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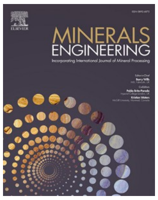

A review on strategies to assess the spatiotemporal heterogeneity of column leaching experiments for heap leaching upscaling

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ABSTRACT

Column leaching experiments are an essential step in the upscaling process towards a heap leaching operation. However, these experiments are often restricted to the optimisation of a reduced set of parameters. In doing so, most column leaching experiments tend to be ore-specific and to neglect key aspects such as mineralogical or structural changes over time, surface-level processes, or hydrodynamic features. The omission of these factors results in the loss of information on the spatiotemporal heterogeneity of column leaching experiments, which could provide insights leading to more environmentally or economically sustainable operations. Certain studies have focused on accessing this spatiotemporal information by using 3D imaging techniques (such as Computed Tomography – CT or Magnetic Resonance Imaging – MRI) or hydrodynamic assessment procedures (residence time distribution – RTD analysis or gravimetric liquid holdup estimation methods) to study column leaching. These studies have demonstrated that there is more information in column leaching experiments than what is acquired during conventional optimisation tests. Despite this, two essential gaps have been identified in the literature: (1) surface-level processes have received very limited attention as part of column leaching experiments and (2) chemical characterisation of the leaching solution is restricted to collection points before and after the column, providing a very low spatial resolution for these measurements. Multimodal methodologies capable of assessing several aspects of the process need to be implemented to bridge these gaps and to unlock information on the spatiotemporal heterogeneity of column leaching experiments.

1. Introduction

Heap leaching is estimated to produce 21% and 9% of global copper and gold, respectively (Marsden and Botz, 2017). While this process is not responsible for most of the global production of these metals, it still accounts for a sizeable fraction of it. Notably, heap leaching operations can treat low-grade ores successfully and economically (Petersen, 2016; Thenepalli et al., 2019). This is relevant as the depletion of high-grade ore deposits worldwide is shifting the focus towards low-grade deposits (Mitra, 2019; Rötzer and Schmidt, 2018).

Heap leaching can also be used to reprocess tailings from other processes like froth flotation. However, a major drawback of this process is the slow rate at which leaching reactions occur. Although it depends on the type and grade of the ore, a single heap is typically run for at least a month and the operