapatite ($k_{\text{sp}} = 10^{-55.9}$ at 25 °C) (Snoeyink & Jenkins, 1980; Mocanu et al., 2021). Observations of changes in crystal size and morphology due to these substitutions were not significant. Typical peaks for hydroxyapatite, carbonated hydroxyapatite and substituted forms were also identified via XRD (**Figure 6.8**) (Manafi et al., 2008; Iancu et al., 2020).

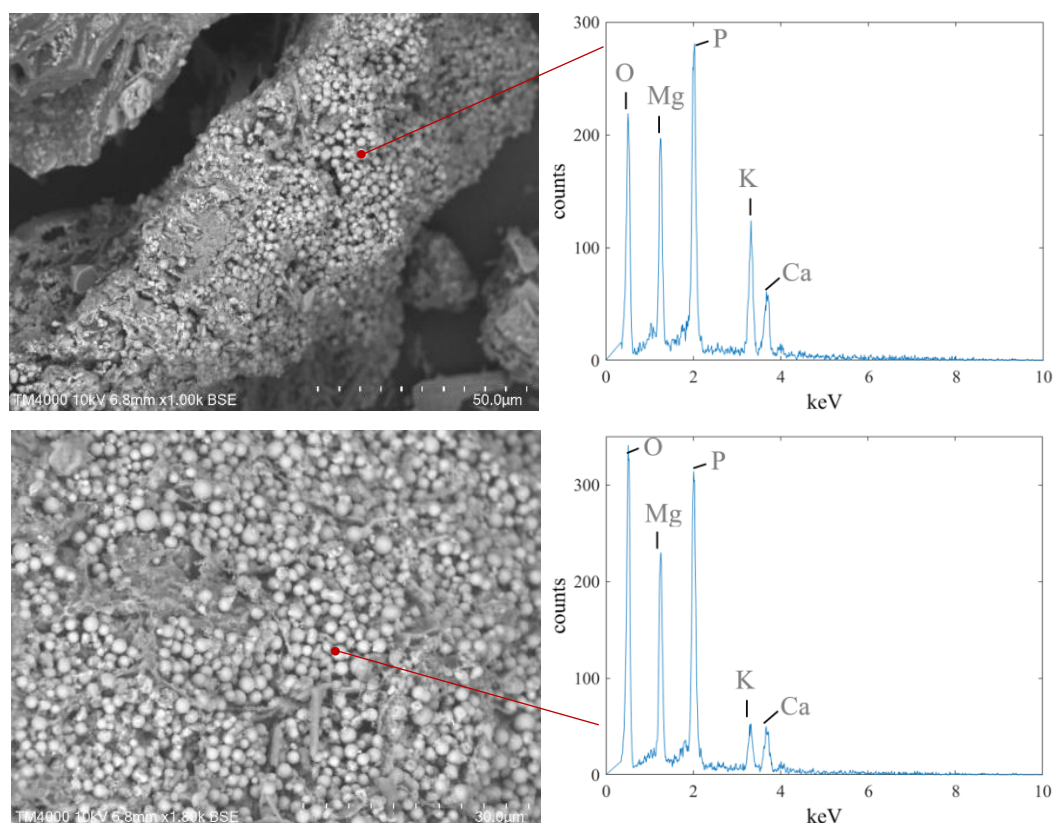


Figure 6.12: Scanning Electron Microscopy (SEM) with Energy Dispersive X-Ray (EDX) analysis for faecal-derived biochars after treatment with stored human urine and MgO (Mg:P=1.5), showing crystals identified as Mg/K(CO₃)-substituted hydroxyapatite. EDX signal noise peaks at low energy levels (keV<0.3), including for C, have been excluded using default instrumental settings for clarity.

The various P-containing precipitates were qualitatively observed for all urine-enriched biochars, regardless of the presence of MgO, and crystals were well-incorporated within the carbon matrix of the biochar. XRD peaks were also similar for Mg:P=0 and Mg:P=1.5 scenarios (**Figure 6.8b,c**) suggesting that the same types of precipitates can be formed regardless of MgO addition. However, the number of SEM observations of these precipitates greatly increased as a result of MgO addition, explaining the significant increase in P recovery from urine (from <20% to >98%) when additional Mg was present. When no MgO was added, the carbon structure of the samples was more evident compared to the crystalline precipitates.

6.4 Summary

In this chapter, faecal-derived biochar was used as a medium to produce and, for the first time, comprehensively characterise NP-enriched biochar fertilisers with the addition of fully hydrolysed, stored human urine. The evolution of urea hydrolysis and nutrient content during urine storage was initially investigated over a 6-month storage period and NH_4^+ adsorption mechanisms studied under varying biochar doses and $\text{NH}_4\text{-N}$ concentrations. The process was further optimised by adding MgO to induce precipitation reactions, enabling the combined recovery of NH_4^+ and P.

For NH_4^+ adsorption, experimental data exhibited a good fit to both the Freundlich ($R^2 = 0.989$) and Langmuir ($R^2 = 0.974$) isotherm models and the rate of the reaction was well described by a pseudo 2nd order kinetics model ($R^2 = 0.988$). The NH_4^+ uptake was rapid during the initial 2 h of the reaction and the adsorption process reached completion after 24 h. The $\text{NH}_4\text{-N}$ adsorption capacity of the faecal-derived biochar was 19.8 mg/g and the main uptake mechanism identified was ion exchange ($\text{K}^+ \leftrightarrow \text{NH}_4^+$), as confirmed by XRD and ICP-OES.

The effect of different biochar doses (0, 25, 50, 100 g/L) and MgO addition scenarios (Mg:P = 0, 1.5, 4) on N and P recovery showed that the combination of MgO (Mg:P = 1.5) with the lower biochar dose (25 g/L) produced the most NP-rich fertiliser product which was easily separated from the urine. Faecal-derived biochar had a limited adsorption capacity for P, with precipitation being the main mechanism for P recovery. When MgO was added to urine, >98% of total P was recovered via precipitation of struvite/struvite-K and Mg/K(CO_3)-substituted hydroxyapatite, as identified via SEM-EDX. Faecal-derived biochar was a successful carrier to recover the P-containing precipitates and facilitate liquid-solid separation after treatment.

The findings of this research provide proof-of-concept for the systemic management of source-separated human excreta and demonstrate the feasibility of the production of marketable, combined fertilisers and soil amendments from on-site sanitation waste.

7 Co-pyrolysis of source-separated human faeces with wood biomass: Effect on carbon flows and carbon storage potential⁷

7.1 Introduction

Previous chapters have established the value of faecal biochars as fertilisers and soil amendments, with or without the addition of urine-derived nutrients. Agricultural applications of biochar can also be used as a soil-based carbon sequestration strategy because the biochar is an aromatic form of carbon that can potentially remain stable for hundreds or thousands of years (Mandlebaum & Nriagu, 2011). This is particularly true when faeces are co-pyrolysed with additional lignocellulosic biomass materials (e.g. wood-based cover materials in CBS toilets) that can be expected to alter the behaviour of carbon within FSM systems (Ippolito et al., 2020). Therefore, pyrolysis can be deployed when aiming to sequester carbon from human excreta and FS, especially for the context of developing countries that have largely agriculture-centred economies and abundant biomass and sanitation waste sources (Tan, 2019). Still, very little is known about carbon flows during pyrolysis of human excreta and the impact that different blending ratios of cover materials have on carbon stability and sequestration potential, for different operational conditions.

This chapter presents the assessment of carbon sequestration potential when co-pyrolysing human faeces with wood biomass at different blending ratios and treatment temperatures by: 1) investigating thermal behaviour and end-product characteristics; 2) quantifying carbon retention and stability in the biochar; and 3) for the first time, mapping the distribution of carbon flows among pyrolysis products. This research can be used to assess the carbon footprint of FS pyrolysis and inform sustainability decision-making in sanitation systems. The research methods presented in this paper can be used for the development of protocols to comprehensively assess the stability and carbon sequestration potential of excreta-derived biochars. Supporting sustainability claims with transparent and systematically documented technical knowledge is crucial to understand and maximise carbon benefits, as well as overcome scepticism around climate finance schemes (Clark et al., 2018).

⁷ This chapter contains work published by Koulouri et al., (2024b) see **Section 1.5**.

7.2 Materials & Methods

7.2.1 Sample collection

Samples of source-separated human faeces (HF⁸) were collected from 12 volunteers in the UK during July 2022, according to the protocol described in **Section 4.1** (and **Appendix C**). The HF samples were dried, dry-sterilised, homogenised and ground into particle sizes <10 mm (see **Section 5.2.1**) before pyrolysis experiments. Wood biomass (WB) (willow wood) was also locally collected, dried, and ground homogeneously to the same particle size. HF and WB were blended at ratios of 25:75, 50:50 and 75:25 (dry % w/w), corresponding to sample names HF25, HF50 and HF75 respectively.

7.2.2 Pyrolysis experiments

Pyrolysis experiments were conducted using the protocol described in **Section 4.2**. A 20 g sample aliquot was placed in the reactor and N₂ was purged through the vessel to ensure an inert atmosphere was maintained. The biochar characterisation experiments (see **Section 7.3.2**) were performed for three prescribed temperatures (HHTs) (450, 550, 650 °C) with a heating rate of 10 °C/min, as described in **Section 5.2.2**. For the investigation of carbon flows among pyrolysis products (see **Section 7.3.3**), several retention times (0.5, 1, 2 h) and inert gas flow rates (0.5, 1, 1.5 L/min) were tested. Pyrolysis conditions were chosen based on preliminary findings and previously published studies (Ward et al., 2014; Liu et al., 2014; Koulouri et al., 2023).

Biochar samples are named B-HF, B-HF25, B-HF50, B-HF75 and B-WB corresponding to the feedstock types defined in **Section 7.2.1**. For the carbon flows assessment, a “FS pyrolysis system” is defined based on the boundaries of the pyrolysis treatment unit; i.e. from the (dry) feedstock entering the pyrolysis furnace, to the final products (biochar, bio-oil, non-condensable gases (NCG)) collected after treatment.

7.2.3 Biochar stability assessment

There is currently very little knowledge on the stability and carbon sequestration potential of biochars produced from human excreta and FS, as well as on the effect of co-pyrolysis with other biomass waste. Existing research for biochars produced from other biomass sources, such as wood or various agricultural waste, indicates that both feedstock composition and operational conditions can have a significant effect on the stability and carbon sequestration potential of biomass pyrolysis applications (Windeatt et al., 2014; Crombie & Mašek, 2015; Somerville & Deev, 2020). Methods to evaluate biochar stability have been categorised as: a) carbon structure analysis (qualitative and quantitative); b) oxidation resistance analysis (thermal and chemical degradation); and c) biochar persistence analysis

⁸ Samples of source-separated human faeces in this chapter are named HF to differentiate from SSF samples introduced in **Chapter 5**, which were collected from a different batch of volunteers (see **Section 4.1**).

(incubation and modelling) (Leng et al., 2019). Although interest in carbon sequestration via FS pyrolysis is increasing rapidly, these technical parameters of biochar stability have not been systematically investigated for FS or human excreta.

In this chapter, the stability of biochars produced from co-pyrolysis of HF and WB was assessed for the first time, using previously established methods for carbon structure and oxidation resistance analysis, and for the different HF:WB ratios and pyrolysis temperatures tested. Proximate analysis and water-extractable dissolved organic carbon (DOC) analysis were carried out as preliminary screenings to test the volatile carbon content of biochars produced under different temperatures and assess their suitability for carbon storage applications (**Section 7.3.2.1**). For the carbon structure analysis, results include the H/C and O/C ratios, SEM and FTIR analyses (**Section 7.3.2.2**). For the oxidation resistance of the biochars, H₂O₂ was used to assess chemical oxidation potential, and the recalcitrance index (R₅₀) method by TGA was implemented to study thermal degradation (**Section 7.3.2.3**) (Harvey et al., 2012; Cross & Sohi, 2013).

7.2.4 Analytical methods and statistical analyses

The carbon flows among pyrolysis products (biochar, bio-oil, NCG) were calculated based on **Eq. 7.1-7.3**. Carbon analysis was performed on a ThermoScientific Flash Smart Elemental Analyzer as described in **Section 4.3.1.3** (ISO, 2015).

$$Carbon_biochar [\%] = \frac{C_{biochar}}{C_{feedstock}} \times Y_{biochar} \quad (Eq. 7.1)$$

$$Carbon_bio_oil [\%] = \frac{C_{bio-oil}}{C_{feedstock}} \times Y_{bio-oil} \quad (Eq. 7.2)$$

$$Carbon_NCG [\%] = 100 - Carbon_bio_oil [\%] - Carbon_biochar [\%] \quad (Eq. 7.3)$$

where Carbon_biochar, Carbon_bio_oil and Carbon_NCG are the carbon flows (%) to the biochar, bio-oil and NCG streams respectively. C_{biochar}, C_{bio-oil}, C_{feedstock} are the C content values (% w/w) for the biochar, bio-oil and feedstock samples. Y_{biochar} and Y_{bio-oil} (% w/w) are the product yields for biochar and bio-oil respectively. For the yield calculations, residues on the surface of the reactor with negligible fixed carbon content were attributed to bio-oil formation. The biochar yield was calculated based on the total weight of recoverable solids.

7.2.4.1 Water-extractable dissolved organic carbon (DOC)

For the water-extractable DOC analysis, (dried) biochar samples were mixed with deionised water (1:50 g:mL ratio) and shaken on a reciprocal shaker at 150 rpm for 16 h at room temperature (method modified from Tang et al. (2019) and F. Wang et al. (2022)). Samples were then centrifuged and the supernatant was analysed in a total organic carbon (TOC) analyser (Shimadzu, TOC-V WS). The TOC analyser was calibrated using a diluted 1000 mg/L solution of potassium hydrogen phthalate (KHP).

7.2.4.2 H₂O₂ oxidation

The carbon chemical stability of the biochars was determined by H₂O₂ oxidation, indicating the oxidation resistance in natural soils (Cross & Sohi, 2013; Mia et al., 2017). Briefly, 40 mL 5% H₂O₂ was added to 0.1 g (dried) biochar and left in a thermostatic water bath at 60 °C for 24 h as suggested by Han et al. (2018). Following contact with the H₂O₂ solution, the biochar samples were filtered through a Whatman no.1 filter under vacuum, rinsed with deionised water to remove chemical residues and dried at 105 °C overnight. The C stability was then determined based on **Eq. 7.4**.

$$C_{stability} [\%] = \frac{C_2 \times M_2}{C_1 \times M_1} \times 100\% \quad (\text{Eq. 7.4})$$

where $C_{stability}$ (%) defines the proportion of stable carbon, M_1 (g) and C_1 (% w/w) represent the mass and carbon content of the initially added biochar, respectively, and M_2 (g) and C_2 (% w/w) represent the mass and carbon content of the remaining biochar after oxidation, respectively.

7.2.4.3 Recalcitrance index (R₅₀)

The recalcitrance index (R₅₀) method proposed by Harvey et al. (2012) was applied to illustrate the relative thermal stability of the biochar compared to that of graphite. TGA was performed under an air atmosphere, heating from room temperature to 900 °C at 20 °C/min. The R₅₀ value was calculated using **Eq. 7.5** after correcting for the moisture and ash content of the biochar (**Eq. 7.6**) (Harvey et al., 2012).

$$R_{50} [-] = \frac{T_{50,biochar}}{T_{50,graphite}} \quad (\text{Eq. 7.5})$$

where R₅₀ is the recalcitrance index value and $T_{50,biochar}$, $T_{50,graphite}$ (°C) are the temperatures representing 50% weight loss for biochar and graphite respectively. The $T_{50,graphite}$ value was 886 °C. The $T_{50,biochar}$ was calculated after correcting for moisture and ash content based on **Eq. 7.6**:

$$W_{corrected} = 100 + [100 \times \frac{W - W_{200}}{W_{200} - W_{cutoff}}] \quad (\text{Eq. 7.6})$$

where $W_{corrected}$ is the sample weight after correction for moisture and ash content, W is the uncorrected sample weight, W_{200} is the sample weight at 200 °C and W_{cutoff} is the sample weight at the temperature where no further weight loss is apparent (Harvey et al., 2012).

7.2.5 Statistical analyses

Results were statistically analysed by one-way ANOVA to investigate the influence of different HF:WB ratios on feedstock characteristics and two-way ANOVA to investigate the influence of different HF:WB ratios and pyrolysis operational parameters on biochar properties and carbon flows (0.05 significance level). Multiple comparison tests were used to localise impacts among factor levels, where significant influences were detected.

7.3 Results & Discussion

7.3.1 Feedstock characteristics

The chemical bonds and organic constituents present in HF, HF50 and WB samples, as qualitatively investigated via FTIR analysis, are shown in **Figure 7.1**. The carbon content and proximate analysis for all feedstock combinations are shown in **Figure 7.2** (ANOVA results in **Table A11** – Appendix F).

Differences in the temperature and intensity of peak decomposition rates during pyrolysis can be expected due to differences in the organic composition of HF compared to the purely lignocellulosic composition of WB (Rose et al., 2015; Fidalgo et al., 2019). Typical peaks of aliphatic C–H bonds ($2920, 2850\text{ cm}^{-1}$) and C=O stretching ($\sim 1750\text{ cm}^{-1}$) were more intense for blends containing more HF, due to the presence of fat and lipids (**Figure 7.1**) (also refer to **Section 5.3.1**). All samples exhibited strong peaks at the $1750\text{--}1450\text{ cm}^{-1}$ wavenumber range but several differences in the vibrational bands are observed, as both protein (more prevalent in HF) and lignin (more prevalent in WB) have characteristic peaks at that range (Krueger et al., 2021b). These different organic constituents are expected to have an effect on the yield distribution of pyrolysis products (biochar, bio-oil, NCG) and the composition of evolved gases (Somorin et al., 2020b). For example, fatty constituents contribute to the bio-oil yield, while protein and lignin contribute to biochar formation (Hassen et al., 2014; Abnisa & Wan Daud, 2014).

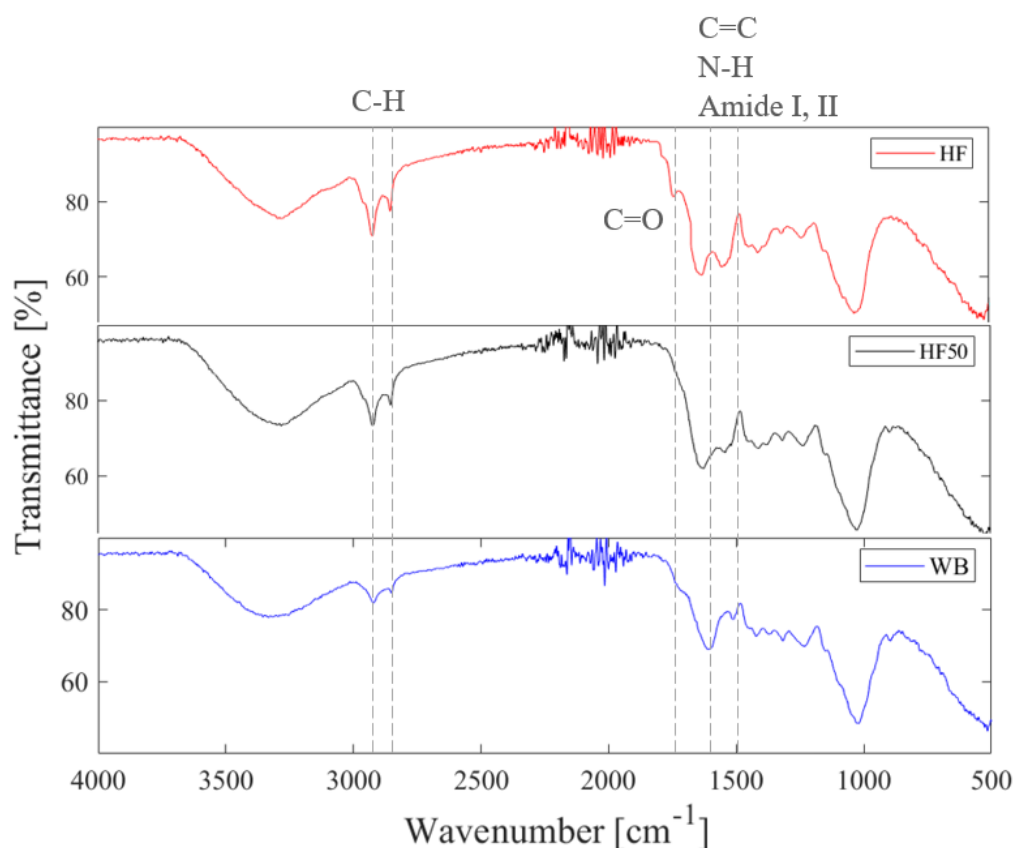


Figure 7.1: Fourier-transform infrared spectroscopy (FTIR) for HF, HF50 and WB samples, in the $4000\text{--}500\text{ cm}^{-1}$ wavenumber range.

Proximate analysis results (**Figure 7.2**) confirm that adding WB in higher ratios increased fixed carbon content (from 14.6% to 20.5% for HF and HF25 respectively) and decreased ash content (from 12.9% to 7.9% for HF and HF25 respectively) in HF:WB blends. These trends indicate the different carbon retention and stability potential of the materials, suggesting that carbon flows between process end-products are expected to be different due to WB addition, despite the feedstocks having almost identical initial total C contents (**Figure 7.2**). Therefore, the total C value does not provide sufficient information on the carbon behaviour of the feedstock materials and is not an appropriate indicator of their carbon retention potential. The nature of the carbon in the feedstock, which is dependent on the organic composition and has an influence on decomposition behaviour and biochar characteristics, needs to be understood - both quantitatively and qualitatively - to enable the optimisation of carbon flows during pyrolysis.

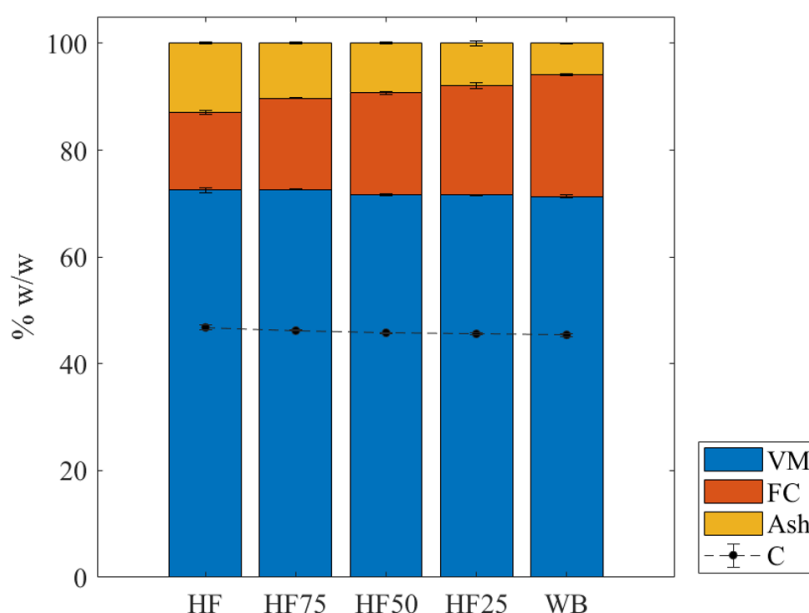


Figure 7.2: Carbon and proximate analysis results for HF, HF75, HF50, HF25 and WB samples (on a dry weight basis). VM = Volatile Matter; FC = Fixed Carbon. Error bars denote the standard deviation of measured values (n=3).

Previous research by Somorin et al. (2020b) on the thermogravimetric analysis of these materials, provided baseline information on their expected behaviour during pyrolysis. The authors found that co-pyrolysis of HF and WB had synergistic effects and enhanced the degradation of HF regardless of the mixing ratio (Somorin et al., 2020b). For the present study, the thermal decomposition behaviour of samples HF, HF50 and WB is depicted in the TGA and DTG curves included in **Figure 7.3**. The maximum rate of decomposition is observed at 310 °C, 335 °C and 345 °C for HF, HF50 and WB respectively. The earlier peak weight loss rate for HF can be attributed to the decomposition of protein and is followed by a distinct shoulder peak between 400-500 °C, due to the decomposition of oil and

grease (see **Section 5.3.1**). This shoulder peak is also visible for HF50, but not for the purely lignocellulosic WB which shows a distinct single peak associated with lignin and cellulose/hemicellulose decomposition (Krueger et al., 2021b).

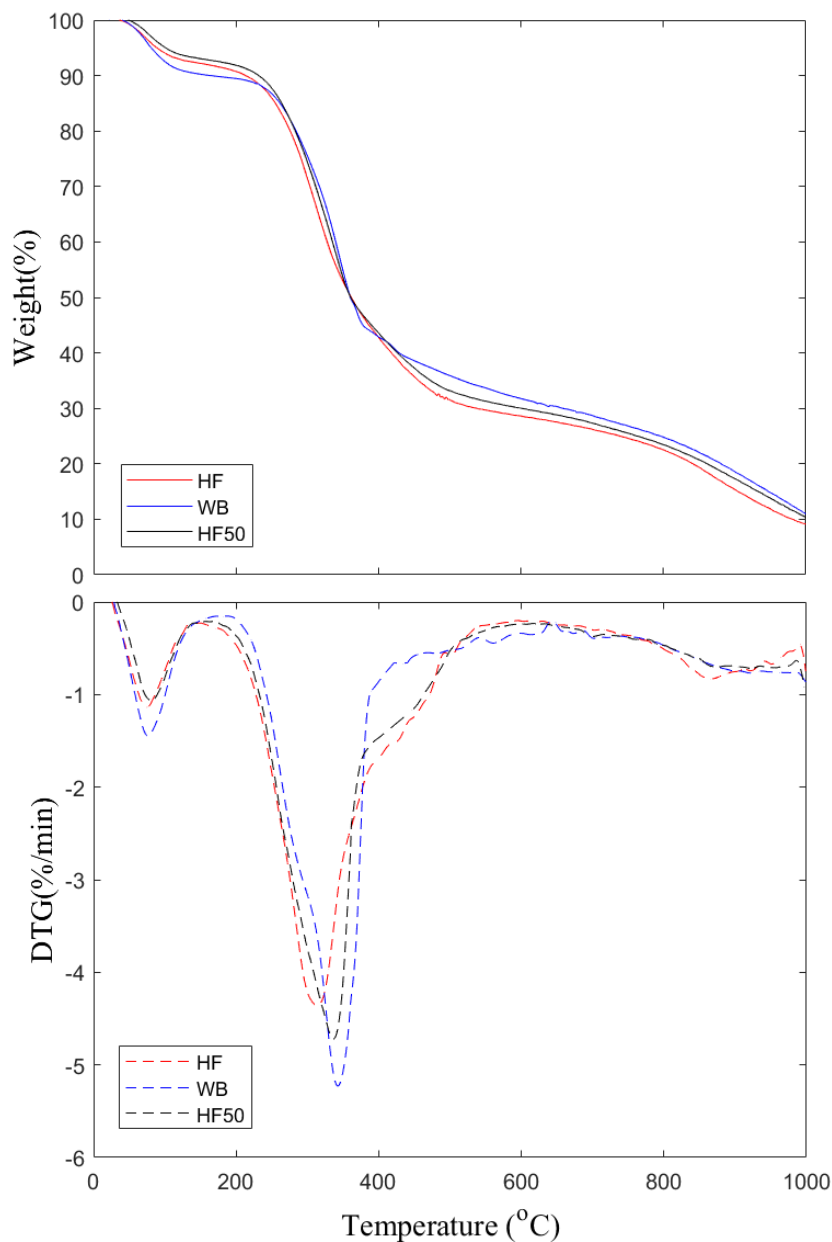


Figure 7.3: Thermogravimetric analysis (TGA) and derivative thermogravimetry (DTG) curves for human faeces (HF), wood biomass (WB) and mixed HF:WB 50:50 (HF50) (up to 1000 °C, 10 °C/min, 50 mL N₂/min).

7.3.2 Biochar stability and carbon storage

7.3.2.1 Preliminary temperature screening

Thermal properties of biochars produced at the different HHTs tested (450, 550, 650 °C)⁹ are shown in **Figure 7.4** and relevant statistical analysis results are included in **Table A12** (Appendix F). The water-extractable dissolved organic carbon (DOC) (**Figure 7.4a**) was used as a preliminary assessment simulating the oxidative nature of biochars applied to the environment. The biochars produced at 450 °C were found to release labile organic carbon, measurable as DOC, at significantly higher concentrations than the other temperatures tested irrespective of the HF:WB ratio (Fang et al., 2014). Biochars produced at 450 °C also had consistently high remaining volatile matter (VM) content (>20%) (**Figure 7.4b**), indicating incomplete carbonisation at temperatures below 500 °C (Ren et al., 2018; Koulouri et al., 2023). Therefore, pyrolysis of HF, WB and HF:WB blends at 450 °C was considered unsuitable for carbon storage applications, as it cannot be safely assumed that the carbon would remain stable long-term, while the high VM content may also have a negative influence on soil properties and environmental safety (e.g. due to the presence of polycyclic aromatic hydrocarbons [PAHs] with phytotoxic effects) (Tomczyk et al., 2020; Odinga et al., 2021). For biochars prepared at 550 °C and 650 °C, the water-extractable DOC dropped to <1 mg/g for all feedstock blending ratios and no statistically significant differences were observed between these temperatures. The low DOC and VM values are indicative of the completion of main pyrolysis reactions after 500 °C, similar to previously reported results for biomass-derived biochars (Gui et al., 2020; Zhang et al., 2022) (also refer to **Sections 5.3.2-5.3.3**).

The WB fraction had a dual beneficial influence upon the ash and fixed carbon content (**Figure 7.4c,d**). The ash content generally increased with temperature, due to the concentration of minerals in the biochar fraction, and decreased with higher WB content, in line with the relative ash content of the feedstock materials (**Figure 7.2**). The fixed carbon content, which is positively correlated to the presence of aromatic, stable forms of carbon (Crombie et al., 2013), increased with WB addition and was maximised at a pyrolysis temperature of 550 °C. Based on these results, wood addition in sanitation facilities can realistically be investigated as a method to increase the carbon-fixing capacity of FS pyrolysis systems, as long as the minimum temperature for complete carbonisation is reached; in this case >500 °C.

⁹ There were no statistically significant differences in biochar characteristics for the various isothermal retention times tested (0.5, 1, 2 h). The results presented in this section correspond to the longest retention time at the HHT (2 h). The heating rate and N₂ gas flow rate were 10 °C/min and 1.5 L/min, as described in **Section 5.2.2**.

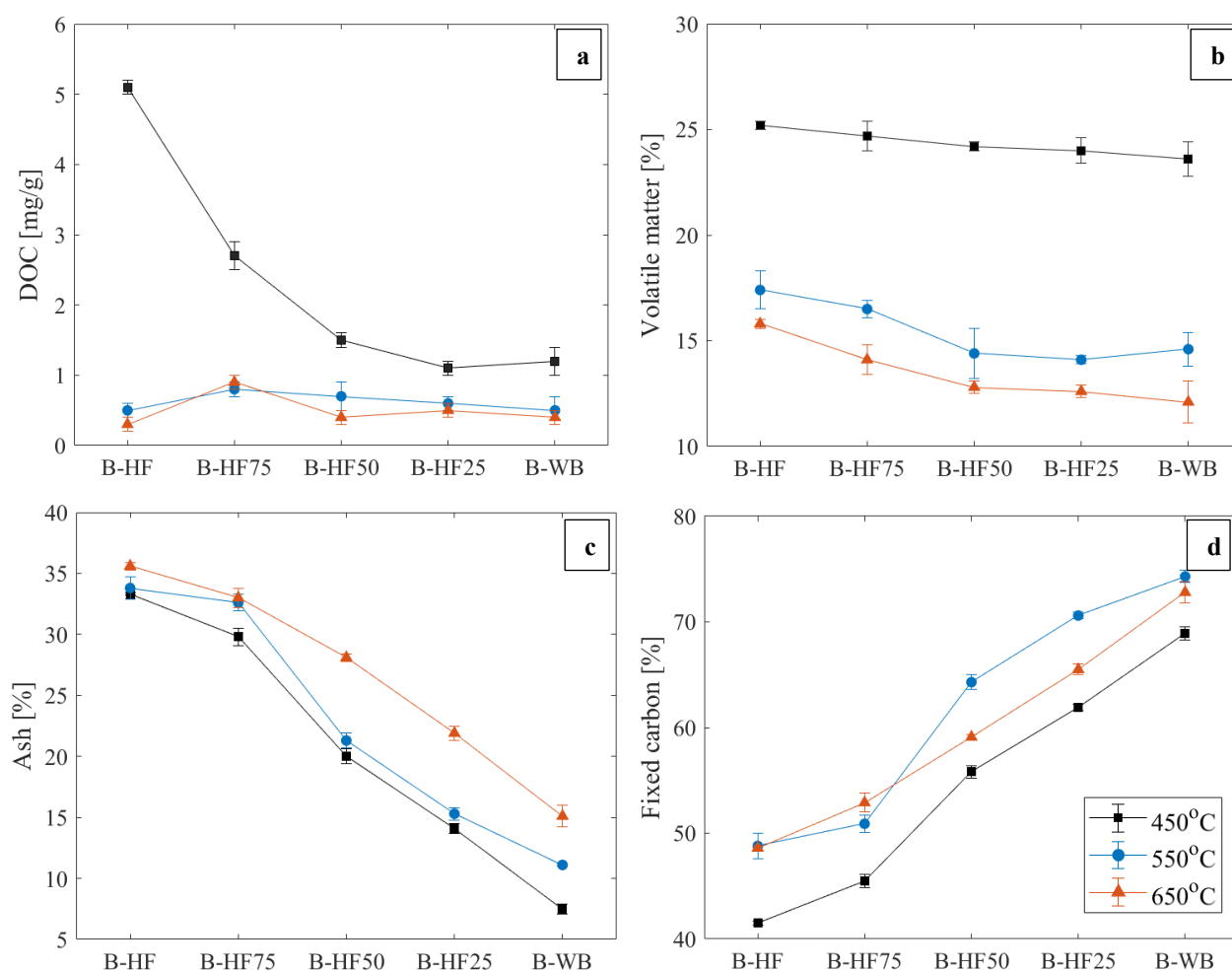


Figure 7.4: Water-extractable dissolved organic carbon (DOC) (a) and proximate analysis results (volatile matter (b), ash (c) and fixed carbon (d)) for biochars B-HF, B-HF75, B-HF50, B-HF25 and B-WB produced at 450 °C, 550 °C, 650 °C (on a dry weight basis), as a preliminary screening of carbon stability. Error bars denote the standard deviation of measured values (n=3).

7.3.2.2 Carbon structure analysis

For the samples prepared at temperatures producing stable biochars (550-650 °C), the CHN(S)O content and H/C, O/C molar ratios are shown in **Figure 7.5** (the measured S values were ≤ 0.2 % for all samples and are not shown). According to international and European standards for biochars, ratios of $H/C < 0.7$ and $O/C < 0.4$ confirm the production of stable, fully pyrolysed biochars (EBC, 2012; IBI, 2015; WBC, 2023). These criteria are met for all biochars produced at these temperatures regardless of feedstock type (also refer to **Section 5.3.2**). According to Spokas (2010), O/C ratios lower than 0.2 indicate at least 1000-year half-life and are associated with the most stable biochars. This threshold was achieved for WB and all HF:WB biochars regardless of blending ratio, but not for pure HF biochars, hence confirming the positive influence of biomass addition in sanitation systems, in terms of long-term biochar stability. Nevertheless, these values are based on the total carbon content that does not necessarily reflect the aromatic structure associated with carbon stability, and therefore, should be complemented with further structural and oxidative assessments (Enders et al., 2012; Leng et al., 2019).

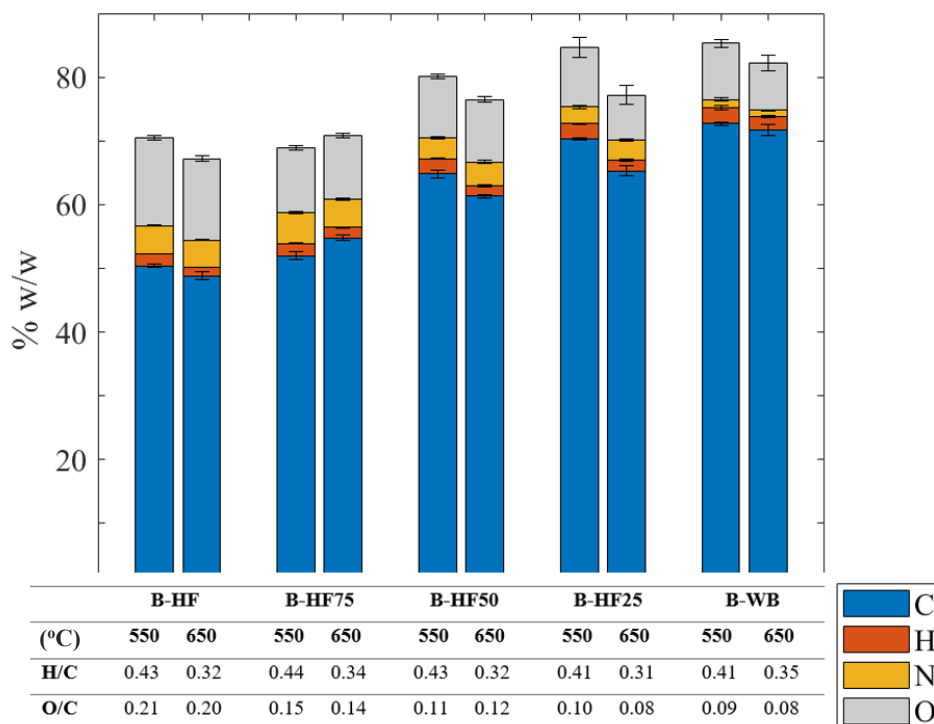


Figure 7.5: CHN(S)O results, and calculated H/C, O/C molar ratios for biochars B-HF, B-HF75, B-HF50, B-HF25 and B-WB produced at 550, 650 °C (on a dry weight basis). The measured S values were $\leq 0.2\%$ for all samples and are not shown at this scale. Error bars denote the standard deviation of measured values ($n=3$).

The change in the nature of carbon present for different HF:WB blending ratios was also observed via SEM analysis (**Figure 7.6**). Biochars containing more HF than WB have a sheet-like texture that is characteristic of the HF-based carbon matrix (B-HF; **Figure 7.6a**), with distinctive mineral constituents that appear as brighter areas, due to compositional contrast (B-HF75; **Figure 7.6b**) (Koulouri et al., 2023). Biochars made from HF50 and HF25 (**Figure 7.6c,d**) exhibited an ordered honeycomb structure with longitudinal cracking that is characteristic of lignocellulosic WB-derived biochars (Zicherman & Williamson, 1981; F. Wang et al., 2022). For HF:WB mixtures, the presence of CaCO_3 on the carbon structure was further observed (B-HF50; **Figure 7.6c**) and confirmed by simultaneous EDX analysis.

The formation of CaCO_3 in faecal biochars can occur spontaneously for Ca-rich biochars. For example, CaCO_3 can be formed during pyrolysis as a result of the decomposition of Ca-containing compounds found in human excreta, such as calcium oxalate (see **Eq. 7.7**) (Haynes et al., 2013; Garland et al., 2020), or during cooling (i.e. when the biochar comes in contact with moisture and CO_2 in the air) based on the reactions shown in **Eq. 7.8-7.9** (Galván-Ruiz et al., 2009; Koulouri et al., 2023). These findings suggest that the high Ca content in HF can enable a supplementary carbon storage mechanism in FS pyrolysis, while the porous structure of WB provides a suitable matrix for the combination of organic and inorganic forms of carbon (Chang et al., 2017; Koulouri et al., 2023).

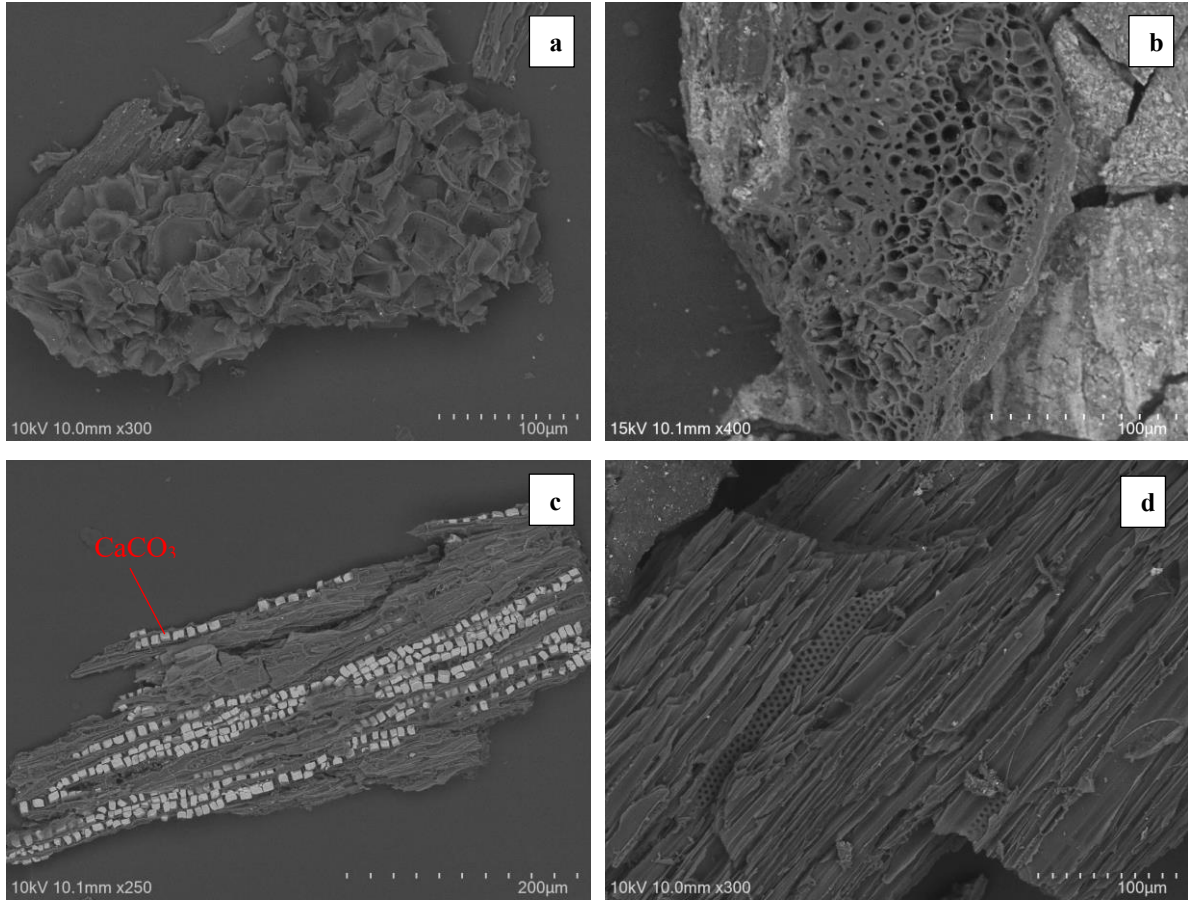
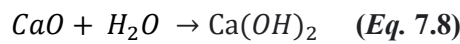
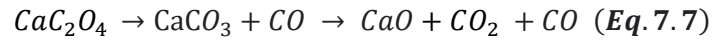


Figure 7.6: Characteristic scanning electron microscopy (SEM) images for biochars B-HF (a), B-HF75 (b), B-HF50 (c) and B-HF25 (d) produced at 550 °C. CaCO_3 precipitates were identified via energy dispersive X-ray spectroscopy (EDX).



FTIR can also be used as a quick method to assess the qualitative changes in chemical bonds and identify the footprints of different constituents (Krueger et al., 2021b). Results are included in **Figure 7.7** and show that C=C stretching vibrations ($1600\text{-}1500\text{ cm}^{-1}$), which are indicative of lignin and aromatic carbon, were stronger for samples containing more WB (Janu et al., 2021). Peaks in the $900\text{-}700\text{ cm}^{-1}$ region (C-H bending), associated with mono-, polycyclic and substituted aromatic compounds, were also stronger for the biochars with more WB, suggesting the enhanced stability of these biochars (Sahoo et al., 2021). In contrast, strong peaks at $1040\text{-}1020\text{ cm}^{-1}$ and 550 cm^{-1} which have been associated with carbohydrates and phosphates, were more intense for the biochars containing more HF and almost eliminated for B-HF25 (Jastrzębski et al., 2011; Krueger et al., 2021b). The presence of carbonates, which was previously validated via SEM analysis, was also confirmed by identified FTIR

peaks ($\sim 1030\text{ cm}^{-1}$, 870 cm^{-1} and vibrations at $1500\text{--}1300\text{ cm}^{-1}$) (also refer to **Section 5.3.3**) (Reig et al., 2002).

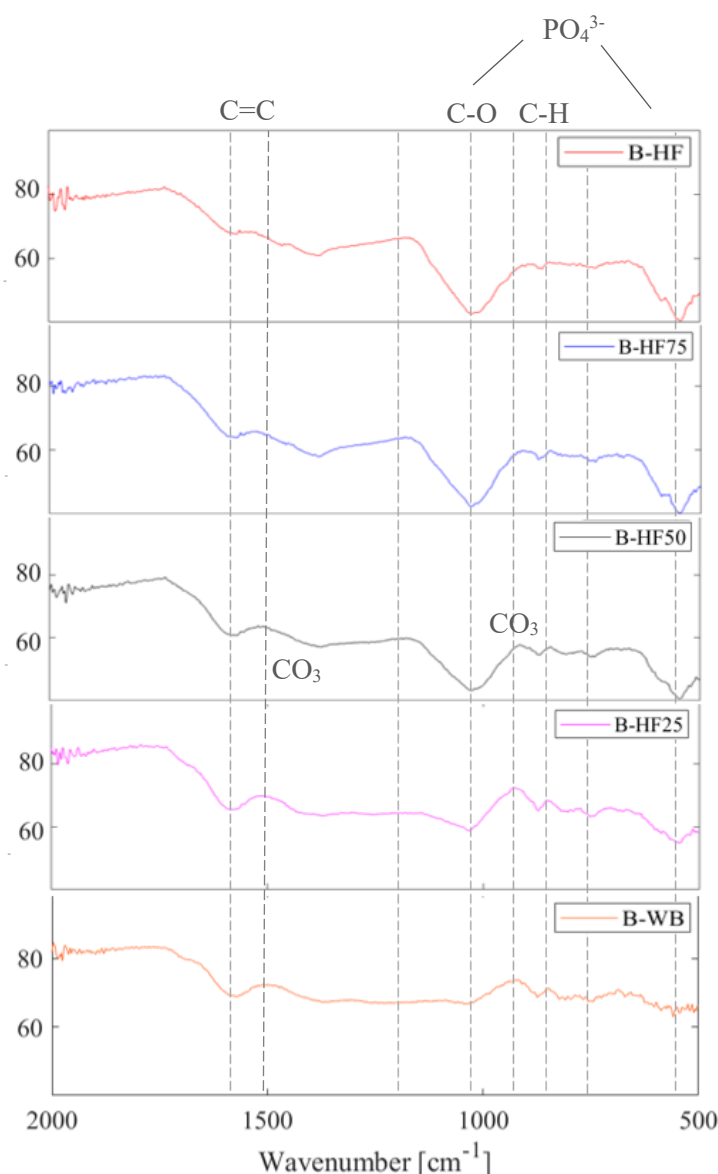


Figure 7.7: Fourier-transform infrared spectroscopy (FTIR) spectra for biochars B-HF, B-HF75, B-HF50, B-HF25 and B-WB produced at $550\text{ }^{\circ}\text{C}$, in the $2000\text{--}500\text{ cm}^{-1}$ wavenumber range. Bands between wavenumbers $4000\text{--}2000\text{ cm}^{-1}$ showing C-H and O-H stretching regions are not shown, due to the limited differences observed among samples and the signal noise due to the presence of moisture.

7.3.2.3 Oxidation resistance analysis

In terms of the carbon chemical stability of the biochars, H_2O_2 oxidation (see **Section 7.2.4.2**) was investigated as an accepted proxy of carbon degradation in natural soils (Cross & Sohi, 2013; Mía et al., 2017). Overall, findings presented in **Figure 7.8a** demonstrate that higher pyrolysis temperature generated biochar with stronger resistance to H_2O_2 oxidation, indicating greater stability due to the release of volatiles and the formation of aromatic C structures (Crombie & Mašek, 2015; F. Wang et al., 2022). However, the difference in carbon stability between the two temperatures tested (550 , $650\text{ }^{\circ}\text{C}$) narrowed with more WB in the feedstock and was negligible between HF50, HF25 and WB, suggesting

that synergistic effects take place during HF:WB co-pyrolysis, in terms of carbon stability. Therefore, 50% WB content is sufficient to alter carbon dynamics and enhance carbon stability, while maximising the quantity of faecal matter safely treated and minimising the required treatment temperature to reach stability.

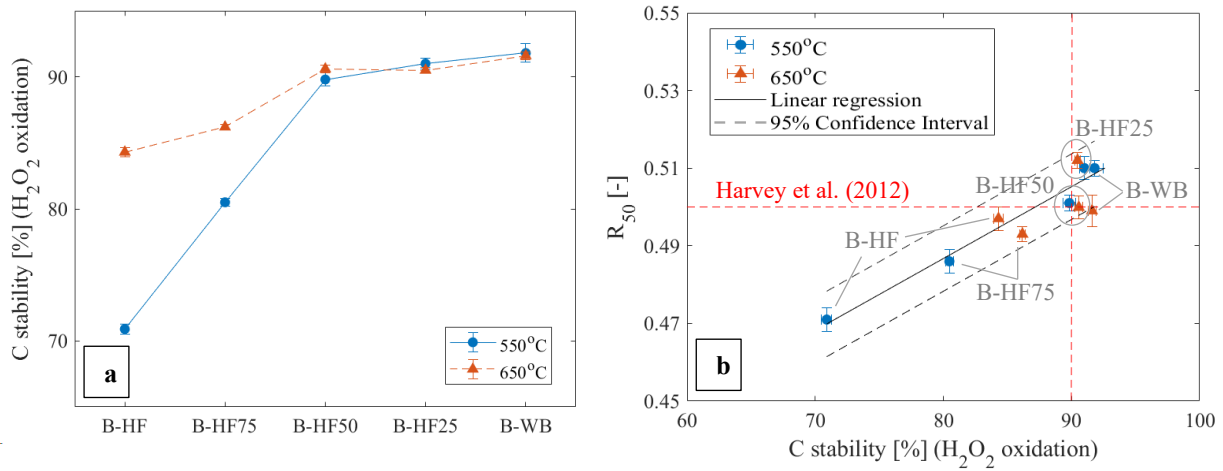


Figure 7.8: (a) Carbon stability (%) (H₂O₂ oxidation method) for biochars B-HF, B-HF75, B-HF50, B-HF25 and B-WB produced at 550 °C, 650 °C; and (b) correlation with the results of the R₅₀ recalcitrance index method. Error bars denote the standard deviation of measured values (n=3).

$$R_{50} = 0.0017 \times CS [\%] + 0.3483 \quad (R^2 = 0.86) \quad (Eq. 7.10)$$

where: R₅₀= Recalcitrance index, CS = Chemical stability by H₂O₂ oxidation

Another established method to assess biochar stability is by investigating its thermal degradation potential in comparison to that of graphite, after applying corrections for moisture and ash content (see **Section 7.2.4.3**). The recalcitrance index (R₅₀) method to assess the stability of plant-derived biochars was proposed by Harvey et al. (2012), and has since been used and modified by other studies (Windeatt et al., 2014; Gómez et al., 2016). According to Harvey et al. (2012) biochars with R₅₀ ≥ 0.7 (Class A) have carbon sequestration potential comparable to graphite, biochars with R₅₀ < 0.5 are comparable to uncharred plant biomass and those with 0.7 > R₅₀ ≥ 0.5 are considered to have intermediate carbon sequestration potential.

Results (**Figure 7.8b**) suggest that biochars produced from HF50, HF25 and WB had R₅₀ ≥ 0.5 for both treatment temperatures (550, 650 °C), while all biochars with <50% WB content did not meet the R₅₀ = 0.5 threshold for adequate stability. Contrary to previous research by Somorin et al. (2020b) reporting that the observed HF:WB synergy is independent of the blending ratio, the R₅₀ and H₂O₂ oxidation results suggest that significant stability synergy takes place when a minimum of 50% WB is included in the feedstock mix. This difference in findings may be a function of the wood type (in this case, willow wood was used), although the exact source/type of wood is not reported by Somorin et al. (2020b).

A strong linear correlation was observed between R_{50} and H_2O_2 oxidation results (**Figure 7.8b** and **Eq. 7.10**) ($R^2=0.86$) and all samples with $R_{50}\geq 0.5$ had H_2O_2 stability $\geq 90\%$, indicating that the threshold suggested by Harvey et al. (2012) can be potentially translated into an oxidation stability threshold value that may be more useful for excreta-derived biochars. The chemical oxidation method showed a wider range of values for different temperatures and feedstock types, with clear statistical significance, while the R_{50} method had a small range of values, potentially due to the applied correction factors to account for high ash and moisture content in faecal biochars, that may hinder the observation of statistically significant differences (ANOVA results shown in **Table A13** – Appendix F). These findings can be useful for at-scale stability assessments or for comparisons among studies, although the correlation relationship should be verified by research with larger sample sizes and additional wood sources.

7.3.2.4 Carbon storage potential

Although all feedstock blends had similar total C content (**Figure 7.2**), the relative proportion in the biochars increased with WB addition (from 50% for B-HF to 70% for B-HF25, produced at 550 °C) (**Figure 7.5**). The measurement of carbon content is a fundamental requirement to calculate carbon mass balances, but relevant analytical methods can be time consuming and require specialised instruments that may not be available in low-income settings. Correlation analyses can provide further information on alternative quick measurements that can be used on-site to compare biochar samples based on their calculated carbon content. The ash content, which is a quick and inexpensive measurement, was suggested by Krueger et al. (2021a) to predict thermally relevant properties of uncharred FS. For this thesis, correlations were investigated for HF:WB biochar samples, and showed that both carbon and fixed carbon values are well predicted based on the ash content of (fully carbonised) biochars, within the 95% confidence interval ($R^2>0.95$) (**Figure 7.9a** and **Eq. 7.11-7.13**). Therefore, the high-level estimation of carbon storage benefits when using FS biochars can be achievable even on-site, for example to quickly screen different feedstock types or operational parameters.

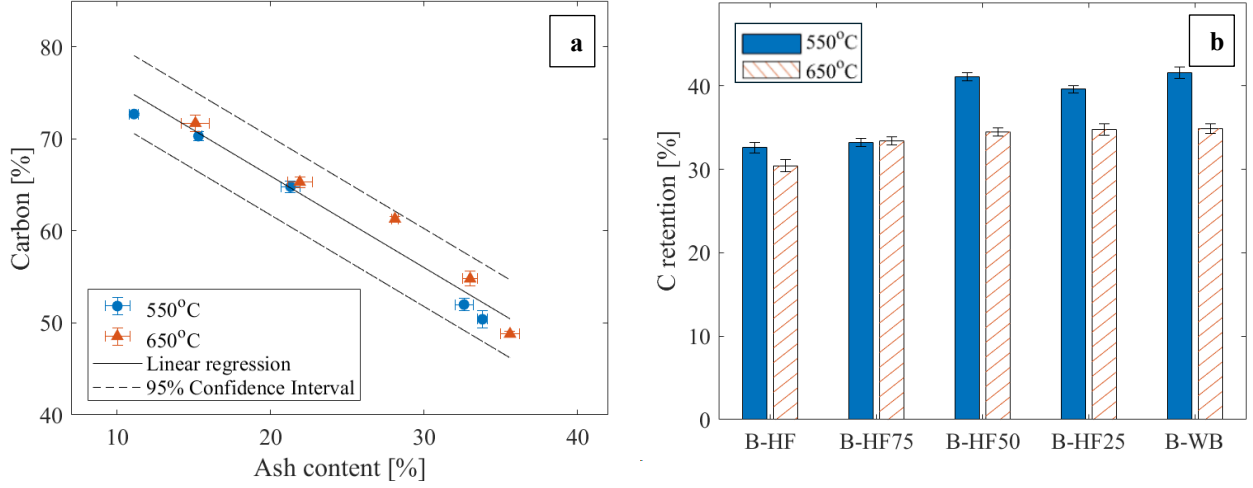


Figure 7.9: (a) Correlation between carbon and ash content (% w/w) in the biochars (based on results shown in Figure 7.4 and Figure 7.5); and (b) carbon retention results (%) calculated based on Eq. (7.1) for biochars B-HF, B-HF75, B-HF50 and B-HF25. Error bars denote the standard deviation of measured values (n=3).

$$C [\%] = -0.9946 \times ASH [\%] + 85.857 \quad (R^2 = 0.96) \quad (Eq. 7.11)$$

$$FC [\%] = -1.1075 \times ASH [\%] + 88.224 \quad (R^2 = 0.98) \quad (Eq. 7.12)$$

$$C [\%] = 0.9035 \times FC [\%] + 6.298 \quad (R^2 = 0.99) \quad (Eq. 7.13)$$

where: FC= Fixed carbon (%), C= Carbon content (%), ASH = Ash content (%)

A more meaningful metric than total C content (**Figure 7.5**) is the C retention % based on **Eq. 7.1** (**Figure 7.9b**), that also takes the biochar yield (**Figure 7.10**) and feedstock C content (**Figure 7.2**) into consideration, and therefore represents the carbon retention in the whole FS pyrolysis system. The biochar yield during pyrolysis decreased with increasing WB content and temperature, from 32% for B-HF (450 °C) to 24.3% for B-HF25 (650 °C), due to the change in ash content and organic composition and the release of volatile compounds (Ippolito et al., 2020). Based on these data, the B-HF50 biochar produced at 550 °C had the highest carbon retention (41.1%) compared to all HF:WB blends (**Figure 7.9b**), despite the higher absolute C concentration in B-HF25 (**Figure 7.5**). In practice, for HF50 more carbon is retained in the system per mass of feedstock treated, while for HF25 more carbon can be stored per mass of biochar applied by the end-user, assuming the biochar is used for agricultural applications or other non-volatilising use (i.e. not as a fuel). From a systemic perspective, HF50 blends had the best performance in terms of carbon retention benefits.

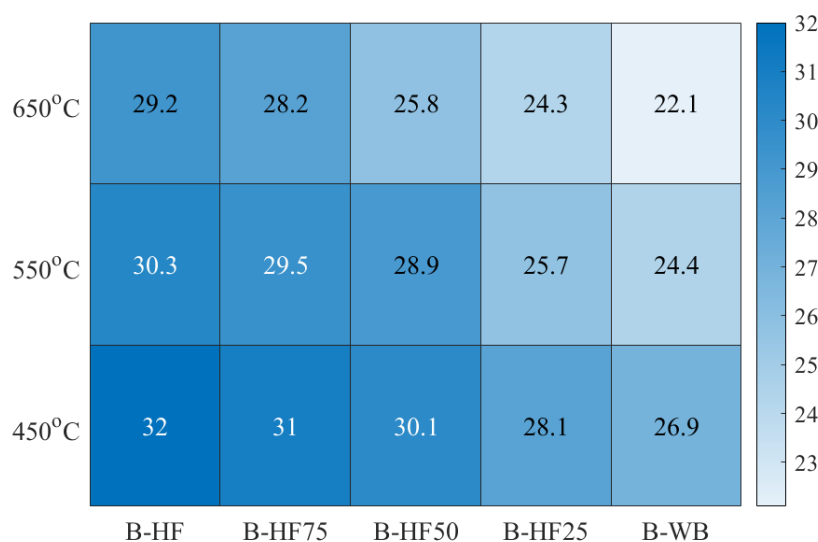


Figure 7.10: Biochar yield for B-HF, B-HF75, B-HF50, B-HF25 and B-WB produced at 450 °C, 550 °C, 650 °C.

For all blends, more C is retained in the biochar at 550 °C compared to 650 °C, indicating that prolonged carbonisation past the temperature where the initial char stability is reached (in this case between 450-550 °C) does not offer additional carbon retention benefits and leads to further carbon loss due to secondary decomposition (carbonisation) reactions (Basu, 2010). These findings highlight the importance of presenting comprehensive carbon management data that also consider operational parameters, biochar yield and feedstock characteristics. The biochar C content can be a misleading figure if used out-of-context, as biochars with higher C concentrations may be produced by systems that have emitted more carbon per mass of feedstock treated.

Overall, the biochar produced at 550 °C by co-pyrollysing HF and WB at a 50:50 ratio had the best performance in terms of both retention and stability of carbon. From a practical perspective, this corresponds to approximately 30 g dry wood being added daily to a toilet as a cover material by each user, according to the expected daily faecal excretion mass (29 g/person/day on a dry basis) (Rose et al., 2015).

7.3.3 Carbon flows distribution during pyrolysis

The distribution of carbon flows among pyrolysis products (biochar, bio-oil, NCG) is shown in **Figure 7.11** for HF, HF50 and WB samples, and for different pyrolysis retention times (0.5, 2 h) and N₂ gas flow rates used (0.5, 1.5 L/min). Preliminary tests showed no significant differences between 1-2 h retention time and 1-1.5 L/min N₂ gas flow rate, hence results for intermediate factor levels are not shown. The chosen HF:WB blend ratio (50:50) and temperature (550 °C) for this analysis were based on previous findings (**Section 7.3.2**), in order to study the change in carbon flows distribution when aiming to maximise carbon stability and retention in the biochar. Further statistical analysis data are included in **Tables A14-A19** (Appendix F).



Figure 7.11: Distribution of carbon flows to biochar, bio-oil and non-condensable gas (NCG) streams expressed as the % of the total carbon content in the feedstock (for human faeces [HF], wood biomass [WB] and HF:WB 50:50 [HF50]) (pyrolysis at 550 °C). Results for different combinations of retention time (RT) at the highest heating temperature (550°C) and N₂ gas flow rate: (a) RT=0.5 h, N₂=0.5 L/min; (b) RT=2 h, N₂=0.5 L/min; and (c) RT=2 h, N₂=1.5 L/min. The reported values are calculated based on Eq.7.1-7.3.

The addition of WB to faecal samples increased C retention in the biochar fraction regardless of the process operational parameters, due to the increase of lignin content in the feedstock mix and the dilution of the fat and protein content of HF which suppressed bio-oil and NCG formation (Abnisa & Wan Daud, 2014; Krueger et al., 2021b; Hubble & Goldfarb, 2021). In terms of operational parameters, the biochar carbon yield reached its maximum values (~40%) when using a low inert gas flow rate (0.5 L/min) that encouraged the recondensation of volatiles on the char fraction, while the isothermal retention time at the HHT did not have a significant influence on solid carbon retention.

Synergy during HF and WB co-pyrolysis was evident in terms of solid carbon retention effects, as there were no significant differences between HF50 and WB for any of the operational parameters tested (ANOVA results shown in **Tables A14-A15** – Appendix F) (i.e. the relationship between the HF:WB ratio and the carbon retention % is not linear). In a theoretical system where no synergistic reactions take place, considering the scenario shown in **Figure 7.11a** where B-HF and B-WB contain 32% and 42% of the total feedstock C respectively, B-HF50 would be expected to contain 37% of the total C. In reality, 41% of the feedstocks' C was retained in B-HF50, confirming the synergistic effect when co-pyrolysing HF and WB.

In terms of oil and gas products, the carbon in the bio-oil fraction increased with decreasing isothermal retention time and inert gas flow rate, while the carbon in NCG followed the opposite trend. Therefore, for traditional pyrolysis applications that may not recover gases for energy/heat recovery, the minimum isothermal retention time and inert gas flow required to achieve full carbonisation and efficient volatile release are preferable to minimise C gas emissions. This is achieved by minimising the solid residence time in the reactor (i.e. short isothermal retention time at the HHT preventing prolonged carbonisation of the formed char) and maximising volatile residence time in the bio-oil condensation system (i.e. slow inert gas flow rate encouraging bio-oil formation from condensable volatiles) (Somerville & Deev, 2020; Bowles et al., 2023). The bio-oil can then be exploited for its energy value as it contains the majority of volatile compounds with higher calorific values (Bond et al., 2018). Interestingly, the bio-oil produced from HF50 contained more C compared to pure WB bio-oil, due to the increased bio-oil yield when co-pyrolysing with a feedstock high in fat (i.e. HF). There was therefore a dual benefit of HF:WB co-pyrolysis, as the WB and HF fractions increase the relative C flows to the biochar and bio-oil products respectively.

For an alternative scenario where the produced gases are exploited for energy or heat recovery, maximising the calorific value of the NCG fraction may be the preferable objective. Ultimately, the optimum choice of operational parameters needs to be considered on a case-specific basis, also depending on the site location and type of kiln used. According to statistical analysis results, the C in the biochar fraction is mainly influenced by feedstock composition, while the bio-oil and NCG fractions are greatly influenced by operational parameters (ANOVA results shown in **Tables A14-A19** –

Appendix F). Findings confirm the influence of chosen operational parameters upon the carbon distribution among FS pyrolysis products (biochar, bio-oil, NCG); this information should be considered when optimising process operation or when comparing the performance of different treatment systems with varying process controls.

For the context of developing countries where traditional kilns are used with minimal operational control, the critical aspects to achieve carbon stability and storage benefits are pyrolysis temperature and feedstock composition respectively (Smebye et al., 2017; Sangsuk et al., 2020). In terms of stability, operators seeking to achieve carbon credit accreditation need to ensure a minimum temperature of $\sim 500^{\circ}\text{C}$ is reached to produce stable biochars. For carbon retention, additional biomass sources, such as wood or agricultural waste, can be used for co-pyrolysis (50:50 ratio recommended) to maximise the amount of carbon contributing to biochar formation. Ultimately, findings indicate that having a condensation system in place to separate bio-oils (to be used for energy generation or heat recovery – subject to further research) and/or exploiting NCG for energy/heat generation and controlling emissions, are also critical aspects towards achieving carbon-neutral FS pyrolysis systems (Karlsson & Nilsson, 2015; Inayat et al., 2022). Efforts to incorporate condensation systems and emission controls into traditional pyrolysis solutions, such as drum kilns, should be investigated by further research, while also considering suitable methods for handling corrosive bio-oils and combustible gases within low-income settings (Namaswa et al., 2023).

7.4 Summary

This chapter has investigated carbon sequestration potential for FS treatment systems that involve co-pyrolysis of HF and WB at different blending ratios HF:WB (100:0, 75:25, 50:50, 25:75, 0:100) and temperatures (450, 550, 650 $^{\circ}\text{C}$). The systematic investigation of analytical biochar stability parameters and the quantification of carbon flows among pyrolysis products (for varying operational parameters) were carried out for the first time in the context of excreta-derived feedstocks. The stability of the produced biochars was assessed based on their remaining volatility, carbon structure (H/C and O/C ratios, SEM and FTIR analyses) and oxidation resistance (chemical oxidation by H_2O_2 and thermal degradation by thermogravimetric analysis [R_{50} index]).

Overall, co-pyrolysis of HF and WB improved carbon fixation and biochar stability, enhancing carbon sequestration potential compared to pyrolysis of pure faecal feedstocks. Biochars produced from 50:50 HF:WB blends at 550 $^{\circ}\text{C}$ had the highest carbon retention (41.1%); this feedstock blending ratio corresponds to ~ 30 g dry wood added in toilets as a cover material (per user/day), based on the expected daily excretion quantities (29 g/person/day on a dry basis) (Rose et al., 2015). For these conditions, the measured H/C, O/C ratios, H_2O_2 oxidation and R_{50} index values suggest that the produced biochars have developed an aromatic structure and are suitable for long-term carbon storage.

The biochar characteristics were more dependent on feedstock composition than pyrolysis temperature – provided the temperature reached was sufficient to ensure completion of the main carbonisation reactions (≥ 500 °C) - while carbon flows to the bio-oil and NCG fractions were significantly influenced by pyrolysis operational parameters (isothermal retention time at the HHT and inert gas flow rate). The formation of CaCO_3 was observed via SEM/EDX and can be further investigated as a potential additional carbon storage mechanism in FSM. The findings of this research can be used to create a methodological dataset to inform carbon assessments and future modelling applications, thereby contributing to the establishment of carbon-neutral (or carbon-negative) sanitation systems.

8 Co-pyrolysis of faecal sludge and biomass waste for resource recovery: A case study in Kampala, Uganda

8.1 Introduction

Currently, only a small number of pyrolysis FS treatment plants (FSTPs) exist around the world, making the statistical interpretation of data on FS pyrolysis challenging (Krueger et al., 2020; Sagor et al., 2022). For low-income countries, operational challenges (e.g. unstable electricity supply) make the adoption of traditional “carbonisation” designs more attractive than commercial pyrolysis kilns, further broadening the variability of operational conditions and hence, product characteristics (Mwamlima et al., 2023). This chapter builds on the findings of previous chapters and investigates their applicability to a real FS carbonisation plant in Kampala, Uganda, where FS is co-treated with biomass waste to produce soil amendments and solid fuels.

The next sections focus on the characterisation of FS char as a potential fertiliser and carbon sequestration medium (**Sections 8.3.1-8.3.2**), and as a constituent of waste-derived solid fuel briquettes (**Sections 8.3.3-8.3.4**). In terms of agricultural uses, char properties were assessed via analytical methods presented in **Chapter 4** and multiple samplings over the course of two years (2022-2023) are presented, allowing the statistical analysis of FS char properties and their variability with time. Regarding solid fuels, the objective was to investigate the composition and calorific value of briquettes produced from co-carbonisation of FS with other biomass materials, that can eventually substitute the use of unsustainable charcoal contributing to deforestation challenges (i.e. the current commercial standard in Kampala).

The results presented in previous chapters are discussed here under the lens of an operating FSTP, allowing the validation of laboratory-based findings and assessing their potential significance for real-world applications. Laboratory data also provided a “best-case scenario” in terms of feedstock purity (i.e. human excreta rather than FS) and operational conditions (i.e. laboratory-scale pyrolysis rather than traditional kilns) that can be used to identify – and quantify – optimisation pathways for real FSTPs. At the same time, limitations of working on an operating FSTP (e.g. varying sample availability and quality; limited control of operational parameters) can be identified as important research parameters for future studies (see **Chapter 10**). Building this knowledge base is essential to allow the critical review of FS pyrolysis applications, which is currently only possible for laboratory data.

A note on terminology

Due to the limited control of feedstock properties, the low carbon content of FS used in this case study (see **Table 8.1**) and the limited operational control during carbonisation, the term “char” is used in this chapter instead of “biochar”, as it cannot be safely assumed that the definition of “biochar” provided in

Section 1.3 is met. According to the IBI (2015), the term “biochar” should be used when the feedstock consists of only biomass and diluents, and includes no more than 2% dry weight contaminants (e.g. fossil fuel-derived compounds, glass, metal objects). Moreover, the term “carbonisation” is used instead of “pyrolysis” to highlight the very slow pyrolysis heating rate, aiming to produce solid char rather than oil and gas products.

8.2 Materials and methods

8.2.1 Location and faecal sludge origin

The sampling location for this study was a briquette production plant set up by Water for People inside the Lubigi Sewage Treatment Plant in Kampala, Uganda (**Figure 8.1**), in partnership with the National Water and Sewerage Corporation (NWSC) of Uganda. The plant treats a combination of FS and sewage with daily capacities of 400 m³ and 5000 m³ respectively. Treatment stages for FS include a screening and grit removal unit, thickening tanks and unplanted drying beds (McConville et al., 2019). The origin of the FS is a combination of pit latrines (lined and unlined) and septic tanks located in Kampala settlements. Properties of the dried FS relevant to this study (thermally relevant properties and concentrations of major nutrients) are shown in **Table 8.1**. Analysis was conducted by Makerere University in Kampala (College of Natural Sciences, Kampala Uganda).

Table 8.1: Proximate analysis, CNS and nutrient content of dried faecal sludge (FS) from the Lubigi Sewage Treatment Plant in Kampala, Uganda (on a dry weight basis).

VM	FC	Ash	C	N	S	P*	K	Ca	Mg
(%)	(%)	(%)	(%)	(%)	(%)	(g/kg)	(g/kg)	(g/kg)	(g/kg)
57.1	7.2	35.7	22.8	2.3	0.14	26.9	6.0	18.9	3.3
(± 14.0)	(± 0.7)	(± 13.8)	(±0.6)	(± 0.4)	(±0.0)	(±7.9)	(±0.4)	(±4.1)	(±0.2)

*P value source: Manga et al. (2022).



Figure 8.1: Aerial view of Lubigi Sewage Treatment Plant located in the Rubaga region in Kampala, Uganda (Map data ©2023 Google Maps).

8.2.2 Carbonisation and briquette preparation

The carbonisation unit consists of a modified air-locked metallic drum kiln, shown in **Figure 8.2a**. For this study, the dried FS (**Table 8.1**) was used as a feedstock for carbonisation, together with other biomass waste (bagasse [BG], sawdust [SD]). The carbonisation process was initiated with available start-up fuels (wood, charcoal or briquettes) (**Figure 8.2b**). Once the dry feedstock was placed in the drum, the kiln was air-locked, samples were left to carbonise overnight and the char was collected the next morning (**Figure 8.2c**). Temperature readings were taken periodically, using a UNI-T portable digital thermometer with a thermocouple attached. Due to the limited operational control possible in these types of traditional kilns, the HHT reached during carbonisation varied, typically between 300-400 °C.



Figure 8.2: Photographs showing (a) the metallic drum kiln in use; (b) process initiation with wood as a start-up fuel; and (c) the produced faecal sludge char. Source: Water for People Uganda (used with permission).

For the briquette production, ground char samples (**Figure 8.3a**) were combined with binders (clay and molasses) before mechanical extrusion. The mixing ratio varied and typically was: 3-4 L molasses, 6-10 kg clay, 40 L water and 200 kg char. Briquettes were produced using a screw press extruder (**Figure 8.3b**) and were placed on drying racks to air dry (**Figure 8.3c**).

8.2.3 Sampling protocol and experimental design

After each batch of carbonisation, char samples were ground into a fine particle size (typically <2mm) (**Figure 8.3a**) and homogenised before sample collection. To ensure samples were representative, carbonisation runs for each feedstock type were repeated three times, on different days, and all analysis was conducted for 3 replicates (**Figure 8.4a**). Two sampling campaigns were carried out, in February 2022 and May 2023. During the 2nd sampling campaign, three samples (≥ 300 g) were collected and analysed for each run to minimise the effect of outliers on statistical power (i.e. 3 samples on-site x 3 laboratory sub-samples = 9 replicates x 3 runs) (**Figure 8.4b**). These data allowed the comprehensive statistical analysis of FS properties based on their variance over the course of two years.



Figure 8.3: Photographs showing (a) ground char ready for extrusion; (b) the briquette extruder (Source: Water for People Uganda [used with permission]); and (c) produced briquettes on drying racks [photo taken April 2024].

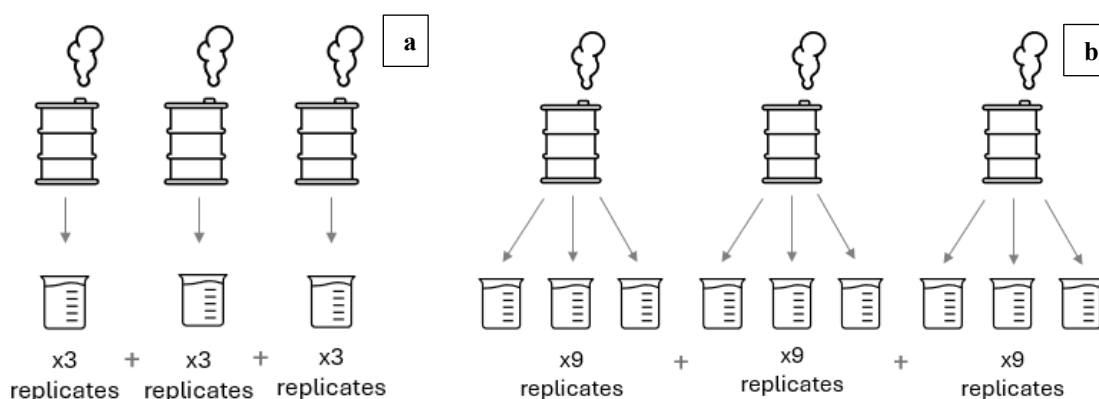


Figure 8.4: Sampling protocol for char samples produced in Kampala, Uganda in 2022 (a) and 2023 (b).

Locally available commercial charcoal dust (CD) was used as the baseline for the assessment of char fuel properties and briquette characteristics. Briquette production and sampling were also conducted in triplicate. The detailed description of produced char and briquette samples is shown in **Table 8.2**. Sample names for char mixtures and briquettes are referred as FS:X r1:r2, where X is the biomass type (BG, SD, CD) and r1, r2 are the mixing ratios for FS and biomass materials respectively.

Table 8.2: Description of char and briquette samples prepared for this study at the carbonisation and briquette production plant set within the Lubigi Sewage Treatment Plant in Kampala, Uganda.

ID	Sample description
FS char	Faecal sludge char
BG char	Bagasse char
SD char	Sawdust char
CD char	Charcoal dust*
FS:BG 50:50 char	Char from a 50:50 mixture of faecal sludge and bagasse (% w/w)
FS:BG 40:60 char	Char from a 40:60 mixture of faecal sludge and bagasse (% w/w)
FS:SD 40:60 char	Char from a 40:60 mixture of faecal sludge and sawdust (% w/w)
FS:BG 50:50 briquette	Briquette produced from FS:BG 50:50 char (incl. binders**)
FS:BG 40:60 briquette	Briquette produced from FS:BG 40:60 char (incl. binders**)
FS:SD 40:60 briquette	Briquette produced from FS:SD 40:60 char (incl. binders**)
FS:CD 50:50 briquette	Briquette produced from FS:CD* 50:50 char mixture (incl. binders**)
FS:CD 40:60 briquette	Briquette produced from FS:CD* 40:60 char mixture (incl. binders**)
FS:CD:SD 40:40:20 briquette	Briquette produced from FS:CD*:SD 40:40:20 char mixture (incl. binders**)
FS:CD:BG 40:40:20 briquette	Briquette produced from FS:CD*:BG 40:40:20 char mixture (incl. binders**)

*Commercially available charcoal dust, as an assessment baseline. **Typically, 3-4 L molasses (C= 48.3%, H= 4.4%, N= 0.8%, S= 0.8%, HHV= 12.8 MJ/kg), 6-10 kg clay (C= 0.5%, H= 0.9%, N= 0.2%, S= <0.1%, non-combustible), 40 L water and 200 kg char.

8.2.4 Analytical methods and statistical analyses

The analytical methods used to characterise char and briquette samples are presented in **Section 4.3**. Experiments were conducted to determine the nutrient and heavy metal content of FS chars, as well as the solid fuel value of all available chars (FS, BG, SD, CD) and briquettes. Carbon stability experiments were conducted for two FS:BG¹⁰ mixing ratios (50:50 and 40:60), based on the analytical methods described in **Section 7.2.4**. For **Sections 8.3.2-8.3.5**, composite homogenised samples (FS, BG, SD, CD) were used for char and briquette preparation, and hence, the arithmetic mean and standard deviation values are presented (refer to **Section 4.3.4**). Results were analysed by one-way ANOVA to assess the influence of feedstock type on char/briquette properties (0.05 significance level). For **Section 8.3.1**, where the occurrence of outliers was observed, median values are presented instead of the arithmetic mean. The variance of FS char characteristics between the two sampling periods (2022 and 2023) was investigated using the non-parametric Mann-Whitney U test (see **Section 4.3.4**).

¹⁰ Carbon stability experiments were conducted only for FS:BG blends (i.e. not for SD blends) and for two mixing ratios (50:50, 40:60), due to operational restrictions at the FSTP in Kampala (incl. limited sawdust availability at the time of sampling).

8.3 Results and discussion

8.3.1 Agricultural value of faecal sludge char

Agricultural uses of char-based fertilisers and soil amendments are widely recognised as a primary resource recovery objective for FS, due to the inherently high nutrient content of human excreta that further concentrates in the char fraction (Woldetsadik et al., 2017; Koulouri et al., 2023). Here, the nutrient content of FS-derived chars is presented for the case study of Kampala and compared against chars produced from pure human excreta in the laboratory (in the UK). The collected data were statistically interpreted for two consecutive years. The heavy metal content of the produced chars was also assessed.

8.3.1.1 Nutrient content

The nutrient content of chars produced in Kampala, Uganda during 2022 and 2023 is shown in **Figure 8.5**. Average values for chars produced from source-separated faeces (SSF) and mixed urine and faeces (MUF) at various temperatures (from **Chapter 5**) are also included. Observed differences can be indicative of the differences between FS versus human excreta and between traditional carbonisation units versus fully controlled pyrolysis systems.

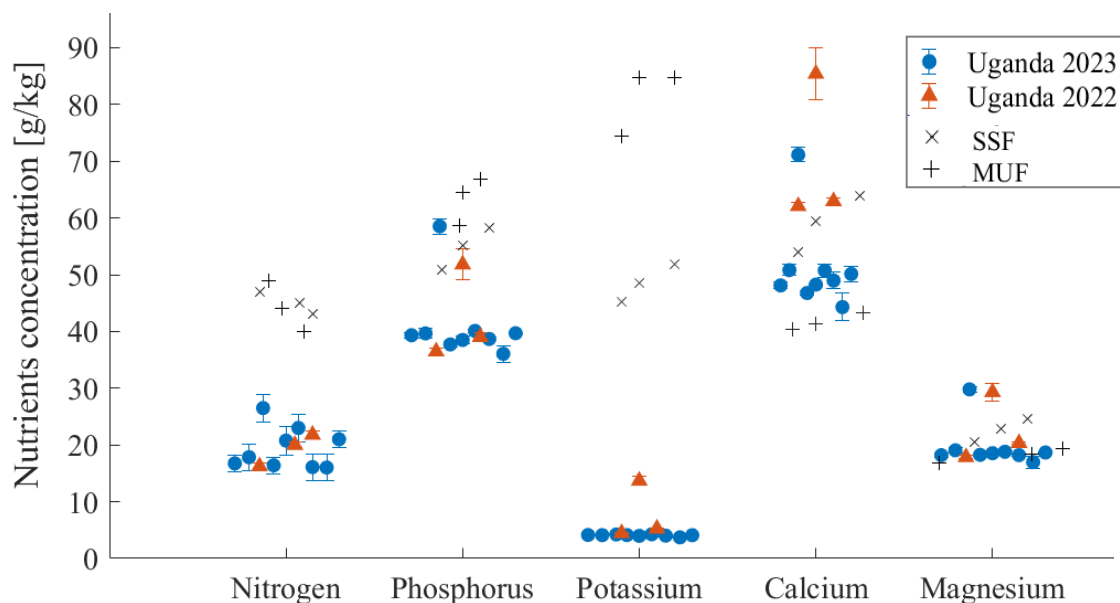


Figure 8.5: Nutrient (N, P, K, Ca, Mg) concentrations (g/kg) of FS chars produced in Uganda in 2022 and 2023, and biochars produced from source-separated faeces (SSF) and mixed urine and faeces (MUF) (as presented in Chapter 5). Results are on a dry weight basis. Error bars denote the standard deviation between sub-samples (n=3).

The N, P, K and Mg content of chars produced in Uganda was relatively consistent for both years of sampling (see statistical analysis results in **Table A20** – Appendix F), although outliers were observed, highlighting the importance of comprehensive quality control testing (WBC, 2023). Calcium exhibited the highest variability over time, which may be influenced by dietary factors and the diverse types of containment facilities found in Kampala (including septic tanks and pit latrines that may be lined or unlined) (Rose et al., 2015; Atwijukye et al., 2018). Based on site observations, sampling may yield extreme outliers due to contamination, inadequate homogenisation or fluctuations in feedstock characteristics and treatment conditions. Multiple samplings are recommended for FSTP testing to detect outliers and observe statistically significant differences in nutrient content (WBC, 2023).

The median P value of 39 g/kg is significantly higher than animal manure and other commonly used organic fertilisers, showing promise for agricultural applications (Leip et al., 2019; Rayne & Aula, 2020). Nevertheless, P concentrations were lower than in biochars derived from pure human excreta (SSF, MUF – see **Chapter 5**), likely due to the inclusion of additional waste materials in traditional on-site sanitation facilities, as well as the variability of operating conditions during treatment (e.g. lower temperature reached) (Crombie et al., 2013; Strande et al., 2014). Separation of soluble nutrients during upstream FSTP stages (e.g. thickening) can also lower the nutrient concentrations of FS chars.

The FS chars were low in N (median value 19 g/kg), meaning that additional N fertilisation would likely be required for agricultural applications (Krounbi et al., 2018). Urine diversion followed by combined char and urine applications, or char enrichment with urine-derived nutrients (see **Chapter 6**) could significantly boost the N content of FS-based fertilisers. For K, a difference of multiple magnitudes can be observed between chars produced in Uganda and the ones produced in the laboratory (in the UK) from pure human excreta. This gap may be attributed to dietary nutrient deficiency factors as the occurrence of K scarcity in East African soils has been reported in various instances (Henao & Baanante, 1999; Brownlie et al., 2024). Therefore, the consideration of regional characteristics when comparing results from different studies is essential. For Mg, no statistically significant differences were observed between FS and excreta-derived chars, while the high variability of Ca values made it impossible to observe statistically significant differences for this element.

Overall, findings confirm the agricultural value of FS chars, while bringing light to the requirement for comprehensive quality control and feedstock homogenisation. Improving the consistency of char properties will be vital for upscaling of resource recovery applications (Strande et al., 2014). Based on the comparison with pure human excreta samples, the implementation of CBS or urine-diverting facilities to accommodate future sanitation infrastructure would provide an opportunity to maximise the NPK content of FS-derived chars. Existing FS sources from traditional on-site containment facilities remain a valuable nutrient-rich material that is deemed suitable for nutrient recovery via pyrolysis, particularly for P.

8.3.1.2 Heavy metals

The heavy metal content of produced FS chars is shown in **Figure 8.6**, also with reference to SSF/MUF samples from **Chapter 5**, and threshold values from international and national standards in Uganda (IBI, 2015; UNBS, 2017; WBC, 2023). Generally, results correspond well to previous findings from **Chapters 5** and **6** reporting that Zn and Cu are potential elements of concern for excreta-based chars. Variability among samples and between the two years of sampling was observed for all detectable trace elements (see statistical analysis results in **Table A21** – Appendix F), suggesting that heavy metal testing should be repeated on a regular basis at FSTPs producing chars for agricultural uses.

Where limit values for heavy metals are set by the Uganda National Bureau of Standards (UNBS), specifically about lead (Pb) and chromium (Cr), these were satisfied for all samples, supporting agricultural applications of FS char in Uganda (UNBS, 2017). For Zn and Cu, while there are currently no Ugandan standards available, measured values complied with the maximum allowed values suggested by the IBI but were higher than the lower limits published (IBI, 2015). Guidelines from the WBC were generally met, but outlier values were identified (WBC, 2023).

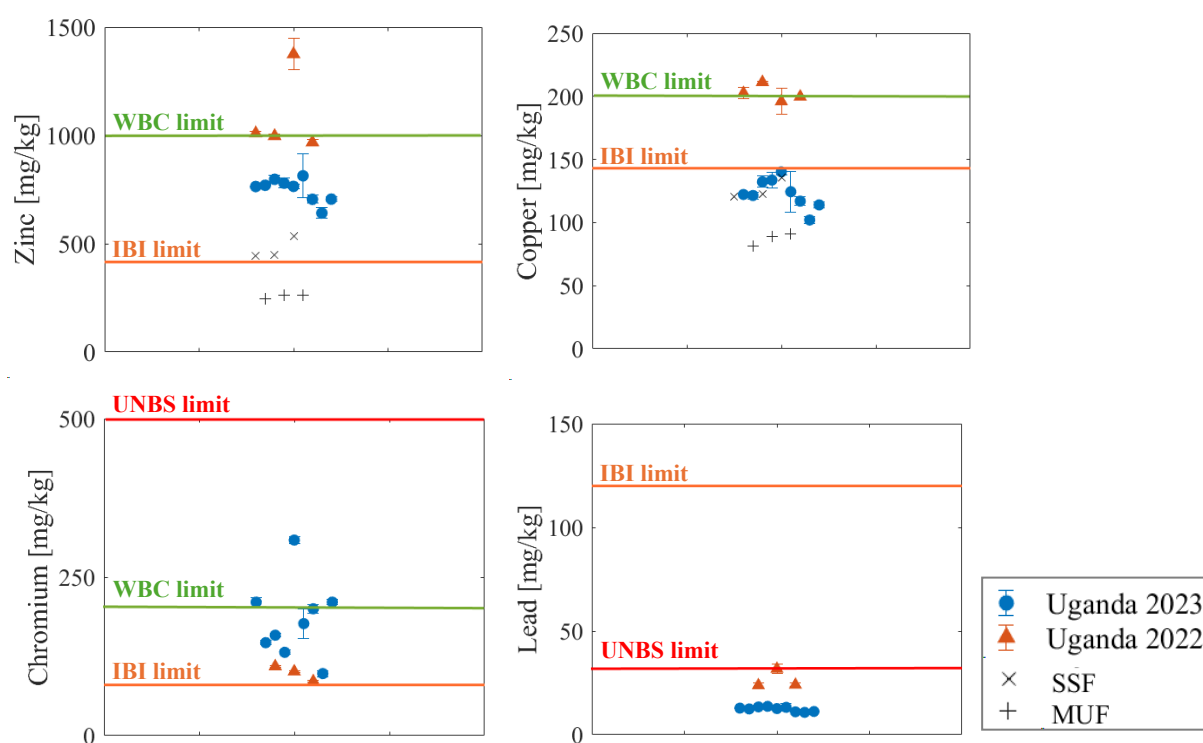


Figure 8.6: Heavy metal (Zn, Cu, Cr, Pb) concentrations (mg/kg) of FS chars produced in Uganda (2022, 2023) and biochars produced from source-separated faeces (SSF) and mixed urine and faeces (MUF) (as presented in Chapter 5), with reference to limit values provided by the International Biochar Initiative (IBI, 2015), the World Biochar Certificate (WBC) (WBC, 2023) and indicative Ugandan standards (UNBS, 2017). Results are on a dry weight basis. Error bars denote the standard deviation between sub-samples (n=3). For SSF and MUF samples, Cr and Pb values were below the detection limit.

As local requirements are not specific to char fertilisers and only provided guidelines for two of the heavy metals identified in these samples (UNBS, 2017), there is still vagueness around the regulatory framework of heavy metal toxicity from char applications. The WBC and IBI recommendations, as a compilation of the maximum allowed values for various countries, can be used as guidance rather than requirements built for local applications in Uganda (Mortvedt, 2005). It is also worth noting that the relative rigorousness of individual standards is inconsistent, i.e. the IBI has a stricter acceptable limit for Cr, but the UNBS has a stricter limit for Pb. Interestingly, some countries have started to introduce limits based on the nutrient-to-heavy-metal ratio, rather than absolute heavy metal concentrations (WBC, 2023). This approach is suitable for concentrated char fertilisers that may require smaller application rates compared to compost or other organic fertilisers.

Results for human excreta samples were generally lower than the FS chars, suggesting that potential elevated values in FS are not attributed to excreta but additional waste discarded in on-site sanitation facilities (Ahmed, 2018). It has been previously reported that human excreta contain fewer heavy metals than FS and sewage sludge, supporting the hypothesis that the purer the source of human excreta, the less toxicity risks from heavy metals can be expected (Gold et al., 2017; Bleuler et al., 2020). Biomass cover materials added in on-site toilets (e.g. sawdust) can further reduce the heavy metal content of sanitation outputs and recovered products (refer to **Section 5.3.3.1.2**) (Krueger et al., 2020). Therefore, better source control and raising awareness of sanitation users to minimise additional waste being discarded in toilets may be successful interventions to decrease heavy metal content where needed (Bünemann et al., 2024).

8.3.2 Carbon sequestration potential

In terms of the carbon sequestration potential of the chars, the methods presented in **Chapter 7** are applied here for (homogenised) FS chars blended with locally available bagasse and pyrolysed using a traditional carbonisation kiln. As the proposed carbon stability methods constitute a novel assessment protocol for FS chars, this section is a valuable validation step using data from a real case study. Results are relevant to agricultural applications, as combined soil improvement and carbon storage benefits can be achieved when applying chars to soil (Mandlebaum & Nriagu, 2011).

Table 8.3 summarises some key stability indicators for chars, including H/C ratios, recalcitrance index (R_{50}) values and chemical oxidation stability results (H_2O_2 oxidation) (ANOVA results included in **Table A22** – Appendix F). The average H/C ratio of FS chars was >0.7 , suggesting low char stability and potentially incomplete carbonisation (Leng et al., 2019). This is confirmed by the low chemical stability value for FS chars, with only 26.4% of the total carbon being resistant to oxidation. Therefore, co-treatment with lignocellulosic biomass was deemed essential for the enhancement of char stability (Bleuler et al., 2020; F. Wang et al., 2022). Bagasse (BG) was considered as a suitable biomass source, as it is an abundant agricultural waste in Uganda.

Table 8.3: The H/C ratio (-), recalcitrance index (R_{50}) (-), H_2O_2 oxidation carbon stability (%) and stable C content C_{STABLE} (%) results for faecal sludge (FS), bagasse (BG) and FS:BG blends (50:50 and 40:60).

	H/C [-]	R_{50} [-]	H_2O_2 [%]	C_{STABLE} [%]
FS	1.05 ± 0.08	0.50 ± 0.02	26.40 ± 0.71	4.59 ± 0.75
FS:BG 50:50	0.61 ± 0.06	0.52 ± 0.03	73.04 ± 1.57	24.28 ± 1.95
FS:BG 40:60	0.58 ± 0.03	0.55 ± 0.04	74.2 ± 1.44	27.97 ± 0.67
BG	0.56 ± 0.02	0.55 ± 0.04	76.26 ± 1.53	42.17 ± 2.44

For FS:BG blends, the observed H/C ratios shifted to <0.7 and H_2O_2 stability values ($>70\%$) were statistically different from pure FS but not BG chars (see ANOVA results in **Table A22** – Appendix F). Therefore, synergistic benefits during co-carbonisation of FS with lignocellulosic biomass can be realistically expected and a 50:50 mixing ratio was deemed sufficient to enhance carbon stability, in line with **Chapter 7** findings for human excreta. SEM images also confirmed the change in structure between FS and FS:BG 50:50 chars (**Figure 8.7**). Based on results presented in **Chapter 7**, further process improvements and technical modifications to increase the treatment temperature to $\sim 500^\circ\text{C}$ are expected to increase the carbon stability of produced chars (see **Section Error! Reference source not found.**).

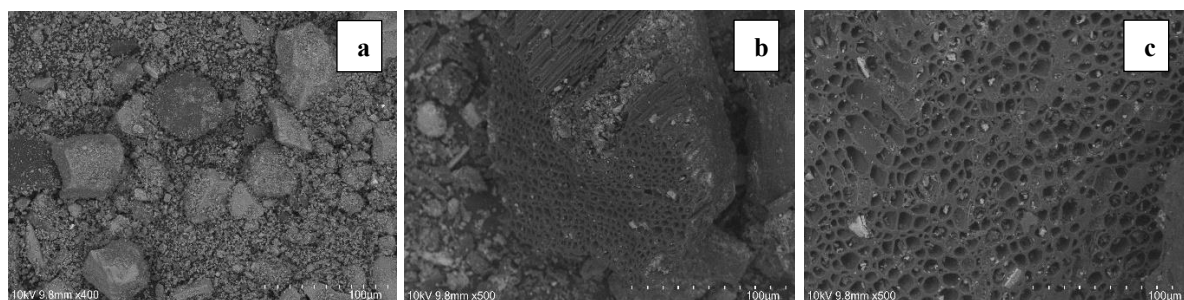


Figure 8.7: Scanning electron microscopy (SEM) images for chars produced from faecal sludge (FS) (a), FS:BG 50:50 (b) and pure bagasse (BG) (c), showing the change in carbon structure for FS:BG blends.

The carbon retention during pyrolysis could not be calculated for these samples, as carbonisation yields were very variable and often not properly documented. Therefore, the concentration of stable carbon in the char was used as an alternative metric (**Table 8.3**). In comparison to pure BG char, the presence of FS significantly decreased the absolute amount of carbon available for storage, regardless of the mixing ratio. This is attributed to the high ash content of FS chars (see **Table 8.4**), which inevitably leads to a dilution of the char carbon content, regardless of synergistic effects for carbon stability. Notably, this FS was collected from traditional on-site sanitation systems that are not source-separating and include significantly more ash than pure human excreta (Strande et al., 2014).

According to results presented in **Chapter 5**, a two-fold increase in char C content would be expected if a CBS toilet was used as the source of FS (i.e. similar to human excreta composition), and a three-fold increase in C content can be expected for source-separated human faeces. At least 70% of this C can be considered suitable for long-term storage, based on the H_2O_2 stability data reported in **Table 8.3** for FS:BG blends and **Chapter 7** findings for pure faeces. Therefore, efforts to minimise the ash content

of FS sources (via source control and during treatment – e.g. avoiding sand drying beds) are recommended to maximise carbon sequestration potential during pyrolysis of FS.

A note on the recalcitrance index method (R_{50})

The recalcitrance index method (R_{50}) was designed to evaluate char stability by comparing its thermal degradation against that of graphite (Harvey et al., 2012). In **Chapter 7**, it was used to assess the stability of chars produced from the co-pyrolysis of human faeces with wood biomass, and results showed good correlation with the chemical oxidation method (H_2O_2 oxidation) (Cross & Sohi, 2013).

The data presented in **Table 8.3** suggest that the R_{50} method may not be suitable for high-ash chars, such as these produced using FS derived from pit latrines and septic tanks. All tested samples had R_{50} values above the threshold of intermediate carbon sequestration potential ($R_{50} > 0.5$), despite high H/C ratios and low chemical stability identified for FS chars. While the method allows for moisture and ash content corrections (see **Section 7.2.4.3**), these calculations may lead to skewed stability results for very high-ash samples, such as FS chars, that are significantly different to the chemical composition of graphite. As a result, no statistically significant differences were observed between samples (see ANOVA results in **Table A22** – Appendix F). Further investigation is recommended for larger sample sizes and different operational conditions (e.g. higher pyrolysis temperatures). Overall, the data presented in this thesis suggest that the chemical oxidation method is more reliable for the assessment of highly variable chars encountered in FSTPs, compared to the recalcitrance index method that was designed for carbon-rich materials.

8.3.3 Solid fuel value of char samples

The solid fuel value of char samples was investigated on account of the fluctuating prices of charcoal and the high demand for waste-derived solid fuels in Kampala. Thermally relevant properties of chars produced from dried (homogenised) FS, BG and sawdust (SD), as well as for commercial charcoal dust (CD) – as the baseline for comparison – are presented in **Table 8.4** (ANOVA results in **Table A23** – Appendix F).

Table 8.4: Proximate analysis, CHNS and calorific value (higher heating value [HHV]) results for char samples produced from faecal sludge (FS), bagasse (BG), sawdust (SD) and charcoal dust (CD) (as received basis).

	MC (%)	VM (%)	FC (%)	ASH (%)	C (%)	H (%)	N (%)	S (%)	HHV (MJ/kg)
FS	3.6 ±0.9	19.6 ±1.9	7.9 ±1.8	68.9 ±2.0	17.4 ±0.5	1.6 ±0.2	1.9 ±0.3	0.8 ±0.5	6.4 ±1.1
BG	3.0 ±0.8	31.1 ±0.7	45.4 ±0.9	20.5 ±1.4	55.3 ±2.1	3.2 ±0.1	0.4 ±0.1	0.2 ±0.0	20.7 ±0.9
SD	4.2 ±0.7	29.8 ±1.4	34.4 ±1.2	31.6 ±1.2	53.3 ±2.4	4.6 ±0.6	0.3 ±0.1	0.4 ±0.1	18.1 ±2.1
CD	4.4 ±0.6	19.6 ±0.6	56.2 ±1.4	19.8 ±1.7	58.5 ±0.9	2.4 ±0.5	1.1 ±0.2	0.2 ±0.1	22.4 ±1.6

The produced FS chars had high ash content (~70%) and an average HHV of 6.4 MJ/kg; approximately half the HHV recorded for human excreta chars (~13 MJ/kg – see **Section 5.3.3.2**). These results highlight the deviation of FS characteristics from pure human excreta, while also confirming the need for co-treatment with additional biomass materials of higher calorific value to produce solid fuels that can compete with commercial standards (Sagor et al., 2022). As an indication, the baseline CD tested in this study contained ~20% ash and had an average HHV of 22.4 MJ/kg, almost four times higher than for FS chars.

Carbonised BG appears to have good fuel properties, with similar characteristics to commercial charcoal (HHV 20.7 MJ/kg) and can therefore realistically be investigated as a potential additive for briquette production. The SD char samples had slightly lower fuel value compared to BG, although this can be partly attributed to operational challenges during carbonisation, based on on-site observations (e.g. higher initial moisture content (MC)). The final MC was similar for all carbonised samples and within FAO standards to avoid breakage and production of char fines during burning (MC <10%) (FAO, 1985). Biomass-derived chars had lower N and S content compared to FS and therefore, their addition can be expected to decrease NO_x and SO_x emissions during burning.

8.3.4 Solid fuel value of briquettes

Figure 8.8 shows the calorific value (HHV) of briquettes produced from different mixtures of carbonised FS and available biomass materials (see **Table 8.4**), relative to an indicative commercial standard (17 MJ/kg) and the value expected for commercial charcoal according to FAO standards (22 MJ/kg) (FAO, 1985; CEN, 2005). It is apparent that the high ash content, and hence low calorific value of FS has a negative effect on briquette quality, with briquettes containing 50% FS not exceeding HHVs of 15 MJ/kg. For briquettes containing 40% FS, the maximum calorific value was 16 MJ/kg for FS:CD 40:60 and 14.5 MJ/kg without the presence of CD (for FS:BG 40:60). These values are below the minimum commercial standard (17 MJ/kg), highlighting that the high ash content of FS is a critical limiting factor for solid fuel production (CEN, 2005).

Despite these results, preliminary assessments of user satisfaction conducted by Water for People were positive, bringing attention to the importance of affordability and accessibility as defining factors for user satisfaction, often despite technical deficiencies. The slow burning properties and low cost of FS-containing briquettes make them an attractive alternative for communities that heavily rely on solid fuels, such as wood (Mwampamba et al., 2013). CHNS analysis (**Table 8.5**) showed low N and S content for all briquette samples (<1.5% and ≤0.2% respectively), confirming that low NO_x and SO_x emissions are expected during burning (Obernberger et al., 2006; Gold et al., 2017). The C content and HHV were higher for briquettes containing BG compared to SD, which can be attributed to the lower ash and moisture content of BG samples (see **Table 8.4**). The low H content of the briquettes indicates that air-drying is sufficient to remove moisture after binder addition.

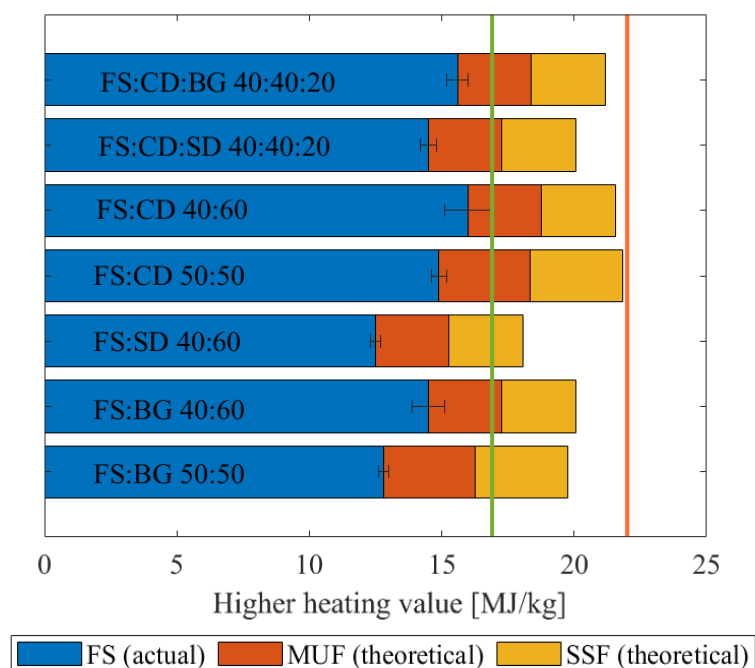


Figure 8.8: Higher heating values (MJ/kg) of briquette samples in comparison to the minimum commercial standard (17 MJ/kg) (CEN, 2005) and the value expected for commercial charcoal, according to FAO standards (22 MJ/kg) (FAO, 1985). The naming convention is described in **Table 8.2**. Theoretical values for briquettes prepared with SSF/MUF samples pyrolysed at 450 °C (instead of FS) are also included (based on **Chapter 5** results). Error bars denote the standard deviation of measured values (n=3).

Table 8.5: CHNS analysis of briquette samples produced from different combinations of faecal sludge (FS), bagasse (BG), sawdust (SD) and charcoal dust (CD). Naming convention described in **Table 8.2**.

	C (%)	H (%)	N (%)	S (%)
FS:CD:BG 40:40:20	44.7 ±0.5	1.5 ±0.2	1.0 ±0.2	0.1 ±0.1
FS:CD:SD 40:40:20	39.4 ±1.5	2.3 ±0.1	1.1 ±0.1	0.2 ±0.2
FS:CD 40:60	48.5 ±1.6	1.9 ±0.2	0.9 ±0.1	0.1 ±0.1
FS:CD 50:50	43.3 ±0.9	1.9 ±0.1	1.7 ±0.3	0.1 ±0.0
FS:SD 40:60	36.6 ±1.8	2.8 ±0.3	1.0 ±0.1	0.2 ±0.0
FS:BG 40:60	42.1 ±0.9	2.0 ±0.4	1.3 ±0.1	0.1 ±0.0
FS:BG 50:50	33.7 ±1.3	2.2 ±0.5	1.1 ±0.1	0.1 ±0.0

Given the high demand for these products, it is essential that efforts for further optimisation of product quality continue, both at the treatment and briquette production level (operational parameters etc.) and via source interventions. Findings from **Chapter 5** can be applied here to estimate the theoretical HHV values if FS from pit latrines and septic tanks was substituted with a) human excreta from enclosed mixed toilets such as CBS (i.e. MUF from Chapter 5); or b) human faeces from source-separating toilets (i.e. SSF from Chapter 5).

Results are included in **Figure 8.8** and show that source interventions can have a positive effect on the fuel value of produced briquettes. When assuming sludge from enclosed containment systems where no other additives except for human excreta and biomass materials are added, almost all briquettes had HHV values >17 MJ/kg (**Figure 8.8**). Further improvements were observed for the urine diversion scenario, in which case briquettes with similar HHV to commercial charcoal (22 MJ/kg) can be realistically produced from human excreta. These results suggest that outputs of CBS toilets may be more suitable for solid fuel production applications, whereas existing high-ash FS sources from traditional on-site containment facilities in Kampala may be better suited to agricultural applications (see **Section 8.3.1**).

8.3.5 Correlation analysis

While the HHV analysis requires specialised equipment, investigating correlations with simple measurements can further support FSTP operations. Several studies have reported data that support a correlation between char ash content and calorific value (Ward et al., 2014; Hübner et al., 2019). Results from previous studies have been plotted in **Figure 8.9** and used for data validation (Ward et al., 2014; Gold et al., 2018a; Krueger et al., 2020). The values measured in this study fall within the 95% confidence interval of the correlation calculated from previous studies (**Eq. 8.1**) and therefore, ash content measurements can be recommended as a quick, inexpensive method to estimate char calorific value. Ward et al. (2014) also provided an equation to calculate HHV based on the CHNSO content of chars, although that is a more complicated measurement requiring fit-for-purpose laboratory facilities. Krueger et al. (2021) calculated a HHV-ash correlation for uncarbonised FS which, notably, is different to the one calculated for char samples, but may be suitable for initial screenings of feedstock materials.

The correlation between ash content and HHV value was also investigated for the briquette samples produced (**Figure 8.9** and **Eq. 8.2**). Notably, while a linear correlation was observed, this is different to the relationship identified for char samples (**Eq. 8.1**). This can be attributed to the presence of additional binding materials in the briquettes (clay and molasses) that alter their composition compared to the chars they originate from. Given the small sample size of briquettes available in this study, it is recommended that the relationship presented in **Eq. 8.2** is validated by future studies on FS-derived briquettes. Nevertheless, the local context, kiln design, operational conditions and individual feedstock properties need to be considered on a case-specific basis (Andriessen et al., 2019).

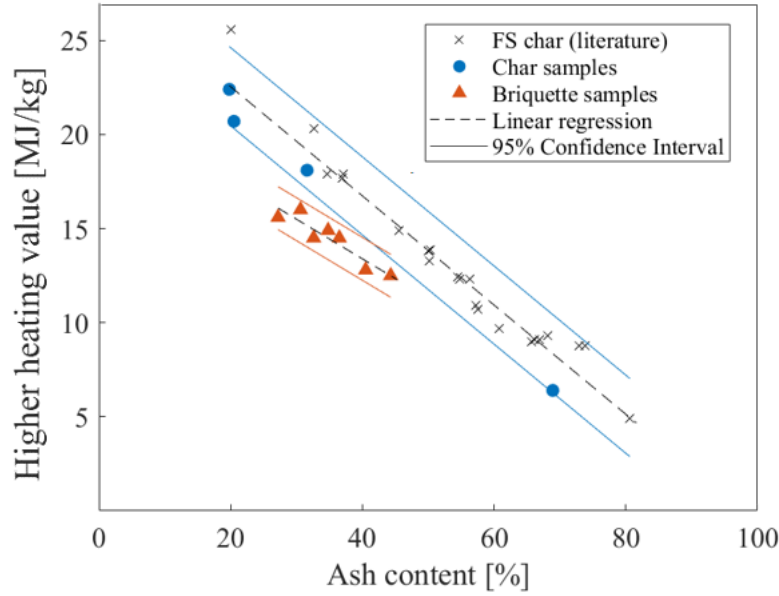


Figure 8.9: Correlation between higher heating value (MJ/kg) and ash content (% w/w) of char and briquette samples, with the inclusion of literature values for FS char samples (Ward et al., 2014; Gold et al., 2018a; Krueger et al., 2020; Koulouri et al., 2023).

$$HHV_{char} [MJ/kg] = -0.2901 \times ASH [\%] + 28.3392 \quad (R^2 = 0.97) \quad (Eq. 8.1)$$

$$HHV_{briquette} [MJ/kg] = -0.2104 \times ASH [\%] + 21.8087 \quad (R^2 = 0.87) \quad (Eq. 8.2)$$

where, HHV_{char} and $HHV_{briquette}$ are the higher heating values (MJ/kg) for char and briquette samples respectively; and ASH (% w/w) is the ash content of the samples.

8.4 Summary

This chapter investigated the operation of a carbonisation FSTP located in Kampala, Uganda where FS is co-treated with biomass waste to produce soil amendments and solid fuel briquettes. Findings were compared to previous results for pure excreta-derived biochars (see **Chapters 5-7**), to allow the investigation of optimisation pathways via feedstock and operational control.

The nutrient value of the chars produced in Kampala was promising for agricultural applications (particularly for P fertilisation) and can be further improved with source control (e.g. urine diversion). Heavy metal levels were below the thresholds set by Ugandan standards (where available), although the need for more comprehensive national (or regional) guidance was identified. Outlier values were observed for almost all elements tested and therefore, regular quality control testing is recommended. Carbon sequestration during soil applications is feasible when FS is co-pyrolised with other biomass sources, such as bagasse, although the high ash content of the FS inevitably diluted the amount of stable carbon available for storage.

In terms of solid fuel value, the high ash content of FS chars (~70% w/w) was the limiting factor for briquette production. Co-pyrolysis with other biomass waste (e.g. bagasse – which is widely available in Uganda) successfully increased the calorific value of briquette samples, allowing the production of marketable briquettes from FS (HHV 14.5 MJ/kg for the FS:BG 40:60 ratio). Further optimisation can be expected with source control (HHV >20 MJ/kg for outputs of source-separating toilets mixed with biomass).

Overall, the data presented in this chapter validated the applicability of laboratory-based findings for practical applications, while also bringing attention to operational challenges observed for traditional carbonisation kilns that cannot be observed for laboratory studies. Results are relevant to both sanitation practitioners and researchers, aiming to bridge the gap between fundamental research and real-world applications, and build a database that allows the statistical interpretation of FS char properties.

9 Discussion

This research investigated resource recovery via pyrolysis of human excreta, by exploring interventions that aim to improve the circularity of sanitation systems. In this chapter, the implications of the research for practical applications and decision-making within FSM will be interpreted and discussed, drawing on the findings presented in previous chapters. Firstly, the role of source control in sanitation, a core theme of this thesis, will be discussed under the context of N retention and circular nutrient management (**Section 9.1.1**), as well as carbon management advantages of human excreta pyrolysis (**Section 9.1.2**). Then, some key considerations to transition from laboratory-based studies to full-scale FS pyrolysis applications will be discussed based on the outputs of this research (**Section 9.2**). Finally, the resource recovery options presented in this thesis will be synthesised into evidence-based indicators and actionable recommendations for various decision-making scenarios (**Section 9.3**).

9.1 Understanding the role of source control in sanitation systems

A common theme within this thesis has been establishing quantifiable relationships between source interventions (namely source separation and biomass addition) and biochar characteristics relevant to resource recovery (e.g. soil improving properties, solid fuel value, carbon sequestration). These evidence-based links between source control and resource recovery create pathways for the production of marketable excreta-based products that can compete with, or exceed the performance of, commercial alternatives associated with heavy environmental burdens (Andriessen et al., 2017). In turn, the operation of sanitation businesses that cover the entire FSM service chain encourages the correct use of container-based and source-separating toilets by providing frequent collection and maintenance services for toilet users (Silveti & Andersson, 2019). Therefore, a reinforcing loop is created whereby improved sanitation services create more opportunities for resource recovery applications, and resource recovery benefits encourage the improved coverage and efficient management of sanitation systems.

Currently, efforts to create circular resource recovery businesses from on-site sanitation systems are in place (see **Chapter 8**), however, these are challenging from an economic perspective and usually rely on external funding and subsidies in order to be sustained (World Bank, 2019). Source control can contribute to the establishment of profitable sanitation businesses that successfully – and consistently – recover resources from human excreta. In this research, this link has been especially evident for the case of N retention and circular nutrient management, through the source separation of human excreta (see **Section 9.1.1**), as well as for improved carbon management through synergistic co-pyrolysis of human faeces with biomass materials (see **Section 9.1.2**). The beneficial effect of source control has also been highlighted in the context of solid fuel production from FS carbonisation (refer to **Sections 8.3.3-8.3.4**), although challenges of at-scale operations still need to be addressed (see **Section 9.2.1**).

The quantifiable resource recovery benefits presented in this thesis can act as incentives to achieve safe sanitation for all, in line with the UN SDGs (Diener et al., 2014; Trimmer et al., 2019). To achieve SDG 6, a significant number of new on-site sanitation facilities needs to be installed and incorporated within sustainable FSM systems. This creates an opportunity to adopt a resource-oriented approach when designing new sanitation systems, rather than assuming that current designs – with little or no source control – should be replicated (Trimmer et al., 2020a). Therefore, resource recovery objectives should be seen as complementary, rather than competing, to the primary waste management objective of sanitation systems. Source control is an important strategic decision to enable this conceptual transition from waste to resource.

9.1.1 Source separation of human excreta for circular nutrient management via pyrolysis

This research used a controlled experimental design to isolate the effects of source separation within human excreta pyrolysis systems (see **Chapter 5**). One of the key outcomes of this investigation was the quantification of N retention benefits when diverting urine from thermal treatment (70% of total N emissions avoided – see calculations in **Section 5.3.3.1.1**). Apart from important nutrient management implications (see **Section 1.2.2**), the avoided N emissions include NO_x gases that contribute to acid rain and other photochemical reactions linked with climate change, as well as N₂O gas directly contributing to global warming and ozone depletion (Munawer, 2018; Filonchyk et al., 2024). Therefore, urine diversion has been emphasised as a required intervention to enable the circularity of pyrolysis (or broader, thermal treatment technologies) in the context of N retention.

To this end, a suitable nutrient recovery scenario entails the separate management of urine-derived nutrients (Maurer et al., 2006; Udert et al., 2015) coupled with pyrolysis of separated faeces to produce biochar soil amendments with high liming potential and cation exchange capacity (see **Table 5.2**). An alternative scenario was presented in **Chapter 6** of this thesis, where faecal-derived biochar was successfully enriched with urine-derived N (+2% N content as readily available NH₄⁺ ions) and P (+2.5% P content as crystalline PO₄-P structures, including struvite) to produce a combined fertiliser and soil amendment from source-separated excreta. This approach not only mitigates environmental risks of NP runoff from liquid urine-derived fertilisers but also introduces a novel circularity concept of human excreta self-treatment, by utilising a faecal-derived material to recover nutrients from urine.

The understanding of adsorption and precipitation mechanisms within biochar-urine contact systems provided in **Section 6.3.3** allows further optimisation of the agricultural value of enriched biochars, through the adjustment of their NPK ratio. For example, the chosen biochar dose had a significant influence on N uptake from urine ($p < 0.001$), while the Mg dose (here, added as MgO – refer to **Figure 6.3**) determined P recovery via precipitation. Furthermore, the main N uptake mechanism was identified

as stoichiometric $K^+ \leftrightarrow NH_4^+$ ion exchange, hence the trade-off between N-K recovery can be strategically balanced when source control is in place, enabling the production of tailor-made biochars. The identification of incorporated struvite(-K) in the urine-enriched biochar (a high-value slow-release NP fertiliser and its K-containing analogue) was also an important step towards the creation of added value at the back-end of human excreta management systems (Yetilmezsoy et al., 2017).

Practical advantages of source separation can also be expected for the nutrient availability of recovered products. During pyrolysis, some N is retained in the biochar in aromatic recalcitrant forms (e.g. nitrile, pyridine, amino acids, and pyrrole groups) that are thermally stable but not readily bioavailable, whereas N in urine is primarily present as urea which hydrolyses to readily plant-available NH_4 -N (NH_3 and exchangeable NH_4^+) (see **Figure 6.1**) (de Oliveira Paiva et al., 2024). Notably, the partial presence of N in stored urine as NH_3 requires careful handling to avoid volatilisation losses during storage or biochar recovery applications (Udert et al., 2006). Similarly, urine enrichment led to an increase of biochar P availability, due to the formation of PO_4 -containing precipitates that are expected to solubilise under neutral or acidic soil pH conditions (Erdal et al., 2024) (see **Section 6.3.2.2**). The simultaneous effects of biochar as a soil amendment can further increase the retention and plant uptake of urine-derived nutrients, due to its ability to regulate soil nutrients and minimise volatilisation and leaching, while also providing an excess of available cation exchange sites, including NH_4^+ , K^+ , Ca^{2+} and Mg^{2+} ions (57.6 cmol/kg cation exchange capacity for faecal biochars produced at 500 °C) (Hematimatin et al., 2024).

Overall, these findings provide proof-of-concept for the systemic management of source-separated human excreta to a) enable N retention within pyrolysis-based treatment solutions; b) combine nutrient recovery from urine with the soil improving properties of faecal biochars, and c) introduce a novel self-treatment method for human excreta. Environmental and financial benefits of this circular approach for sanitation-driven nutrient management can be vital to support and sustain resource businesses that effectively collect and treat human excreta (Mallory et al., 2020).

9.1.2 Synergistic co-pyrolysis with biomass for carbon management

In the context of climate change mitigation, the on-site sanitation sector is starting to be recognised as an important actor for global carbon management, estimated to contribute to up to 6% of global CH_4 emissions (McNicol et al., 2020). The opportunity of pyrolysis to store part of this carbon in stable forms, effectively removing it from the atmospheric cycle, is a significant advantage within the wider landscape of technological solutions for human excreta management (Cross & Sohi, 2013; McLaughlin et al., 2023). Nevertheless, climate change impact assessments for FSM are still limited and mostly based on the use of global databases that include several assumptions and simplifications (Lam et al., 2020).

The findings presented in **Chapter 7** can therefore be used to develop methodologies for detailed sustainability assessments, including carbon storage calculations. In particular, simple carbon flow mapping (see **Figure 7.11, Section 7.3.3**) and carbon stability tests (see **Sections 7.3.2** and **8.3.2**) can be used to create a library of data based on real measurements for human excreta/FS and biomass pyrolysis, hence allowing the development of accurate databases for on-site sanitation. When incorporated into wider methodologies for carbon assessments during containment, collection and transport of human excreta (CBSA, 2023), this approach can provide a systematic way to measure and analyse carbon offsetting opportunities for sanitation business models.

The non-linear interactions during faeces-biomass co-pyrolysis observed and quantified in this research further improve our understanding of carbon storage potential in sanitation systems. Co-pyrolysis of faeces with wood (see **Chapter 7**) or other available agricultural waste (e.g. bagasse – see **Section 8.2.2**) used as cover materials and/or feedstock additives, can increase the carbon fixation of the faecal fraction through synergistic interactions of the organic building blocks of the materials (e.g. promotion/inhibition effects) (Ma et al., 2022). This mechanism allows the optimisation of carbon storage within sanitation systems, while also informing on the minimum quantity of biomass materials needed to achieve these benefits (50:50 faeces:biomass ratio to store 40% of total C as shown in **Figure 7.9**), minimising trade-offs with reforestation or food security (Muscat et al., 2020). Furthermore, the outlined correlation between C content and easily measured parameters (e.g. ash) allows the quantification of these benefits even at low-resource settings (refer to **Section 8.3.5**). At the moment, several CBS providers are participating in voluntary carbon credit markets and could directly benefit from this simple carbon mapping approach (CBSA, 2023).

For detailed emissions calculations, the composition of emitted gases should also be accounted for, as different C forms will have different climate change implications. For human faeces, CO₂ and CH₄ have been identified as the main gaseous emissions at mid-scale pyrolysis temperatures (Yacob et al., 2018). While CO₂ emissions from biomass are considered as biogenic (Agunyo et al., 2019; Rowles et al., 2022), several studies suggest that at least part of the emissions from FS should be included in assessments as non-biogenic due to potential contamination and additional inputs in sanitation systems (Gallego-Schmid & Tarpani, 2019). Nitrous oxide emissions also need to be reported in climate change assessments and are expected to be reduced both via urine diversion (see **Section 9.1.1**) and biomass addition. For the calculation of the overall carbon footprint of a pyrolysis system, the heating source of the furnace needs to be considered. For example, if electricity is used, the energy mix (i.e. renewable versus non-renewable sources) needs to be known. For developing countries, solid fuels are often used as the heating source of traditional kilns (see **Section 8.2.2**), in which case the use of recovered products (e.g. waste-derived briquettes) should be prioritised over the use of wood-based fuels.

These calculations can be supplemented with data for other parts of the FSM chain, including indirect emissions or avoided impacts from the replacement of linear agricultural products with faecal-derived biochars. In this context, trade-offs between biochar agricultural value and carbon sequestration should be considered. For example, biochars derived from pure human excreta/FS (see **Sections 5.3.3 and 8.3.1**) were more nutrient-dense than biomass-derived biochars (Ding et al., 2016; Bleuler et al., 2020), while faeces:wood blends produced biochars with higher carbon sequestration potential compared to pure faecal feedstocks (see **Chapter 7 and Section 8.3.2**). The influence of environmental conditions (e.g. temperature, pH) and soil interactions also need to be considered for context-specific carbon assessments (Fang et al., 2014; Lorenz & Lal, 2014).

Overall, this research provides critical information towards the establishment of carbon-neutral (or carbon-negative) human excreta management systems. Container-based sanitation business models are particularly relevant to the context of these findings, as they provide a service-based approach with frequent collection and treatment services, involve the use of biomass-based cover materials and allow strategic interventions (and data collection) along the entire FSM chain. Nevertheless, several institutional, operational and financial challenges need to be overcome before rapid upscaling is feasible (Mallory et al., 2022). Quantifying the added value of climate change mitigation can provide meaningful incentives for operators and governments to maintain effective collection services for on-site sanitation systems.

9.2 Moving from laboratory-based research to full-scale applications

The information presented in previous chapters included laboratory-based research on the optimisation of resource recovery via pyrolysis of human excreta (**Chapters 5-7**), as well as the investigation of an operating carbonisation FSTP in Kampala, Uganda (**Chapter 8**). Several differences were observed between laboratory-based and full-scale pyrolysis applications, including related to feedstock properties, operational conditions during treatment and end-product characteristics. This research identified opportunities for upscaling and enhanced resource recovery, while also noting limitations and providing insights on how these can be addressed.

9.2.1 The case of faecal-derived solid fuels in Kampala, Uganda

For the case of Kampala, there was high demand for affordable and sustainable solid fuels, making FS-derived briquettes a valuable product for recovery. Existing users have provided positive feedback due to the affordability and slow burn time of FS-derived fuels, however, FS briquettes are in direct competition with charcoal and other biomass-derived briquettes that have favourable fuel properties (typically 17-22 MJ/kg compared to 13-16 MJ/kg for FS-derived briquettes presented in **Chapter 8**). The high ash content of currently available FS sources (including sludge from unlined or partially lined latrines) was therefore identified as a significant barrier that needs to be addressed via source control

(see the ash-calorific value correlation in **Figure 8.9**). Efforts to minimise the ash present in FS will be critical to optimise resource recovery, including lining latrines to limit the entry of soil or other inorganic contaminants at the source, and avoiding the use of sand in drying beds at FSTP-level. In terms of interventions at the toilet-level, both urine diversion and biomass addition can significantly decrease FS ash content, hence increasing the calorific value of produced briquettes (from 6.4 MJ/kg for traditional FS to >17 MJ/kg for mixed excreta and biomass, and >20 MJ/kg for separated faeces and biomass – see data in **Figure 8.8**).

From an operational perspective, several practical recommendations for the optimisation of FSTPs in developing countries can be drawn from the knowledge presented in this thesis. A main challenge identified for traditional non-electric kilns was the high residual volatile matter of produced chars ($\geq 20\%$ as shown in **Table 8.4**), indicating that the temperature reached inside the kiln may not be sufficient to complete carbonisation reactions (i.e. not exceeding 450 °C) (Liu et al., 2014; Krounbi et al., 2018). While briquettes with high volatile matter can ignite easily, their burn time is lower than fully carbonised fuels and they generate more smoke during their use, negatively affecting air quality, particularly for indoor activities such as cooking (Otieno et al., 2022). Therefore, the completion of carbonisation reactions is essential to recover “smokeless” fuels that are safe for indoor use. Based on findings presented in **Chapter 5**, pyrolysis between 450-500 °C is recommended to ensure complete carbonisation of excreta-derived materials, without compromising the energy content (i.e. calorific value) at higher treatment temperatures (>500 °C) (Liu et al., 2014; Ren et al., 2018).

In terms of the kiln design, while traditional kilns are a realistic solution for low-resource settings, targeted modifications are recommended to allow for accurate temperature monitoring and control (e.g. installation of thermocouples inside the kiln) (Lohri et al., 2016). Further measures could include the incorporation of insulation materials to increase the highest heating temperature reached inside the kiln, as well as improved heat convection mechanisms (e.g. gas recirculation). Maintaining an oxygen-free or oxygen-limited environment inside the kiln is also essential. Efforts to effectively “air-lock” the kiln from the onset of the process are recommended to avoid partial ashing of the feedstock.

Wider environmental issues that need to be addressed include the installation of emission control systems and the optimisation of process energy efficiency (Lohri et al., 2016; Rowles et al., 2022; Zhang et al., 2022). Ideally, the thermal energy of produced gases should be used to (partially) replace external heating requirements, as well as for pre-treatment steps (e.g. thermal drying) (Gold et al., 2018; Myers et al., 2019). Improving the energy efficiency and environmental performance of FS pyrolysis applications will be essential for upscaling and to ensure their long-term sustainability (both environmental and financial) and social acceptance. Ultimately, changing the perception of sanitation systems to a resource-focused paradigm is easier when the feasibility of recovering safe, affordable and sustainable products is consistently proven at-scale (Russel et al., 2015; Mamera et al., 2020).

9.2.2 Addressing the variability of faecal sludge

The variability of FS characteristics is highlighted in the previous sections and throughout this thesis as it constitutes a significant limitation for sanitation research. As the number of studies on FS pyrolysis grows, further investigation of the statistical significance of char properties and their geographical and temporal variance becomes imperative (Nicholas et al., 2022b). Fundamental research alone cannot guarantee applicability of findings to specific cases or provide knowledge on important implementation aspects. Similarly, case-studies do not guarantee the representative nature of samples and might not be applicable to other contexts (e.g. due to contamination and outliers). The combination of the two, either in one research piece – as in this thesis – or via critical reviews of previous studies, allows the development of knowledge that is both broad and deep, being relevant to both future theoretical research and case-specific practical applications led by researchers and/or practitioners.

To this end, critical reviews and comparisons of results presented in different studies need to take the variability of FS into account, to avoid inaccurate conclusions based on unaccounted differences in feedstock composition (Doglas et al., 2021). All studies using FS as the feedstock for resource recovery need to comprehensively present data on its origin, the sampling protocol followed and the characterisation of (representative) samples (including organic and inorganic composition and key physiochemical characteristics) (Velkushanova et al., 2021). Findings presented in **Chapter 8** highlight the importance of the statistical analysis of FS (and char) characteristics, to allow the accurate interpretation of data. Critical reviews should either exclude studies with inadequate information on feedstock origin and characterisation, or clearly highlight the lack of data provided, to avoid the reproduction of unsafe conclusions within relevant literature. The terminology used to describe feedstock materials in sanitation settings is also important. The commonly used term “faecal sludge” is vague as it refers to a wide variety of containment facilities and treatment processes, that may yield products of significantly different composition (Strande et al., 2023). Therefore, additional information on feedstock origin needs to be provided.

The choice made in this thesis to focus on human excreta as a fundamental material relevant to FS research creates some applicability limitations that need to be accurately addressed and justified, while also creating opportunities to answer research questions that cannot be fully addressed for a variable material such as FS. Similar to FS studies, experimental research using human excreta needs to include a comprehensive characterisation of the feedstock material and report on its origin (e.g. number of volunteers, diet, sampling protocol and duration, etc.), to enable critical comparisons with literature data. The use of human excreta for sanitation research is useful for research objectives that need to be investigated against consistent feedstock properties, such as those relevant to source interventions and resource recovery. Findings remain applicable to wider FS research and applications, as long as they are transparently reported and statistically interpreted. The translation of results for human excreta to a

real case study for FS was the focus of **Chapter 8**, allowing the direct comparison of the two types of samples. Results provided insights into the suitability of different FS sources for various resource recovery applications and were particularly relevant to the comparison of traditional on-site sanitation facilities versus CBS toilets, as well as mixed-stream toilets versus urine-diverting systems.

Although the benefits of source interventions for resource recovery via pyrolysis are clearly identified – and quantified – in this research, studies using donor-based samples cannot investigate the user acceptance of these interventions, which may vary between different cultural and social contexts (Uddin et al., 2012; Mkhize et al., 2017). Therefore, studies using FS samples from commonly used on-site sanitation technologies (e.g. VIPs, UDDTs) are also essential to understand user behaviour and additional factors influencing FS composition (Krueger et al., 2021). In the context of FS pyrolysis, the combination of this knowledge with the fundamental understanding of human excreta behaviour provided in this thesis is critical to optimise process performance and determine its suitability for different feedstock properties and resource recovery objectives – see **Section 9.3** below.

9.3 Suitable options and decision-making scenarios for resource recovery via pyrolysis of human excreta

Overall, this research investigated resource recovery from pyrolysis of human excreta and FS for several recovery options, including agricultural uses, solid fuel production, carbon sequestration and adsorption applications. Findings demonstrate that the suitability of different recovery scenarios is dependent on the feedstock composition (as influenced by potential source interventions – see **Section 9.1**) and the operational conditions during pyrolysis. Here, this knowledge has been integrated to form an accessible decision-making framework for sanitation researchers and practitioners (**Table 9.1**). Laboratory findings have been translated into key analytical indicators and actionable recommendations for optimisation of the four selected resource recovery objectives. Further information for each recovery option can be found in the relevant chapters of this thesis denoted in **Table 9.1**, while analytical methods for the suggested indicators are included in **Chapter 4**, as well as the Methods sections of individual chapters.

Table 9.1: Technical decision-making parameters for various options of resource recovery via pyrolysis of human excreta (including analytical indicators of biochar quality, barriers to monitor, source control recommendations and recommended operational parameters).

Recovery options	Key analytical indicators of biochar quality	Barriers to monitor	Source control recommendations	Operational parameters recommendations
Agricultural uses (see Chapters 5, 6, 8)	- Nutrient content (and availability) - Liming potential - pH, EC - CEC - Heavy metals	- Biochar salinity can inhibit plant growth - Heavy metal content requires monitoring (especially Zn, Cu) - Incomplete carbonisation (<500 °C) may lead to leaching of toxic compounds	- Source separation of excreta to enable N retention (+70% of TN) and minimise biochar salinity - Urine on-site storage should take place in closed containers to limit N losses (≤5-10% of TN feasible)	500-550 °C under slow pyrolysis conditions for ≥30 mins - Post-pyrolysis, faecal biochars can be enriched with urine-derived plant-available nutrients
Energy recovery and/or solid fuel production (see Chapters 5, 7, 8)	- Calorific value* - Ash content - Moisture content - Volatile matter - CHNS content	- Moisture and ash reduce energy value - High volatility leads to more emissions during burning of solid fuels - NOx/SOx gases may be emitted during burning of solid fuels - Oil/gas recovery requires “know-how” and safety measures	- Biomass addition (≥50% dry w/w) to reduce ash and NS content, maximise calorific value and enhance biochar stability (Figure 8.8 and Table 8.5) - Lined or CBS containment to reduce inorganic inputs (ash) - Source separation can further reduce ash and moisture content, maximising the calorific value (Table 5.2)	450-500 °C under slow pyrolysis conditions for ≥30 mins, for solid fuels >500 °C to maximise oil/gas production - Inert gas flow and retention time to be chosen based on oil/gas recovery objectives and capabilities (Figure 7.11)
Carbon sequestration (see Chapters 5, 7, 8)	(Fixed) C content* - C stability (H₂O₂ oxidation test recommended) - H:C, O:C ratios - FTIR to identify aromatic C structures	- High ash content dilutes the carbon available for storage - Incomplete carbonisation (<500 °C) leads to unstable carbon forms	- Biomass addition (50% dry w/w) to synergistically enhance C retention and stability (40% of C saved) - Lined or CBS containment to reduce inorganic inputs (ash) - Source separation can further increase C content (Table 5.2)	500-550 °C under slow pyrolysis conditions, with a low inert gas flow rate (e.g. N₂) for 30 mins
Adsorption uses (see Chapters 5, 6)	- BET surface area - CEC - SEM analysis, porosity - FTIR to identify surface functional groups	Macroporous nature of faecal biochars with low surface area (<20 m²/g)	- Compounds present in urine can act as activation agents for faecal biochars (mixed excreta sources or urine activation) - Biomass addition can increase surface area	≥650 °C to maximise surface area for physical adsorption 450-550 °C to maximise CEC for ion exchange uptake (see Section 6.3.2)

* correlation with ash content (see **Figure 7.9** and **Figure 8.9**).

From a strategic standpoint, the knowledge presented in **Table 9.1** can be utilised in three scenarios, depending on the starting point of the decision-making process:

- A. To determine the most suitable **resource recovery strategy** for an existing FS feedstock, via the assessment of (key) analytical indicators.
- B. To identify the best **FS feedstock** for a specific resource recovery objective, via the assessment of suitable source control options.
- C. To identify the optimal **pyrolysis operational parameters** for a specific resource recovery objective and FS feedstock.

Scenario A is relevant to the design and strategic planning of new or existing FSTPs treating current quantities of FS that may be variable in composition. Some resource recovery objectives can be complementary (e.g. agricultural uses and carbon sequestration), while others are competing (e.g. carbon sequestration versus solid fuel production). Apart from technical indicators, an understanding of the context-specific market landscape is also critical. For example, in Kampala, the need for affordable solid fuels was driving demand for FS-derived briquettes despite the high ash content of the currently available feedstocks.

Therefore, **Scenario B** is useful when the recovery objective precedes the feedstock selection. This scenario is particularly relevant to new sanitation infrastructure (e.g. CBS toilets) designed with a specific resource recovery strategy in mind (e.g. carbon sequestration) or to enhance the operational efficiency of an existing FS pyrolysis plant, through source control (e.g. adjusting the biomass addition ratio).

Scenario C is relevant to operators aiming to maximise the recovery efficiency of new or existing pyrolysis FSTPs, being complementary to scenarios A and B.

While this research has focused on technical and resource management aspects of human excreta pyrolysis, the upscaling of the technology will also require the assessment of economic, social, and environmental indicators (Trimmer et al., 2020a; Rowles et al., 2022). The techno-economic feasibility and environmental sustainability of pyrolysis applications is expected to vary across different scales and technological sophistication levels, ranging from engineered furnace designs with energy recovery and emission control capabilities (e.g. laboratory-scale furnace design described in **Section 4.2**) to low-cost carbonisation kilns with limited operational control and potentially uncontrolled emissions (e.g. traditional kiln design in **Section 8.2.2**). A life-cycle approach will be essential to evaluate the comparative performance of pyrolysis solutions against other available treatment technologies, taking into account case-specific parameters (e.g. material flows, end-use and demand of recovered products) and locally significant criteria (e.g. spatial characteristics, environmental receptors etc.) (see future work recommendations in **Section 10.2**).

10 Future work

10.1 Applied research on excreta-derived biochars

10.1.1 Agricultural applications

Further research is recommended to upscale the production of faecal-derived biochars and urine-enriched fertiliser products, including suitable urine-biochar contact systems, and to investigate agricultural benefits via plant growth trials (Jellali et al., 2022). As biochars are generally slow-release fertilisers (see **Section 5.3.3.1**), it is recommended that studies conducting plant growth trials investigate effects over multiple growing cycles and for several plant types of varying fertilising needs (Woldetsadik et al., 2017). The influence of biochar application can vary significantly depending on environmental conditions, initial soil properties and plant requirements, and should be further studied as FS biochar fertilisers become more widely applied (Kutu et al., 2010; Sohi et al., 2010). Future research should aim to further optimise and simplify treatment and reuse technologies (e.g. pH control for improved N recovery from urine or tailor-made biochar fertilisers with adjusted NPK ratios – see **Section 6.3.3**).

Currently, very limited knowledge is available on potential long-term benefits (e.g. carbon sequestration) or unintended consequences (e.g. salt stress) of biochar fertilisers and soil amendments. Studies monitoring the effect of biochar fertilisers over extended periods will be useful to allow the systematic assessment of their performance over time and across locations. This is particularly relevant to carbon sequestration applications assessing the long-term storage of stable C forms present in biochars (see **Chapter 7**). Safety limitations and social acceptance issues also need to be addressed, particularly over the potential spread of pharmaceuticals to the environment and the lack of regulatory context of urine-derived fertilisers (Bischel et al., 2015; Moya et al., 2019a). Findings of recent research direct future work towards modified biochars that selectively adsorb nutrients or pharmaceuticals to separate the two streams before environmental applications (Masrura et al., 2022). Knowledge on potential health effects of pharmaceuticals and other emerging contaminants present in soils is currently limited, creating an interesting subject for interdisciplinary research (Clarke & Smith, 2011; X. Li et al., 2023).

In terms of heavy metal safety, the results of this research are generally positive but highlight some trace elements of concern (Zn, Cu), according to current international and European standards (EBC, 2012; IBI, 2015; WBC, 2023). Nevertheless, most countries do not yet have regulations specific to biochars, while thresholds for other organic fertilisers may fail to account for the high nutrient content, and hence potential lower application rates, of excreta-derived biochars. Further work can focus on the development of fit-for-purpose thresholds (including nutrient-to-heavy-metal ratios), as well as the investigation of potential health effects to validate the efficacy and required rigidity of standards (Bünemann et al., 2024).

10.1.2 Other resource recovery options

In this thesis, the most widely applied resource recovery options were investigated, namely nutrient and energy recovery, and carbon storage applications. Additional resource recovery routes were identified during the literature review (see **Section 3.3.4**) and can be further investigated, although the expected demand for recovered products needs to be established first, on a case-specific context. The activation of excreta-derived biochars was identified as an interesting field for future research. This thesis highlighted the potential for urine activation of faecal-derived biochars (see **Section 5.3.3.3**), which would further support circularity efforts within FS treatment processes, by avoiding the use of other materials (e.g. KOH, H₃PO₄) for activation. Another recovery option could include the production of building materials, although the feasibility of using excreta-derived biochars in the construction industry has not been assessed yet. For these emerging recovery routes, market analyses and techno-economic feasibility assessments are interesting subjects for further research, including on the comparative performance of different available options.

In terms of energy recovery, this thesis investigated the production of excreta-derived solid fuels, with promising initial responses from users in Uganda. Nevertheless, in the context of available literature, much less knowledge is available compared to nutrient recovery applications, highlighting a gap that will require more research on optimisation and practical applications. For example, future studies can focus on further optimisation of briquette composition, investigate burning properties using real kilns, or validate correlations presented in this thesis that can be used by practitioners (see **Section 8.3.5**). Moreover, in **Chapter 8**, it was reported that outputs of CBS toilets would be more suitable for energy recovery applications compared to traditional FS sources, bringing attention to a promising area for applied research.

Less is known about the energy recovery potential from the oil and gas fractions of FS pyrolysis, particularly as traditional carbonisation applications commonly found in developing countries focus on solid recovery and often do not recover oils and gases. The mapping of carbon flows presented in **Section 7.3.3** included calculations for the oil and gas fractions, forming a novel research area for excreta-based feedstocks that can be used as a baseline for future studies on FS pyrolysis.

In terms of carbon sequestration potential, this research has focused on the carbon stability and mass balance when FS is co-pyrolysed with wood (or bagasse) (see **Chapters 7-8**), but the presented methods can be applied for other biomass materials or alternative treatment systems where biochar is used in conjunction with other technologies (e.g. biochar used in composting or as a urine treatment medium) (Castro-Herrera et al., 2023; Koulouri et al., 2024a).

10.2 Investigating the sustainability of on-site sanitation systems

The need to conduct sustainability assessments that cover the entire life cycle of sanitation and FSM systems has been highlighted in many instances throughout this thesis. Life cycle assessment (LCA) and input-output analysis studies will be useful during the upscaling of resource recovery processes, to ensure that only the most sustainable technological choices are carried forward (Lam et al., 2020). This is particularly important for novel treatment technologies, such as pyrolysis, that are still at development stage and have not been widely applied for FS treatment at full-scale. **Chapter 7** findings, on the carbon flows during co-pyrolysis of faecal matter with wood biomass, can be used alongside data collected for upstream and downstream processes, including energy consumption and (avoided) emissions, to form comprehensive sustainability assessments with expanded system boundaries (also refer to **Section 9.1.2**). Some practical considerations for future LCA studies on FS pyrolysis are described below:

- Studies in the context of developing countries will need to be designed and delivered by (or with) local researchers and practitioners that understand the local context and data collection requirements;
- The methodologies and indicators presented in this thesis (often with a focus on quick, inexpensive measurements) can be used to allow data collection for real case studies, rather than conducting sustainability assessments based on generic databases that are not specific to the investigated regions or countries;
- Future research will need to consider the multifunctionality of resource-oriented FSM systems (including waste management, nutrient recovery, energy recovery, etc.), in order to attribute impacts to different functions (ISO, 2016; Lam et al., 2020);
- The LCA system boundaries should be extended to reflect all activities of resource-oriented FSM systems (e.g. including end-product uses); and
- Life cycle impacts should be assessed within the wider context of current practices (e.g. open defecation, long-term storage in pit latrines) and alternative treatment technologies (e.g. compared to widely used technologies, such as anaerobic digestion) to allow for valuable conclusions on relative impacts.

10.3 The future of faecal sludge pyrolysis research

In the context of literature, the majority of studies on FS pyrolysis have been conducted using laboratory-scale or pilot-scale pyrolysis reactors, with very little knowledge currently available on the operation of full-scale pyrolysis/carbonisation FSTPs. More research will be essential to allow the statistical interpretation of findings and eventually allow the systematic review of data on full-scale FS pyrolysis. This thesis provides fundamental knowledge on the effects of source interventions on resource recovery, that can be applied as a baseline for future studies on FS. For example, chars

produced from VIP sludge can be compared against those produced from UDDT sludge, using the comparison of SSF/MUF chars (presented in **Chapter 5**) as a baseline to assess the efficacy of real urine diversion toilets (i.e. are urine and faeces really separated by users?). Further research on the user behaviour and social acceptance of source interventions will also be valuable for upscaling of new technologies and providing recommendations to practitioners at a local level. It is also very likely that future at-scale studies will highlight new areas for fundamental and pilot-scale research (e.g. **Chapter 8** showed the need to improve the design – and level of operational control – of traditional carbonisation kilns that are still in use in developing countries).

In terms of feedstock types, this thesis showed that CBS technologies are particularly suitable for resource recovery via pyrolysis and should be further researched. Human excreta samples are a good proxy of CBS outputs and therefore, the sampling protocol presented in **Section 4.1** can be used by future studies. Due to COVID-19 restrictions during the sampling conducted for this research, the number of participating volunteers was limited (12 volunteers for each sample group). It is recommended that future studies include a larger sample size and, if possible, categorise sample groups based on diet habits. While several studies agree that diet can impact FS composition, and therefore resource recovery potential (Krueger et al., 2020; Somorin, 2020; Aluko et al., 2020), no publications exploring this relationship have been published to this date. In terms of cover material addition, this thesis studied the effect of willow wood addition, but future studies can assess other widely available materials (e.g. coconut husk, other wood sources, etc.) or investigate the comparative performance of different materials for carbon sequestration and other recovery routes.

Overall, future research on the treatment of human excreta and FS can be broadly categorised as:

- A. aiming to investigate and improve the management of existing FS quantities and qualities (e.g. from pit latrines, septic tanks, etc.); and
- B. planning for the management of future FS quantities and qualities, for emerging on-site sanitation technologies (such as CBS) and new toilets that will need to be designed, built and managed to serve the people that currently do not have access to safely managed sanitation.

Both research areas will be crucial to ensure safely managed sanitation for all, and therefore the two subjects should be seen as complementary, rather than competing. For both cases, further research should focus on the urgent requirement of recovering resources at full-scale and assessing the market value and environmental benefits of recovered products. Ultimately, achieving safe and sustainable sanitation for all is, and should be, the core of FSM research.

11 Conclusions and contributions to knowledge

The following conclusions were drawn in answer to the research objectives stated in **Chapter 2**:

Objective 1: Establish the relationship between source separation of human excreta (faeces and urine) and resource recovery via pyrolysis.

1a. Compare the compositional characteristics of the two excreta components and identify their influence upon the thermal decomposition behaviour during pyrolysis, when maintaining all other parameters constant.

Faeces and urine have very distinct characteristics, which influence their behaviour during thermochemical treatment. Impacts of source separation on the thermal decomposition behaviour during pyrolysis were attributed to differences in their moisture and fat content, and to the varying nature of nitrogenous compounds present in each stream. Source-separated faeces had significantly lower ash content compared to mixed excreta (12.5% compared to 19%), as well as higher carbon content (50% compared to 41%) and calorific value (22 MJ/kg compared to 18 MJ/kg), due to the higher presence of inorganic salts in urine.

1b. Assess the effect of two feedstock scenarios (with and without source separation) and three pyrolysis temperatures (450, 550, 650 °C) on biochar quality, via the comprehensive characterisation of biochar characteristics.

Source separation had a statistically significant effect on all biochar parameters tested (organic and inorganic composition, surface morphology and crystalline structure). Pyrolysis temperature also influenced biochar characteristics, with most statistically significant changes observed at the 450-550 °C range. Nutrient and carbon behaviour during pyrolysis suggested that temperatures around 500 °C are preferable in terms of biochar stability, nutrient availability and solid fuel value objectives, for both separated and non-separated human excreta feedstocks.

1c. Investigate the influence of source separation on potential end-uses of biochars, including agricultural uses and solid fuel production.

All human excreta biochars had good P recovery potential, while source separation significantly benefited N recovery (70% of total N losses during thermal treatment avoided) and allowed the production of faecal biochars with higher liming potential. Mixed excreta produced PK-rich biochars, but their high salinity may place limitations on agricultural applications, especially for salt-sensitive plants.

While most studies to date have focused on the effect of source separation on nutrient recovery applications, this research also quantified benefits for solid fuel production, a valuable recovery path for regions that rely heavily on solid fuels. Source-separated faeces produced more energy-dense fuels (50% increase in char calorific value) with lower ash content and better burning properties (e.g. lower NO_x and SO_x emissions). At the same time, source separation enables the combination of nutrient and energy recovery objectives, from urine and faeces respectively.

Objective 2: Investigate a circular method to recover nutrients from stored human urine using faecal-derived biochars (via adsorption and precipitation).

2a. Investigate the evolution of the composition and nutrient content of separated human urine during on-site storage (for up to 6 months).

Storing human urine in sealed containers prevented N losses. Rapid urea hydrolysis reactions occurred over the first two weeks of storage (RTP, no mixing) and the urine solutions were fully stabilised after four months. The pH stabilised at 9.2, indicating a dynamic equilibrium is reached between NH₃ and NH₄⁺ phases. Mg availability was the limiting factor for the spontaneous precipitation of phosphates in urine.

2b. Study the mechanisms of NH₄-N adsorption from urine onto faecal-derived biochars and model the adsorption isotherms and kinetics.

NH₄-N was successfully adsorbed onto faecal-derived biochars and the adsorption capacity of the biochar was 19.8 mg NH₄-N/g. Experimental data exhibit a good fit to both the Freundlich ($R^2 = 0.989$) and Langmuir ($R^2 = 0.974$) isotherm models and the rate of the reaction was well described by pseudo 2nd-order kinetics ($R^2 = 0.988$), corresponding to a rapid initial rate of adsorption followed by a continuous decrease in rate until reaching equilibrium (after 24 h). The main NH₄-N recovery mechanism was attributed to ion exchange ($K^+ \leftrightarrow NH_4^+$), as confirmed by XRD and ICP-OES analyses.

2c. Assess the suitability of faecal-derived biochar as a carrier for simultaneous N and P recovery, via batch adsorption and precipitation experiments.

Faecal-derived biochar had limited adsorption capacity for P, with precipitation being the main mechanism for P recovery. When adding an external Mg source (MgO), >98% of total P was recovered from the stored urine solution, through precipitation of struvite/struvite-K and Mg/K-substituted hydroxyapatite. The biochar dose had a statistically significant influence on N recovery via adsorption and the MgO dose on P recovery via precipitation ($p < 0.001$).

Faecal-derived biochar was a successful carrier to recover the P-containing precipitates and facilitate liquid-solid separation after treatment. The combination of MgO addition (Mg:P=1.5 molar ratio) with the lower biochar dose tested (25 g/L) produced the most NP-rich fertiliser product. Overall, these

findings confirmed that faecal-derived biochars can be used to recover nutrients from separated urine, supporting a novel circular approach to human excreta management.

Objective 3: Investigate the carbon sequestration potential when co-pyrollysing source-separated faeces with wood biomass.

3a. Identify potential synergistic effects during co-pyrolysis of faeces and wood biomass, in terms of carbon retention and biochar stability.

Co-pyrolysis of human faeces (HF) and wood biomass (WB) improved carbon fixation in produced biochars, enhancing carbon sequestration potential compared to pyrolysis of pure faecal feedstocks. Synergistic effects took place during HF:WB co-pyrolysis, both in terms of carbon stability and carbon storage benefits. A 50% WB content (dry % w/w) was sufficient to alter carbon dynamics and enhance carbon stability. From a practical perspective, this intervention corresponds to each user adding approximately 30 g dry wood to a toilet as a cover material daily.

3b. Investigate the effect of various faeces-to-wood mixing ratios (100:0, 75:25, 50:50, 25:75, 0:100) and pyrolysis temperatures (450, 550, 650 °C) on biochar carbon properties.

Overall, biochar characteristics were more dependent on feedstock type and blending ratios rather than pyrolysis temperature, provided the temperature reached was sufficient to ensure completion of the main pyrolysis reactions (≥ 500 °C according to thermal analysis results).

The measured H/C, O/C ratios, R_{50} index and H_2O_2 oxidation values at 550 °C and 650 °C confirmed the stability of HF:WB biochars containing $\geq 50\%$ WB. Biochars produced from 50:50 HF:WB blends at 550 °C had the highest carbon retention (41.1%). Pyrolysing at a higher temperature (650 °C) did not show any benefits in terms of carbon retention, while biochars produced at 450 °C showed potential to release labile organic carbon.

3c. Determine the distribution of carbon flows among pyrolysis products (biochar, bio-oil, non-condensable gases).

The C flows to the biochar fraction were mainly dependent on feedstock composition, while the bio-oil and non-condensable gas fractions were greatly influenced by pyrolysis operational parameters. Wood biomass addition coupled with short retention times and low inert gas flow rates reduced C gas emissions during pyrolysis, while maximising C retention in the biochar and bio-oil fractions. Absolute values of C flows are system-specific, but the observed statistically significant trends related to specific operational parameters and wood addition ratios can be used for technical and high-level decision making related to carbon management in sanitation systems.

Objective 4: Apply findings to evaluate an operating faecal sludge (FS) treatment plant where FS is co-carbonised with biomass waste to produce char fertilisers and solid fuel briquettes.

4a. Determine the key properties of FS-derived fertilisers and solid fuels, and how these vary statistically.

The agricultural value of FS chars produced in Uganda was promising, particularly for P recovery. Heavy metal levels were below local standards (where available) but exceeded some international guidelines for char fertilisers. Carbon sequestration potential was limited, due to the high ash content of FS, but can be improved with biomass addition. Solid fuel briquettes prepared from co-carbonised FS and biomass waste were successfully incorporated into the local market, mainly due to their slow burning properties and affordability, and despite their lower calorific value compared to commercial standards (HHV<17 MJ/kg). Comprehensive sampling protocols are essential for the assessment of FSTP operations due to the observation of outliers for all parameters of interest.

4b. Investigate the influence of feedstock composition and pyrolysis operational conditions on end-product quality.

Both feedstock properties and operational conditions during carbonisation determined the final quality of recovered products and their suitability for different resource recovery options. In general, FS chars with high ash content (~70%) were deemed suitable for agricultural applications but can be challenging for solid fuel production due to the strong correlation between ash content and calorific value ($R^2 = 0.97$). In terms of operational parameters, technical improvements of traditional carbonisation kilns are recommended to increase (and accurately monitor) the highest heating temperature reached, and to ensure an oxygen-limited environment is maintained throughout the carbonisation process.

4c. Assess how the investigated source interventions can improve the resource recovery potential of at-scale FS carbonisation applications.

The source interventions studied in **Chapters 5-7** can have significant benefits for at-scale resource recovery from FS carbonisation. The P recovery potential of FS chars was promising (4% P content), but feedstocks from urine-diverting toilets would allow further N recovery from urine and maximise the total nutrient recovery potential, based on findings presented in **Chapters 5-6**. For carbon stability, the synergistic benefits of excreta-biomass co-pyrolysis established in **Chapter 7** were also confirmed for FS chars, although their high ash content inevitably reduced the total carbon storage potential compared to pure excreta sludges (e.g. CBS outputs). In terms of solid fuel production, the high ash content of FS was identified as the limiting factor for quality improvement and findings from **Chapter 5** were used to estimate the theoretical maximum energy value of produced briquettes (HHV>20 MJ/kg possible for outputs of source-separating toilets).

Summary of the contributions to knowledge

Several knowledge contributions arise from this research:

- The sampling protocol for volunteer-based human excreta collection that is presented in **Chapter 4** constitutes a novel research methodology that can be used by future experimental studies in the context of sanitation research.
- **Chapter 5** establishes a quantifiable relationship between human excreta source separation and biochar quality, for the first time.
- **Chapter 6** introduces a method to enrich faecal-derived biochars with NH_4^+ and P recovered from stored, fully hydrolysed human urine via adsorption and precipitation. For the first time, urine-enriched faecal-derived biochars are comprehensively characterised for their agricultural value and the mechanisms of nutrient uptake are investigated in depth.
- **Chapter 7** introduces a protocol to determine the carbon stability and carbon sequestration potential when co-pyrolysing human faeces with wood biomass. The distribution of carbon flows among pyrolysis products (biochar, bio-oil, non-condensable gases) is quantified for the first time in the context of human excreta pyrolysis.
- In **Chapter 8**, findings presented in previous chapters are applied to the case study of an operating FS treatment plant in Uganda, to enable the identification – and quantification – of optimisation pathways for real resource recovery applications. Multiple samples were collected over the course of two years to allow the statistical interpretation of FS char properties.

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Appendices

Appendix A. List of conference presentations

The author's presentations at scientific conferences during this PhD are listed below:

- **Koulouri, M.E.**, Templeton, M.R., Fowler, G.D., 2024. [3rd International Conference on Sustainable Chemical and Environmental Engineering](#), Crete, Greece (September 2024). Oral presentation, “Nutrient recovery from urine diverting dry toilets via pyrolysis”.
- **Koulouri M.E.**, Templeton M.R., Fowler G.D., Batarda C., Owomuhangi L., Lugali Y., Tuyishimire O., Nteziyaremye F., 2024. [11th International Conference on Sustainable Solid Waste Management](#), Rhodes, Greece (June 2024). Oral presentation, “Co-carbonisation of faecal sludge and biomass waste for resource recovery: Lessons learned from Uganda and Rwanda”.
- **Koulouri, M.E.**, Qiu M., Templeton, M.R., Fowler, G.D., 2023. [1st IWA Non-Sewered Sanitation Conference](#), Johannesburg, South Africa (October 2023). Oral presentation, “Resource recovery from faecal sludge pyrolysis: Insights on source interventions and biochar quality”.
- **Koulouri, M.E.**, Templeton, M.R., Fowler, G.D., 2023. [Water Innovation and Circularity Conference](#), Athens, Greece (June 2023). Oral presentation, “Long-term storage of source-separated urine: insights on nutrient recovery potential”.
- **Koulouri, M.E.**, Qiu M., Templeton, M.R., Fowler, G.D., 2023. [21st African Water Association \(AfWA\) International Congress & Exhibition and the 7th International Faecal Sludge Management \(FSM\) Conference](#), Abidjan, Côte D'Ivoire (February 2023). Oral presentation, “Co-pyrolysis of human faeces and wood biomass: Carbon flows and carbon sequestration potential”.
- **Koulouri, M.E.**, Templeton, M.R., Fowler, G.D., 2022. [9th International Conference on Sustainable Solid Waste Management](#), Corfu, Greece (June 2022). [Poster presentation](#), “Source-separation of human excreta as a driver for optimised resource recovery via pyrolysis”. **M.E. Koulouri awarded 2nd best poster prize.**

Appendix B. Additional data tables

Table A1: References and analysed parameters in studies investigating properties of **faecal sludge** samples (shown in **Figure 3.4** and **Figure 3.6**).

Reference	Analysed parameters								
	N	P	K	Ca	Mg	MC	HHV	Ash	Carbon
(Häfner et al., 2023)	✓	✓	✓	✓	✓				
(Beik et al., 2023)	✓					✓	✓	✓	✓
(Manga et al., 2022)	✓	✓	✓	✓					✓
(Kalulu et al., 2021)		✓				✓			
(Somorin et al., 2021)						✓			
(Krueger et al., 2020)	✓	✓	✓	✓	✓		✓	✓	✓
(Bleuler et al., 2020)	✓	✓	✓	✓				✓	✓
(Getahun et al., 2020)		✓				✓			
(Aluko et al., 2020)	✓	✓	✓	✓	✓				✓
(Septien et al., 2020)	✓	✓	✓	✓	✓	✓	✓		✓
(Singh et al., 2020)	✓		✓	✓	✓				
(Barani et al., 2020)	✓						✓	✓	✓
(Krounbi et al., 2019)		✓	✓	✓	✓	✓			
(Hafford et al., 2018)	✓								✓
(Gold et al., 2018a)	✓	✓					✓	✓	✓
(Krounbi et al., 2018)	✓	✓						✓	✓
(Fakkaew et al., 2018)	✓						✓	✓	✓
(Bai et al., 2018)	✓								
(Recalde et al., 2018)	✓						✓	✓	✓
(Amoah et al., 2017)	✓	✓	✓						
(Nartey et al., 2017)	✓	✓	✓						
(Gold et al., 2017)	✓	✓					✓	✓	✓
(Koetlisi & Muchaonyerwa, 2017)	✓								✓
(Afolabi et al., 2017)	✓					✓			✓
(Koottatep et al., 2016)							✓	✓	
(Hübner et al., 2016)						✓	✓	✓	✓
(Afolabi et al., 2015)	✓					✓	✓		✓
(Liu et al., 2014)	✓	✓	✓			✓	✓	✓	✓
(Muspratt et al., 2014)							✓		
(Cofie et al., 2009)	✓	✓	✓	✓	✓				✓

MC = Moisture content, HHV = Higher heating value

Table A2: References and analysed parameters in studies investigating properties of **separated faeces and/or mixed human excreta** samples (shown in **Figure 3.4** and **Figure 3.6**).

Reference	Analysed parameters								
	N	P	K	Ca	Mg	MC	HHV	Ash	Carbon
(Somorin et al., 2020)	✓						✓	✓	✓
(Jurado et al., 2018)	✓						✓	✓	✓
(Hafford et al., 2018)	✓						✓	✓	✓
(Yacob et al., 2018)	✓	✓	✓	✓			✓	✓	✓
(Yahav Spitzer et al., 2018)	✓						✓	✓	✓
(Somorin et al., 2017)	✓						✓	✓	✓
(Afolabi et al., 2017)	✓								✓
(Onabanjo et al., 2016a)	✓						✓	✓	✓
(Onabanjo et al., 2016b)	✓						✓	✓	✓
(Hijikata et al., 2015)	✓	✓	✓	✓	✓				✓
(Monhol & Martins, 2015)	✓								
(Meinzingner & Oldenburg, 2009)		✓	✓						
(Vinnerås et al., 2006)	✓	✓	✓						
(Schouw et al., 2002)	✓	✓	✓	✓	✓			✓	
(Czemiel, 2000)	✓	✓							
(Eastwood et al., 1984)			✓	✓	✓				
(Calloway & Margen, 1971)		✓	✓	✓	✓				
(Goldblith & Wick, 1961)		✓	✓	✓	✓				

MC = Moisture content, HHV = Higher heating value

Table A3: References and analysed parameters in studies investigating properties of **human urine** samples (shown in **Figure 3.5**).

References	Analysed parameters				
	N	P	K	Ca	Mg
(Pocock et al., 2022)	✓	✓	✓	✓	✓
(Simha et al., 2021)	✓	✓	✓	✓	✓
(Chipako & Randall, 2020b)	✓	✓	✓	✓	✓
(Xu et al., 2019)	✓	✓	✓	✓	✓
(Tarpeh et al., 2018)	✓	✓	✓		
(Moore et al., 2018)			✓	✓	✓
(Wei et al., 2018)	✓	✓	✓	✓	✓
(Krounbi et al., 2018)	✓				
(Bai et al., 2018)	✓	✓	✓		
(Pradhan et al., 2017)	✓	✓	✓		
(Amoah et al., 2017)*	✓	✓	✓		
(Barbosa et al., 2016)	✓	✓			
(Pratumvinit et al., 2013)		✓		✓	✓
(Etter et al., 2011)	✓	✓	✓	✓	✓
(Meinzinger & Oldenburg, 2009)	✓	✓	✓		
(Pradhan et al., 2009)	✓	✓	✓		
(Tilley et al., 2008)		✓		✓	✓
(Heinonen-Tanski et al., 2007)	✓	✓	✓		
(Kabdaşlı et al., 2006)	✓	✓			
(Udert et al., 2006)	✓	✓	✓	✓	✓
(Ban & Dave, 2004)	✓	✓			
(Jönsson & Vinnerås, 2004)	✓	✓	✓		
(Udert et al., 2003a)	✓	✓	✓	✓	✓

*values converted from g/kg based on daily excretion ratios presented in Rose et al. (2015)

Table A4: Faecal sludge pyrolysis publications to date, including feedstock type (and origin) and study objectives.

Reference	Feedstocks (+ country of origin)	Study objective
(Koulouri et al., 2024b)	Source-separated human faeces collected from volunteers (UK)	Carbon flows and biochar stability during co-pyrolysis of human faeces with wood biomass
(Sagor et al., 2024)	Dry faecal sludge from a treatment plant (Bangladesh)	Physical and thermal properties of briquettes produced from faecal sludge char
(Koulouri et al., 2024a)	Source-separated human excreta collected from volunteers (UK)	Faecal biochar as a carrier for N and P recovery from hydrolysed human urine via adsorption and precipitation
(Mwamlima et al., 2023)	Pit latrines, septic tanks and soakaways (Tanzania)	Faecal sludge derived char briquettes as an alternative cooking energy source
(Nicholas et al., 2023b)	-	Review of faecal sludge biochar production for agricultural applications
(Nicholas et al., 2023a)	Septic tank sludge (India)	Effect of faecal biochars (from full-scale pyrolysis plants) on tomato plant growth yield
(Jiang et al., 2023)	Human faeces samples from a source-separating toilet (China)	Faecal biochars used as a urine pre-treatment stabilisation method
(Beik et al., 2023)	Sludge from on-board train toilets (UK)	Development of a microscale twin auger pyrolysis reactor for faecal sludge treatment
(Koulouri et al., 2023)	Source-separated human excreta collected from volunteers (UK)	Effect of human excreta source separation on biochar characteristics
(Saluz et al., 2022)	Composting toilet sludge (human excreta, toilet paper, woodchips, leaves) (Switzerland)	Faecal biochar as a nutrient carrier in urban tree substrates
(Rowles et al., 2022)	Modelling study (India case study)	Environmental and financial assessment of faecal sludge pyrolysis plant
(Nicholas et al., 2022b)* *(under open peer review)	Septic tank sludge (India)	Physiochemical characterisation of biochars
(Nicholas et al., 2022a)	(survey-based)	Public perceptions of faecal sludge biochar used in agriculture
(Bittencourt & Martins, 2022)	-	Review of thermochemical treatment technologies for faecal sludge
(Islam et al., 2021)	-	Review of biochars (including faecal-derived) for water and soil remediation
(Beik et al., 2021)	-	Review of thermochemical treatment technologies for faecal sludge
(Krueger et al., 2021b)	Pit latrine and septic tank sludge (South Africa)	Feedstock characterisation (of organic composition) to inform thermal decomposition behaviour

(Krueger et al., 2021a)	Pit latrine and septic tank sludge (South Africa)	• Feedstock characterisation to inform thermal treatment processes
(Bleuler et al., 2020)	'Dry toilet' faecal sludge (mix of human faeces, urine, toilet paper and wood chips (25%)) (Switzerland)	• Soil amendment performance
(Somorin et al., 2020b)	Human faeces, simulated faeces and wood biomass (UK)	• Thermal analysis • Evolved gases characterisation and quantification
(Krueger et al., 2020)	Septic tank sludge (India)	• Resource recovery performance (soil amendment production, fuel production)
(Nuagah et al., 2020)	Faecal sludge with sawdust and sewage sludge (Ghana)	• Fertiliser performance
(Krounbi et al., 2020)	Sludge from five urine-diversion latrines (Kenya)	• Sequential ammonia and carbon dioxide adsorption on biochar
(Barani et al., 2020)	Septic tank sludge (India)	• Energy recovery performance
(Hübner et al., 2019)	Composting toilet sludge (faecal sludge, sawdust and toilet paper) (Germany)	• Suitability of feedstock for carbonisation (pyrolysis, HTC) • Mass and energy balance calculations.
(Andriessen et al., 2019)	-	• Review of FS treatment technologies producing carbonised (pyrolysis, HTC) and non-carbonised fuels (drying, pelletising)
(Fidalgo et al., 2019)	Human faeces, simulated faeces and wood biomass (UK)	• Thermal and kinetics analysis to inform thermochemical conversion processes
(Krounbi et al., 2019)	Container-based sludge (human faeces and sawdust) (Kenya)	• Soil amendment performance • Financial analysis • Comparison against biological treatment (composting) and torrefaction
(Koetlisi & Muchaonyerwa, 2019)	Pit latrine sludge, sewage sludge and pine-bark (South Africa)	• Sorption of various heavy metals in single- or multiple- metal solutions
(Myers et al., 2019)	Wood pellets as simulated faecal sludge (India ¹¹)	• Energy recovery analysis
(Bai et al., 2018)	Septic tank sludge (China)	• Ammonium adsorption from urine using FS biochar as a carrier • Soil amendment performance of ammonium-saturated biochar
(Bond et al., 2018)	Pit latrine sludge (n/a – data from multiple studies)	• Suitability of pit latrine sludge as a feedstock for bio-oil and biochar production • Energy balance

¹¹ Although surrogate material was used, the study focuses on India case-studies.

(Gold et al., 2018a)	Pit latrine and septic tank sludge (Uganda)	• Resource recovery performance (soil amendment production, fuel production, carbon sequestration) for varying pyrolysis operational parameters
(Yacob et al., 2018)	Human faeces (USA)	• Thermal analysis • Evolved gases quantification and qualification • Kinetics modelling
(Krounbi et al., 2018)	Container-based sludge (human faeces and sawdust) and urine (Kenya)	• Nutrient availability in faecal-derived soil amendments • Comparison against biological treatment (composting) and torrefaction • Effects of additional urine application
(Woldetsadik et al., 2017)	Septic tank sludge (Ethiopia)	• Fertiliser performance
(Starkenmann et al., 2017)	Container-based sludge (human faeces, sawdust and toilet paper) (Kenya)	• Producing a perfume delivery system
(Koetlisi & Muchaonyerwa, 2017)	Pit latrine and sewage sludge (South Africa)	• Cadmium adsorption performance
(Woldetsadik et al., 2016)	Septic tank sludge (Ethiopia)	• Cadmium immobilisation performance
(Ward et al., 2014)	Human faeces and simulated faeces (USA)	• Solid fuel production for energy recovery
(Liu et al., 2014)	Septic tank sludge (<i>'human manure'</i>) (China)	• Energy recovery performance

Appendix C. Ethical approval documents for human excreta sampling

1. Participant Information Sheet

Version 3.0

Date: 28/05/2021

Author: Maria Koulouri

Study Title:

Experimental study on resource recovery from human excreta

You are being invited to take part in a research study. Before you decide it is important for you to understand why the research is being done and what it will involve. Please take time to read the following information carefully and discuss it with others if you wish.

The purpose of this study:

The objective of the study is to investigate methods for recovering nutrients from human excreta (which is faeces and urine) in order to produce faecal-based organic fertilisers. This is particularly important for developing countries, where management of human excreta, access to safe and sustainable sanitation and food security are prevalent issues to be addressed.

One option for human excreta treatment and resource recovery is pyrolysis, the thermochemical conversion of biomass in an oxygen-limited or oxygen-free environment at temperatures from 300 °C to 700 °C. Pyrolysis of faecal sludge produces a nutrient-rich biochar that can be used in agriculture. This study will investigate ways to optimise nutrient recovery from source-separated human excreta through pyrolysis and assess the benefits of applying human excreta-derived fertilisers to soil properties and plant growth.

Why have I been invited?

We are looking for ~20 healthy adults to take part in this study.

Do I have to take part?

It is up to you to decide whether or not to take part. If you do decide to take part, you will be given this information sheet to keep and be asked to sign a consent form. If you decide to take part, you are still free to withdraw at any time without giving a reason for doing so.

What do I have to do if I take part?

There will be no lifestyle, dietary or any other restrictions during your participation. You will be asked to voluntarily disclose your usual diet type (vegetarian/vegan or non-vegetarian) but there is no need to change any of your habits to take part.

For this study, you will need to provide samples of faeces and urine for **10-15 days**. These days do not need to be consecutive and can be spread over the duration of ~6 weeks (~2-3 collection days per week). Each time, you will be provided with a kit containing suitable containers and plastic bags to collect the samples, as well as detailed instructions on how to collect samples.

When should I NOT collect samples?

Please **do not** collect samples if you are feeling unwell (including if you have COVID-19 symptoms or have tested positive for COVID-19 over the past 14 days), have symptoms of diarrhoea, see any blood in your poo, have a high temperature/ fever ($\geq 37.2^{\circ}\text{C}$), or if you have had any of these symptoms over the last 72 hours.

For female participants: please **do not** collect samples during menstruation.

If during the course of the study you develop any gastrointestinal condition, urinary infection or any condition that can be transmitted through human excreta, please **stop collecting samples** until you recover or withdraw from the study.

Please know that you **do not** have to provide a reason for choosing not to collect samples or withdrawing from the study and you will not be asked to share that information in any case.

What are the possible disadvantages and risks of taking part?

There are no risks for you if you choose to take part in this study.

What if something goes wrong?

If you are harmed by taking part in this research project, there are no special compensation arrangements. If you are harmed due to someone's negligence, then you may have grounds for a legal action. Regardless of this, if you wish to complain, or have any concerns about any aspect of the way you have been treated during the course of this study then you should immediately inform one of the Investigators (contact details in cover page). If you are still not satisfied with the response, you may contact the Imperial College [Research Governance Integrity Team \(rgitcoordinator@imperial.ac.uk\)](mailto:rgitcoordinator@imperial.ac.uk).

What will happen to the results of the research study?

Results are likely to be published during the course of the PhD project (i.e. over the next 3 years) and will be included in the final thesis of the project. If you wish, you can be informed of any published results via e-mail through the course of the project. Please initial the relevant box of the attached Consent Form to express your interest in receiving an e-mail when results have been published.

All analyses and results will be pseudonymised and you will not be identified in any report or publication. Some results may be grouped based on diet type (vegetarian/vegan and non-vegetarian), in which case you will know which group your data have been included in.

Who is organising and funding the research?

This study is part of a PhD research project funded by the Department of Civil and Environmental Engineering of Imperial College.

Who has reviewed the study?

This study was given ethical approval by [REDACTED].

Contact for further information:

If you have questions you can contact Maria Koulouri (PhD Researcher) at:

e-mail: [REDACTED].

contact number: [REDACTED].

Transparency Notice**How will your information be used?****Research Study Title: Experimental study on resource recovery from human excreta**

Imperial College London is the sponsor for this study and will act as the data controller for this study. This means that we are responsible for looking after your information and using it properly. Imperial College London will keep your personal data for:

10 years after the study has finished in relation to data subject consent forms.

10 years after the study has completed in relation to primary research data.

We will need to use information from you from this research project. This information will include your initials, name, contact details (e-mail and/or phone number) and diet type. People will use this information to do the research or to check your records to make sure that the research is being done properly.

People who do not need to know who you are will not be able to see your name or contact details. Your data will have a code number instead.

We will keep all information about you safe and secure.

Once we have finished the study, we will keep some of the data so we can check the results. We will write our reports in a way that no-one can work out that you took part in the study.

Legal basis

As a university we use personal information to conduct research that will either be in the public interest, or the legitimate interests of the College.

As a publicly-funded organisation we have to ensure that when we use personally-identifiable information from people who have agreed to take part in research, it is either in the public interest, or that our legitimate purposes are both properly notified to you and are not overridden by your rights.

We will conduct scientific research in compliance with the law and the recommendations and guidance published by the UK Information Commissioners Office (ICO).

International Transfers

There may be a requirement to transfer information to countries outside the European Economic Area (for example, to a research partner). Where this information contains your personal data, Imperial College London will ensure that it is transferred in accordance with data protection legislation. If the data is transferred to a country which is not subject to a European Commission (EC) adequacy decision in respect of its data protection standards, Imperial College London will enter into a data sharing

agreement with the recipient organisation that incorporates EC approved standard contractual clauses that safeguard how your personal data is processed.

Sharing your information with others

For the purposes referred to in this privacy notice and relying on the bases for processing as set out above, we will share your personal data with certain third parties.

- Other College employees, agents, contractors and service providers (for example, suppliers of printing and mailing services, email communication services or web services, or suppliers who help us carry out any of the activities described above). Our third party service providers are required to enter into data processing agreements with us. We only permit them to process your personal data for specified purposes and in accordance with our policies.

What are your choices about how your information is used?

You can stop being part of the study at any time, without giving a reason, but we will keep information about you that we already have.

- We need to manage your records in specific ways for the research to be reliable. This means that we won't be able to let you see or change the data we hold about you.
- If you agree to take part in this study, you will have the option to take part in future research using your data saved from this study.

Where can you find out more about how your information is used?

You can find out more about how we use your information:

- by asking one of the research team
- by sending an email to [REDACTED], or
- by ringing us on [REDACTED].

Complaint

If you wish to raise a complaint on how we have handled your personal data, please contact Imperial College London's Data Protection Officer via email at [REDACTED], via telephone on [REDACTED] and/or via post at [REDACTED].

If you are not satisfied with our response or believe we are processing your personal data in a way that is not lawful you can complain to the Information Commissioner's Office (ICO). The ICO does recommend that you seek to resolve matters with the data controller (us) first before involving the regulator.

Thank you for taking the time to read this information sheet and considering participating in this study. Please ask us if there is anything that is not clear or if you would like more information. Take time to decide whether you wish to take part. If you decide to take part in the study, you will need to sign the attached Consent Form.

A copy of the written information and signed Informed Consent form will be given to the participant to keep.

2. Instructions provided to volunteers

For faeces collection

Sampling kit:

- 750 mL plastic rectangular containers with lid
- Cling film
- Disposable nitrile gloves
- Nappy bags
- Plastic zip-lock bags for double containment

Instructions:

- “You will be given a designated container, labelled with a number, and a plastic zip-lock bag. Do not write your name or any other personal information on the container.
- To collect the faeces, you can place the cling film provided over the rim of the toilet to catch the sample. It is recommended that you place the cling film at the back of the toilet rim so you can use the front for urination.
- You can wear a pair of clean disposable gloves if you want and use a nappy bag to carefully move the sample from the cling film inside the plastic container. You should not need to come in contact with the faeces sample at all for this process.
- Immediately turn the used nappy bag inside-out.
- You can then remove the cling film (touching it only from the sides) and place it inside the nappy bag. Also place the gloves in the nappy bag, without touching the outside. Again, you should not need to come in contact with faeces at all for this process.
- Close the nappy bag by making a tight knot. You can discard this in your household trash.
- Place the lid on the container and ensure it is shut well. Then place the container inside the provided plastic zip-lock bag.
- Wash your hands thoroughly.”

For urine collection

Sampling kit:

- HDPE wide-neck leak proof bottles (autoclavable) (500 mL)
- Plastic zip-lock bags for double containment

Instructions:

- “You will be given a designated screw-top container, labelled with a number, and a plastic zip-lock bag. Do not write your name or any other personal information on the container.
- Start to pee and collect the urine in the container.

- Screw the lid of the container shut. Please give care to ensure the lid is fully closed.
- You can repeat the process more than once until the container is near-full but always allowing some space at the top to ensure no spillage. Screw the lid well after each urination.
- Place the container inside the provided plastic zip-lock bag.
- Wash your hands thoroughly.”

Storing and handing in samples

“If you collect your samples at home, we understand that you may not be able to hand them in immediately. Please try to collect samples no more than 24 h before handing them in. In the meantime, you should keep samples in a cool place (inside containers and sealed plastic bags).”

Safety & hygiene

Additional (opaque) bags were provided to transport the samples. The plastic containers used for faeces collection were tested in the laboratory oven at 110 °C to ensure they are safe for drying of the samples. Samples were only removed from containers after an initial drying step (48 h or until constant weight) to ensure the safety of the researchers. The lid was removed during drying. A second dry-sterilisation step, at 150 °C for 150 min, was performed in glass Pyrex containers.

Urine bottles were autoclaved and washed in a biological cycle between uses. Faeces containers were autoclaved and disposed as biologically hazardous waste after use.

3. Consent Form for Participants Able to Give Consent

Full Title of Project:

Experimental study on resource recovery from human excreta

Name of Principal Investigator:

**Please initial box to
consent**

1. I confirm that I have read and understand the participant information sheet version 3.0 dated 28/05/2021 for the above study and have had the opportunity to ask questions which have been answered fully.	
2. I understand that my participation is voluntary, and I am free to withdraw at any time, without giving any reason and without my legal rights being affected.	
3. I give/ do not give consent for pseudonymised information collected about me to be used to support other research in the future, including those outside of the European Economic Area (EEA).	
4. I give/ do not give consent for samples (human faeces and urine) collected during this study to be used in future ethically approved studies. I give permission for my samples to be sent to other organisations, including those outside of the European Economic Area (EEA).	
5. I consent to take part in the above study.	
6. I give consent to pseudonymised results of this study being published in the form of a dissertation, academic journals/ books or other scientific publications. (If you want to be contacted via e-mail about published results of this study, please provide your preferred contact address here:)	

7. I give/ do not give consent to being included in a draw for a chance to win a £50 Amazon voucher (1 of 2) and being contacted via e-mail if I win. (The draw will take place immediately after the end of the samples collection period.) (If yes, please provide your preferred contact address here:)	
8. I give/ do not give consent to being contacted about the potential to take part in other research studies.	
9. I give permission for Imperial College London to access my research records that are relevant to this research.	

Name of participant Signature Date

Name of person taking consent Signature Date
(if different from Principal Investigator)

Principal Investigator Signature Date

1 copy for participant; 1 copy for Principal Investigator

01 June 2021

Dear [REDACTED]

Study Title: Experimental study on resource recovery from human excreta

Study number: 21IC6817

The above study was approved by your Head of Department on 27/04/21 and by the Research Governance and Integrity Team on 01/06/21.

Under the Science Engineering Technology Research Ethics Committee process, a study that has been reviewed by the Research Governance and Integrity Team and Head of Division/Department, where no significant ethical issues have been identified in the protocol or ethics application, can be given RGIT approval without requiring it to go to full committee.

Documents

The documents reviewed were:

- Application form (v3.0 28/05/2021)
- Protocol (v3.0 28/05/2021)
- Participant Information Sheet (v3.0 28/05/2021)
- Consent Form (v3.0 28/05/2021)
- Oral Script/ Recruitment email/ Instruction Sheet (v3.0 28/05/2021)

Yours sincerely,

[REDACTED]

Digitally signed by [REDACTED]

Date: 2021.06.01 16:10:21
+01'00'

Head of Research Governance and Integrity,
Imperial College London

Imperial College Healthcare Tissue Bank
Department of Surgery and Cancer,
Charing Cross Hospital
Fulham Palace Road
London W6 8RF
Tel: 020 3311 7173
Email: tissuebank@imperial.ac.uk

ICHTB HTA licence: 12275
REC approval: 17/WA/0161

25th October 2021

Dear [REDACTED]

**Re: Application for Access Approval.
N21005: Resource Recovery from Human Excreta (expiring Oct 2024)**

This is to confirm that the above project has been approved by ICHTB. REC3 Wales has confirmed that approval via ICHTB Application Review Panel provides you with deemed ethics approval for your research project. Details of your research project will be provided to REC 3 Wales through ICHTB's annual report.

ICHTB is supported by funding from the NIHR Imperial Biomedical Research Centre (BRC). Acknowledgement of this infrastructural report is required in any publication that results from your research project, and you have agreed to this by signing our MDTA as part of your application. The recommended wording is as follows: "Human samples used in this research project were obtained from the Imperial College Healthcare Tissue Bank (ICHTB). ICHTB is supported by the National Institute for Health Research (NIHR) Biomedical Research Centre based at Imperial College Healthcare NHS Trust and Imperial College London. ICHTB is approved by Wales REC3 to release human material for research (17/WA/0161)". Further information on Imperial's BRC can be found here <https://imperialbrc.nihr.ac.uk/about-us/acknowledgement/>.

Should you wish to amend any details of your project, please contact the [Tissue Bank](#).

We would be grateful if you would send copies of or DOI links for any publication that results from this study to the [Tissue Bank](#). Your support with this helps us to keep the Tissue Bank running as a research resource for all.

If you have any questions, please see contact details above.

Yours sincerely,

[REDACTED]

DI, HTA Research Licence.

Appendix D. Quality control assurance

Pilot-scale pyrolysis

The accuracy of temperature readings during pyrolysis experiments was validated via blank runs (using a thermocouple placed at the centre of the quartz reactor), which showed minimal deviation (<5%) between the set and actual temperature in the furnace (see **Figure A1**).

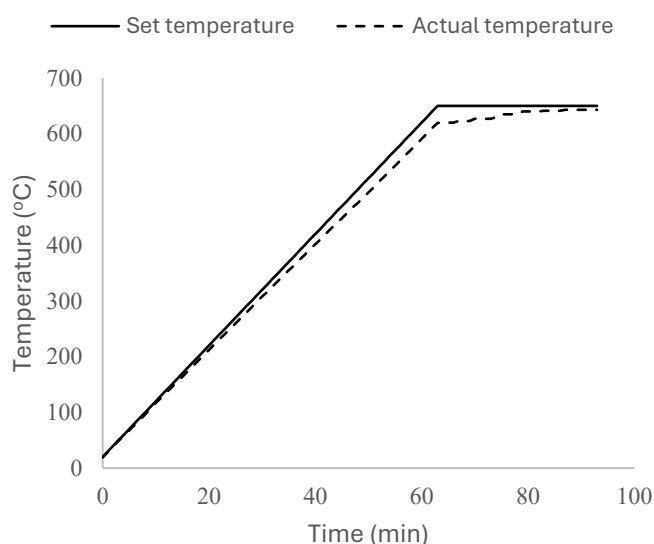


Figure A1: Temperature readings (set and actual) during pyrolysis at the experimental set-up at the Roger Perry Laboratory, Imperial College London (for 650 °C highest heating temperature, 30 min hold time, 1.5 L/min N₂ flow rate and 10 °C/min heating rate).

Inductively Coupled Plasma Optical Emission spectroscopy

For Inductively Coupled Plasma Optical Emission spectroscopy (ICP-OES) analysis, quality control was assured by complying with requirements for blank and spiked replicates set out in standard method 6010 D (US EPA, 2018). The instrument was calibrated before each analysis, with solutions prepared by diluting ICP standards in a 1M HNO₃ matrix. The standards used were the Sigma Aldrich ICP 1000 mg/L phosphorus and 1000 mg/L multi-element IV (23 elements) standard solutions in diluted HNO₃. Indicative calibration curves for selected elements are shown in **Figure A2**. Additional quality control checks were carried out during each analysis, by analysing solutions of known concentrations at regular intervals. All analysed solutions were diluted in 1 M HNO₃ to match the matrix of the standard solutions used for calibration. XRF analysis, which has higher sensitivity for trace elements, was used for validation of the acid digestion method followed by ICP-OES analysis and results are presented in **Table A5**.

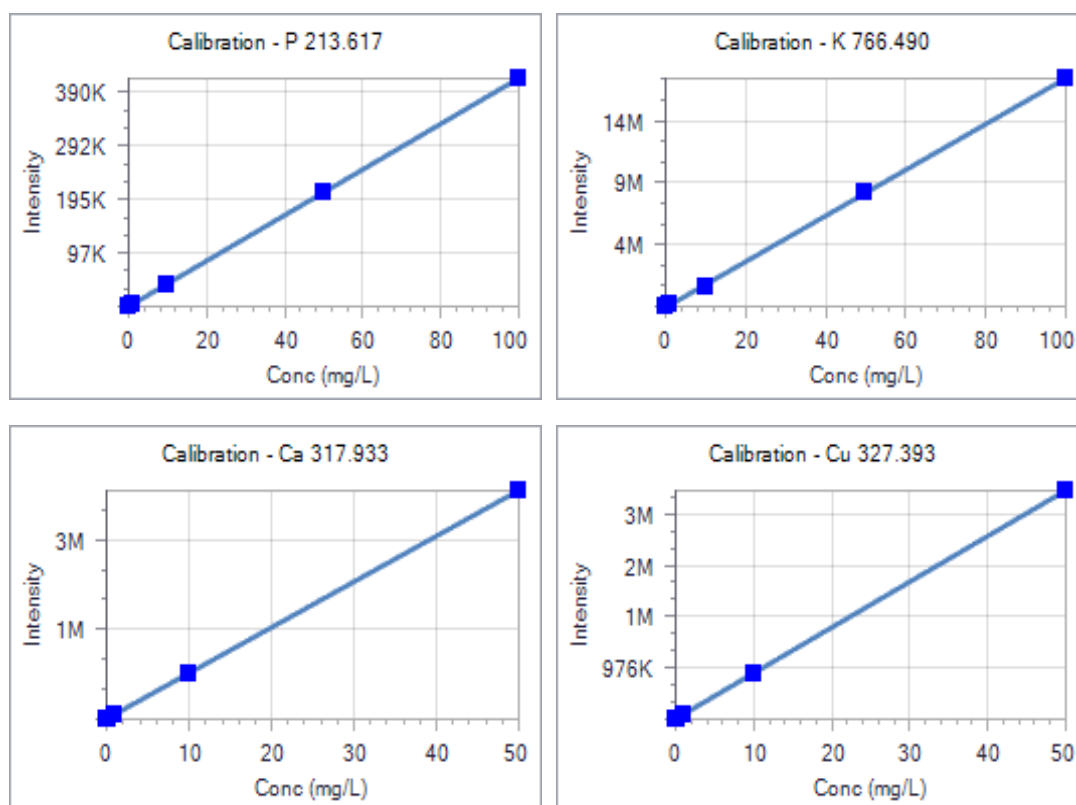


Figure A2: Indicative calibration curves for selected elements during Inductively Coupled Plasma Optical Emission spectroscopy (ICP-OES) analysis on an Avio 500 (Perkin Elmer, USA) [data acquired on 21 July 2022].

Table A5: X-ray fluorescence (XRF) analysis of source-separated faeces (SSF) biochar (produced at 550 °C) supplementing data from Inductively Coupled Plasma Optical Emission spectroscopy (ICP-OES) analysis presented in **Chapter 5**.

Compound	Unit	Concentration
Na ₂ O	[g/kg]	3.83
MgO	[g/kg]	19.09
Al ₂ O ₃	[g/kg]	0.56
SiO ₂	[g/kg]	7.17
P ₂ O ₅	[g/kg]	109.71
SO ₃	[g/kg]	2.97
K ₂ O	[g/kg]	54.15
CaO	[g/kg]	77.33
MnO	[mg/kg]	492
Fe ₂ O ₃	[mg/kg]	1668
NiO	[mg/kg]	49.8
CuO	[mg/kg]	176.88
ZnO	[mg/kg]	598
SrO	[mg/kg]	176.4
I	[mg/kg]	199.11

Carbon, Hydrogen, Nitrogen, Sulphur and Oxygen analysis

For CHNS(O) analysis, quality control was assured by analysing a blank sample and a bypass sample (i.e. qualitative analysis of standard compound) before each run as well as by analysing standard compounds of known CHNSO composition at regular intervals. The ThermoScientific Flash Smart Elemental Analyzer was calibrated based on the requirements of standard method ISO 16948:2015 and recommended reference materials were used for the calibration and quality control testing (e.g. cystine, methionine, sulphanilamide) (ISO, 2015). An indicative calibration curve for C (% w/w) is shown in **Figure A3**.

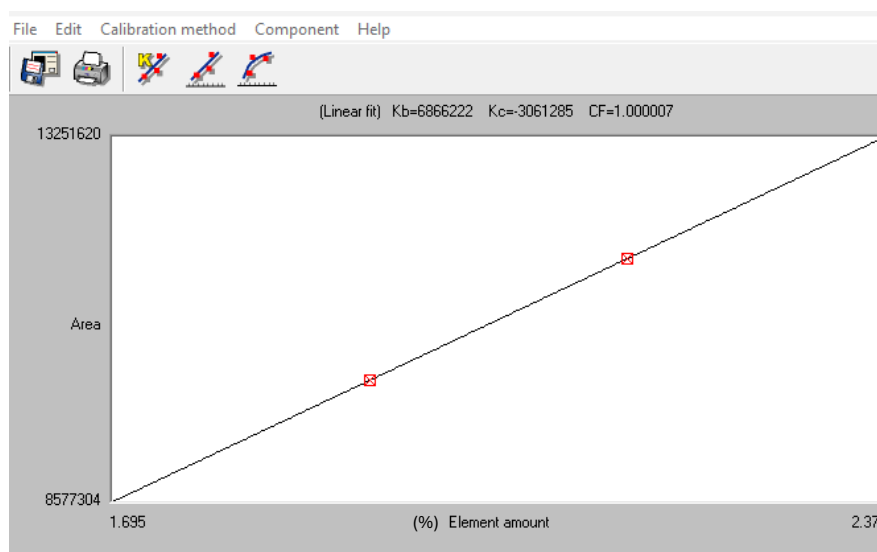


Figure A3: Indicative calibration curve for carbon on a ThermoScientific Flash Smart Elemental Analyzer [data acquired on 6 March 2024].

pH and electrical conductivity testing

The pH meter was calibrated biweekly using buffer solutions with pH 4, 7 and 10. The electrical conductivity (EC) meter was calibrated biweekly using freshly prepared KCl solutions with concentrations of 0.1, 0.01 and 5.94×10^{-4} mol/L.

Skalar analysis for total $\text{NH}_4\text{-N}$

For $\text{NH}_4\text{-N}$ analysis, quality control was assured via the use of blanks and spiked samples, using a standard NH_4Cl solution of known concentrations. The machine was calibrated before each run using a stock NH_4Cl solution (100 mg N/ L) subsequently diluted by the Skalar autosampler. An indicative calibration curve is shown in **Figure A4**.

During $\text{NH}_4\text{-N}$ adsorption experiments (refer to **Chapter 6**), water leaching tests were carried out for biochar samples, as quality control blanks, to ensure elemental concentration differences measured during experiments referred to actual adsorption and/or ion exchange reactions and were accurately quantified. Volatile N losses were also monitored and were <5% of TN for all experiments (from original TN concentration of fresh urine, throughout storage [6 months] and adsorption experiments).

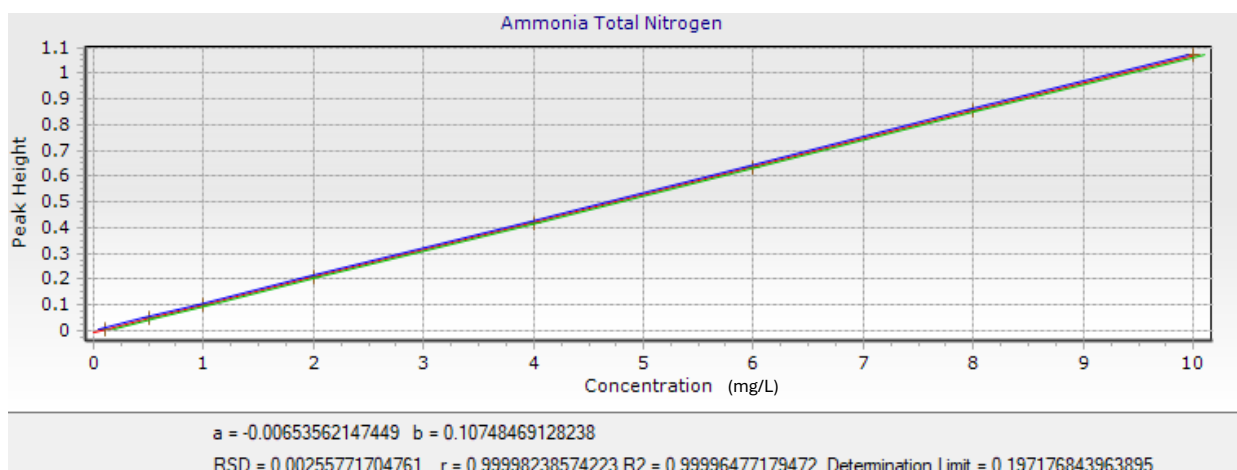


Figure A4: Indicative calibration curve for total $\text{NH}_4\text{-N}$ on a Skalar auto-analyser (Skalar Analytical BV) [data acquired on 8 February 2023].

Thermogravimetric analysis

For thermogravimetric (TG) analysis, the TG data were internally corrected with regular blank tests using an empty crucible to account for buoyancy effects.

Proximate analysis

The determination of proximate analysis results (moisture, volatile matter, ash and fixed carbon content) was performed based on TGA analysis outputs (as described in **Section 4.3.1.1**). An indicate TGA plot showing how each value was determined is shown in **Figure A5**.

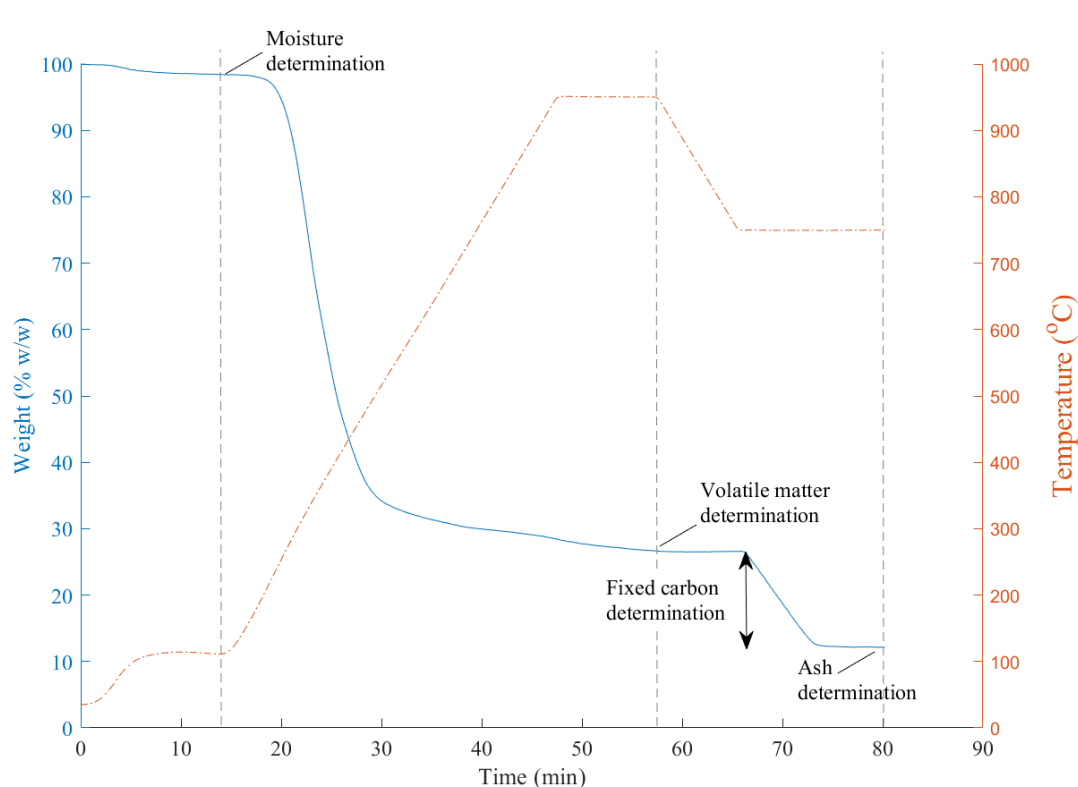


Figure A5: Thermogravimetric analysis (TGA) plot for proximate analysis determination (on an STA 449 F5 Jupiter).

Appendix E. Preliminary assessment of pyrolysis retention times ¹²

A preliminary assessment of the effect of pyrolysis retention time at the three temperatures tested (450 °C, 550 °C, 650 °C) was performed via thermogravimetric analysis on an STA 449 F5 Jupiter (using ~15 mg sample of separated human faeces). The remaining weight (% of total) at the end of each run is shown in **Table A6**. The loss of weight per minute for each 15 min period is noted in brackets. Weight loss was considered insignificant when <0.1%/min, hence 30 min was considered acceptable as the isothermal retention time for this study.

This preliminary assessment was validated for bigger sample sizes (30 g), at the 30 min and 60 min retention time scenarios, using a Carbolite rotary furnace (see **Section 4.2**), to account for potential heat transfer limitations of the laboratory-scale furnace used for the experimental design. The difference in weight loss between the two scenarios was 0.05%/min at 450 °C and 550 °C, and 0.07%/min at 650 °C, confirming that insignificant weight loss occurs after the first 30 min at these temperatures. Therefore, an isothermal retention time of 30 min was deemed adequate to ensure the designated temperature is reached for the whole sample mass and that all pyrolytic reactions at this temperature range are complete.

Table A6: Effect of pyrolysis isothermal retention time on weight loss during thermogravimetric analysis of human faeces samples (remaining % of total) for three pyrolysis temperatures (450, 550, 650 °C).

Hold time	Pyrolysis temperature		
	450 °C	550 °C	650 °C
1min	39.53%	34.39%	32.66%
15min	36.76% (0.18%/min)	33.09% (0.09%/min)	31.07% (0.11%/min)
30min	35.8% (0.06%/min)	32.15% (0.06%/min)	29.74% (0.09%/min)
45min	35.18% (0.04%/min)	31.39% (0.05%/min)	28.55% (0.08%/min)
60min	34.68% (0.03%/min)	30.7% (0.05%/min)	27.49% (0.07%/min)

¹² Supplementary information for the data presented in **Chapter 5**.

Appendix F. Statistical analyses results

Table A7: p-values of two-way ANOVA for differences in biochar characteristics with source separation and for different pyrolysis temperatures (including multiple comparison test p-values) (**bold** for significant differences $p < 0.05$).

Parameter tested	Two-way ANOVA p-values			Multiple comparison test: Pyrolysis temperature (°C)		
	Temperature	Source separation	Interaction	650-550	650-450	550-450
Char yield	<0.001	<0.001	0.013	<0.001	<0.001	<0.001
VM	<0.001	<0.001	0.282	0.001	<0.001	<0.001
FC	<0.001	<0.001	0.005	0.182	<0.001	<0.001
Ash	<0.001	<0.001	0.001	<0.001	<0.001	<0.001
HHV	0.003	<0.001	0.113	0.677	0.015	0.003
C	<0.001	<0.001	0.008	0.029	<0.001	0.062
H	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001
N	<0.001	0.019	<0.001	<0.001	<0.001	<0.001
S	<0.001	<0.001	0.256	0.228	<0.001	0.008
pH	<0.001	<0.001	0.002	<0.001	<0.001	<0.001
EC	<0.001	<0.001	<0.001	0.048	<0.001	0.010
CaCO ₃ -eq.	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001
CEC	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001
BET	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001
P	<0.001	<0.001	0.692	0.009	<0.001	<0.001
P (2% FA)	<0.001	<0.001	<0.001	0.160	<0.001	<0.001
Olsen-P	<0.001	<0.001	<0.001	0.086	<0.001	<0.001
K	<0.001	<0.001	<0.001	0.056	<0.001	<0.001
Ca	<0.001	<0.001	0.007	0.011	<0.001	0.009
Mg	<0.001	<0.001	0.568	0.077	<0.001	0.006
B	0.004	<0.001	0.613	0.007	0.008	0.999
Zn	0.002	<0.001	0.010	0.009	0.002	0.720
Mn	0.018	<0.001	0.194	0.093	0.017	0.607
Fe	0.304	<0.001	0.065	0.286	0.553	0.859
Cu	<0.001	<0.001	<0.001	<0.001	<0.001	0.013
Ba	0.747	<0.001	0.078	0.955	0.730	0.883

VM = Volatile matter, FC = Fixed carbon, HHV = Higher heating value, EC = Electrical conductivity, CaCO₃-eq. = Calcium carbonate equivalence, CEC = Cation exchange capacity, P (2% FA) = 2% formic acid extractable P

Table A8: One-way ANOVA results for the evolution of NH₄-N content over time during urine storage at room temperature and over a period of 180 days (**bold** for significant differences p<0.05).

Storage period	Lower 95% CI	Mean difference	Upper 95% CI	One-way ANOVA p-values
1 day – 4 days	-2.186	-1.961	-1.736	<0.001
4 days – 7 days	-1.444	-1.187	-0.929	<0.001
7 days – 10 days	-1.127	-0.779	-0.430	0.003
10 days – 13 days	-1.475	-0.910	-0.345	0.011
13 days – 20 days	-457.522	15.772	489.066	0.931
20 days – 30 days	-680.419	-408.701	-136.983	0.014
30 days – 90 days	-667.637	-369.603	-71.569	0.026
90 days – 120 days	-642.012	-342.800	-43.588	0.034
120 days – 160 days	-500.830	-157.200	186.430	0.273
160 days – 180 days	-318.145	-50.000	218.145	0.632

Table A9: p-values of one-way ANOVA for differences in NH₄-N adsorption from urine onto faecal-derived biochar over time, during the kinetics study experiments (**bold** for significant differences p<0.05).

	05h-1h	1h-2h	2h-4h	4h-8h	8h-24h	24h-28h	28h-30h	30h-48h
Real stored urine	0.172	0.009	0.088	0.022	0.044	0.560	0.907	0.717
Synthetic NH ₄ Cl solution	0.254	0.014	0.001	0.007	0.028	0.635	n/a	0.335

Table A10: p-values of two-way ANOVA for differences in N and P recovery with changes in biochar and MgO dosing, including multiple comparison test p-values (**bold** for significant differences p<0.05).

		N recovery	P recovery
Two-way ANOVA p-values	Biochar dose	<0.001	0.567
	MgO dose	0.097	<0.001
	Interaction	0.048	0.1978
Multiple comparison test: Biochar dose	25 g/L – 50 g/L	0.014	0.987
	25 g/L – 100 g/L	<0.001	0.675
	50 g/L – 100 g/L	0.005	0.583

Table A11: p-values of one-way ANOVA for differences in feedstock C and proximate analysis results, with changes in HF:WB blending ratio (HF, HF75, HF50, HF25, WB) (**bold** for significant differences $p < 0.05$).

	C	VM	FC	Ash
HF vs. HF75	0.1314	0.7497	<0.001	<0.001
HF vs. HF50	0.0344	0.0819	<0.001	<0.001
HF vs. HF25	0.0226	0.0577	<0.001	<0.001
HF vs. WB	0.0159	0.0397	<0.001	<0.001
HF75 vs. HF50	0.008	<0.001	<0.001	0.0014
HF75 vs. HF25	0.0097	<0.001	<0.001	<0.001
HF75 vs. WB	0.0086	0.0025	<0.001	<0.001
HF50 vs. HF25	0.1963	0.3731	0.0098	0.0066
HF50 vs. WB	0.0739	0.1554	<0.001	<0.001
HF25 vs. WB	0.3608	0.2476	0.0011	0.0011

VM= Volatile matter, FC = Fixed carbon

Table A12: p-values of two-way ANOVA for differences in biochar yield, proximate analysis and water-extractable dissolved organic carbon (DOC) results, with changes in pyrolysis temperature (450, 550, 650 °C) and HF:WB ratio (B-HF, B-HF75, B-HF50, B-HF25, B-WB) (**bold** for significant differences $p < 0.05$).

	Yield	VM	FC	Ash	DOC
Pyrolysis temperature	<0.001	<0.001	<0.001	<0.001	<0.001
HF:WB ratio	<0.001	<0.001	<0.001	<0.001	<0.001
Interaction	0.0278	<0.001	<0.001	<0.001	<0.001
Multiple comparison test - Pyrolysis temperature					
450-550 °C	<0.001	<0.001	<0.001	<0.001	<0.001
450-650 °C	<0.001	<0.001	<0.001	<0.001	<0.001
550-650 °C	<0.001	<0.001	<0.001	<0.001	0.0553
Multiple comparison test – HF:WB ratio					
B-HF vs. B-HF75	0.0177	0.9884	<0.001	<0.001	<0.001
B-HF vs. B-HF50	<0.001	0.03623	<0.001	<0.001	<0.001
B-HF vs. B-HF25	<0.001	0.02035	<0.001	<0.001	<0.001
B-HF vs. B-WB	<0.001	0.00100	<0.001	<0.001	<0.001
B-HF75 vs. B-HF50	<0.001	0.0516	<0.001	<0.001	<0.001
B-HF75 vs. B-HF25	<0.001	0.00992	<0.001	<0.001	<0.001
B-HF75 vs. B-WB	<0.001	<0.001	<0.001	<0.001	<0.001
B-HF50 vs. B-HF25	<0.001	0.9581	<0.001	<0.001	0.2541
B-HF50 vs. B-WB	<0.001	0.3704	<0.001	<0.001	0.0954
B-HF25 vs. B-WB	<0.001	0.7763	<0.001	<0.001	0.9846

VM= Volatile matter, FC = Fixed carbon, DOC = Dissolved organic carbon

Table A13: p-values of two-way ANOVA for differences in biochar stability (H_2O_2 oxidation, R_{50} recalcitrance index) and carbon retention, with changes in HF:WB blending ratio (B-HF, B-HF75, B-HF50, B-HF25, B-WB) and pyrolysis temperature (550, 650 °C) (**bold** for significant differences $p < 0.05$).

	H_2O_2 oxidation	R_{50} index	Carbon retention
Pyrolysis temperature	<0.001	0.1994	<0.001
HF:WB ratio	<0.001	<0.001	<0.001
Interaction	<0.001	0.0145	<0.001
Multiple comparison test – HF:WB ratio			
B-HF vs. B-HF75	<0.001	0.90544	0.00139
B-HF vs. B-HF50	<0.001	0.02448	<0.001
B-HF vs. B-HF25	<0.001	<0.001	<0.001
B-HF vs. B-WB	<0.001	0.00316	<0.001
B-HF75 vs. B-HF50	<0.001	0.13885	<0.001
B-HF75 vs. B-HF25	<0.001	<0.001	<0.001
B-HF75 vs. B-WB	<0.001	0.02169	<0.001
B-HF50 vs. B-HF25	0.09070	0.18429	0.62127
B-HF50 vs. B-WB	<0.001	0.8844	0.73968
B-HF25 vs. B-WB	<0.001	0.64576	0.09980

Table A14: Two-way ANOVA results for differences in carbon flows to the biochar fraction, with changes in wood addition (HF, HF50, WB) and isothermal retention time (0.5, 2 h) (**bold** for significant differences $p < 0.05$).

ANOVA table for biochar carbon flows (1)					
Effect of wood addition & isothermal retention time					
	SS	df	MS	F	p-Value
Retention time	5.445	1	5.445	4.4	0.0577
Feedstock type	318.69	2	159.345	128.85	<0.001
Interaction(time:type)	0.09	2	0.045	0.04	0.9644
Error	14.84	12	1.237		
Total	339.065	17			
Multiple comparison test: Feedstock type (HF, HF50, WB)					
	Lower 95% CI	Mean difference	Upper 95% CI		p-Value
HF vs. HF50	-10.7129	-9	-7.2871		<0.001
HF vs. WB	-10.5629	-8.85	-7.1371		<0.001
HF50 vs. WB	-1.5629	0.15	1.8629		0.9704

Table A15: Two-way ANOVA results for differences in carbon flows to the biochar fraction, with changes in wood addition (HF, HF50, WB) and N₂ gas flow rate (0.5, 1.5 L/min) (**bold** for significant differences p<0.05).

ANOVA table for biochar carbon flows (2)					
Effect of wood addition & N₂ gas flow rate					
	SS	df	MS	F	p-Value
N ₂ gas flow	37.845	1	37.845	28.03	<0.001
Feedstock type	144.39	2	72.195	53.48	<0.001
Interaction(gas:type)	36.39	2	18.195	13.48	<0.001
Error	16.2	12	1.35		
Total	234.825	17			
Multiple comparison test: Feedstock type (HF, HF50, WB)					
	Lower 95% CI	Mean difference	Upper 95% CI		p-Value
HF vs. HF50	-8.2897	-6.5	-4.7103		<0.001
HF vs. WB	-7.1397	-5.35	-3.5603		<0.001
HF50 vs. WB	-0.6397	1.15	2.9397		0.24

Table A16: Two-way ANOVA results for differences in carbon flows to the bio-oil fraction, with changes in wood addition (HF, HF50, WB) and isothermal retention time (0.5, 2 h) (**bold** for significant differences p<0.05).

ANOVA table for bio-oil carbon flows (1)					
Effect of wood addition & isothermal retention time					
	SS	df	MS	F	p-Value
Retention time	450	1	450	346.6	<0.001
Feedstock type	196	2	98	75.48	<0.001
Interaction(time:type)	84	2	42	32.35	<0.001
Error	15.58	12	1.2983		
Total	745.58	17			
Multiple comparison test: Feedstock type (HF, HF50, WB)					
	Lower 95% CI	Mean difference	Upper 95% CI		p-Value
HF vs. HF50	1.2449	3	4.7551		0.017
HF vs. WB	6.2449	8	9.7551		<0.001
HF50 vs. WB	3.2449	5	6.7551		<0.001

Table A17: Two-way ANOVA results for differences in carbon flows to the bio-oil fraction, with changes in wood addition (HF, HF50, WB) and N₂ gas flow rate (0.5, 1.5 L/min) (**bold** for significant differences p<0.05).

ANOVA table for bio-oil carbon flows (2)					
Effect of wood addition & N₂ gas flow rate					
	SS	df	MS	F	p-Value
N ₂ gas flow	722	1	722	613.6	<0.001
Feedstock type	57	2	28.5	24.22	<0.001
Interaction (gas:type)	13	2	6.5	5.52	0.0199
Error	14.12	12	5.52		
Total	806.12	17			
Multiple comparison test: Feedstock type (HF, HF50, WB)					
	Lower 95% CI	Mean difference	Upper 95% CI		p-Value
HF vs. HF50	-2.1708	-0.5	1.1708		0.7110
HF vs. WB	1.8292	3.5	5.1708		<0.001
HF50 vs. WB	2.3292	4	5.6708		<0.001

Table A18: Two-way ANOVA results for differences in carbon flows to the non-condensable gases (NCG) fraction, with changes in wood addition (HF, HF50, WB) and isothermal retention time (0.5, 2 h) (**bold** for significant differences p<0.05).

ANOVA table for NCG carbon flows (1)					
Effect of wood addition & isothermal retention time					
	SS	df	MS	F	p-Value
Retention time	612.5	1	612.5	136.67	<0.001
Feedstock type	124	2	62	13.83	<0.001
Interaction(time:type)	76	2	38	8.48	0.0051
Error	53.78	12	4.482		
Total	866.28	17			
Multiple comparison test: Feedstock type (HF, HF50, WB)					
	Lower 95% CI	Mean difference	Upper 95% CI		p-Value
HF vs. HF50	2.7392	6	9.2608		0.001
HF vs. WB	-2.2608	1	4.2608		0.6993
HF50 vs. WB	-8.2608	-5	-1.7392		0.0039

Table A19: Two-way ANOVA results for differences in carbon flows to the non-condensable gases (NCG) fraction, with changes in wood addition (HF, HF50, WB) and N₂ gas flow rate (0.5, 1.5 L/min) (**bold** for significant differences p<0.05).

ANOVA table for NCG carbon flows (2)					
Effect of wood addition & N ₂ gas flow rate					
	SS	df	MS	F	p-Value
N ₂ gas flow	1058	1	1058	233.47	<0.001
Feedstock type	163	2	81.5	17.98	<0.001
Interaction(gas:type)	49	2	24.5	5.41	0.0212
Error	54.38	12			
Total	1324.38	17			
Multiple comparison test: Feedstock type (HF, HF50, WB)					
	Lower 95% CI	Mean difference	Upper 95% CI		p-Value
HF vs. HF50	3.7211	7	10.2789		<0.001
HF vs. WB	-1.7789	1.5	4.7789		0.4642
HF50 vs. WB	-8.7789	-5.5	-2.2211		0.002

Table A20: p-values of Mann-Whitney U hypothesis testing for differences in faecal sludge char nutrient content, for different years of sampling (2022, 2023) and compared to pure human excreta samples (SSF = source-separated faeces and MUF = mixed urine and faeces) (**bold** for significant differences p<0.05).

	N	P	K	Ca	Mg
2022 vs. 2023	0.4719	0.8182	0.1255	0.0411	0.9740
2022 vs. SSF	0.0022	0.0043	<0.001	0.0440	0.0649
2022 vs. MUF	0.0022	0.0022	<0.001	<0.001	0.5606
2023 vs. SSF	<0.001	0.0260	<0.001	0.0649	0.0649
2023 vs. MUF	<0.001	0.0022	<0.001	0.0022	0.7922

Table A21: p-values of Mann-Whitney U hypothesis testing for differences in faecal sludge char heavy metal content, for different years of sampling (2022, 2023) and compared to pure human excreta samples (SSF = source-separated faeces and MUF = mixed urine and faeces) (**bold** for significant differences p<0.05).

	Zn	Cu	Cr	Pb
2022 vs. 2023	0.0093	0.0023	0.0108	0.0022
2022 vs. SSF	<0.001	<0.001	<0.05*	<0.05*
2022 vs. MUF	<0.001	<0.001	<0.05*	<0.05*
2023 vs. SSF	0.0111	0.7944	<0.05*	<0.05*
2023 vs. MUF	<0.001	0.0389	<0.05*	<0.05*

*values for SSF and MUF were below the detection limit and are therefore considered statistically different to detected values.

Table A22: p-values of one-way ANOVA for differences in char stability (H/C ratio, R₅₀ recalcitrance index, H₂O₂ oxidation) and carbon content (stable fraction) with changes in (homogenised) feedstock types and blending ratios (faecal sludge [FS], bagasse [BG] and FS:BG 50:50, 40:60 blending ratios) (**bold** for significant differences p<0.05).

	H/C	R ₅₀	H ₂ O ₂	C _{STABLE}
FS vs. BG char	< 0.001	0.1249	< 0.001	< 0.001
FS vs. FS:BG 50:50	0.0016	0.3911	< 0.001	< 0.001
FS vs. FS:BG 40:60	< 0.001	0.1249	< 0.001	< 0.001
BG vs. FS:BG 50:50	0.2427	0.3574	0.0637	< 0.001
BG vs. FS:BG 40:60	0.3911	0.9240	0.1647	< 0.001
FS:BG 50:50 vs. FS:BG 40:60	0.4818	0.3574	0.3990	0.0362

Table A23: p-values of one-way ANOVA for differences in char characteristics (proximate analysis, CHNS and higher heating value (HHV) analysis) for different types of (homogenised) feedstocks (faecal sludge [FS], bagasse [BG], sawdust [SD], charcoal dust [CD]) (**bold** for significant differences p<0.05).

	MC	VM	FC	ASH	C	H	N	S	HHV
FS vs. BG char	0.4368	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001	0.0011	0.1064	< 0.001
FS vs. SD char	0.4136	0.0017	< 0.001	< 0.001	< 0.001	0.0012	< 0.001	0.2431	0.0010
FS vs CD char	0.2694	0.9783	< 0.001	< 0.001	< 0.001	0.0618	0.0184	0.1086	< 0.001
BG vs. SD char	0.1222	0.2237	< 0.001	< 0.001	0.3384	0.0163	0.1786	0.0137	0.1200
BG vs. CD char	0.0724	< 0.001	< 0.001	0.6112	0.0723	0.0531	0.0046	0.9408	0.1840
SD vs. CD char	0.7262	< 0.001	< 0.001	< 0.001	0.0246	0.0082	0.0030	0.0297	0.0478

MC = Moisture content, VM = Volatile matter, FC = Fixed carbon, HHV = Higher heating value

Appendix G. The Dubinin-Radushkevich adsorption model¹³

The Dubinin-Radushkevich (D-R) model was originally adopted for gas adsorption and has been extended to be applied for adsorption from aqueous solutions (Hu & Zhang, 2019; Worch, 2021):

$$q_e = q_m \exp \left[- \left(\frac{R T \ln \frac{c_{sat}}{c_e}}{E_c} \right)^2 \right] \quad (\text{Eq. A. 1})$$

where q_e is the amount of adsorbate adsorbed at equilibrium (mg/g / mol/g); c_e (mg/L / mol/L) is the adsorbate concentration in the solution after equilibrium is reached; and c_{sat} (mg/L / mol/L) is the saturation concentration (solubility) of the adsorbate (here estimated as 372,000 mg/L for NH_4Cl [value for 20°C]). The gas constant R was considered 8.314 J/mol/K and the temperature T at the time of the experiment was 294.65 K. The D-R isotherm parameters are q_m (mg/g / mol/g) which is the theoretical maximum monolayer adsorption capacity; and E_c (kJ/mol) is the adsorption energy.

Here, the model is applied as supplementary information to the data presented in **Chapter 6**. The D-R model was only applied for the synthetic NH_4Cl solution (parameters shown in **Table A24**) and not for real urine, as it would not be safe to assume that the solubility of a single adsorbate can characterise a complex solution such as human urine, where multiple NH_4^+ complexes likely participate in the process (Worch, 2021). Previous studies on NH_4^+ adsorption using the D-R model have used a modification of **Eq. A.1** assuming $[c_{sat}/c_e = 1 + 1/c_e]$, however this has been criticised as being mathematically incorrect (equation units are not balanced) and leading to a miscalculation of E_c , thus potentially misinterpreting the adsorption mechanisms taking place (Mahanty et al., 2023). For the E_c values, it has been reported that physical adsorption is the dominant mechanism when $E_c < 8$ kJ/mol, while $8 < E_c < 16$ kJ/mol correspond to ion exchange processes (Chabani et al., 2006; Hu & Zhang, 2019). Here, the model showed a good fit for the synthetic NH_4Cl solution ($R^2 = 0.996$), but the theoretical adsorption capacity (32.96 mg $\text{NH}_4\text{-N/g}$) was a severe overestimation of the measured actual value (23 mg $\text{NH}_4\text{-N /g}$). Interestingly, the value for the adsorption energy falls within the range identified for ion exchange processes, supporting findings from XRD and ICP-OES analysis that suggest an exchange occurs between K^+ and NH_4^+ ions from the biochar and urine respectively.

Table A24: Parameters of the Dubinin-Radushkevich isotherm model for $\text{NH}_4\text{-N}$ adsorption from a synthetic NH_4Cl solution onto faecal-derived biochar (25 g/L biochar dose) (based on data presented in **Chapter 6**).

Dubinin-Radushkevich			
	q_m (mg/g)	E_c (kJ/mol)	R^2
NH_4Cl synthetic solution	32.96	15.39	0.996

¹³ Supplementary information for the data presented in **Chapter 6**.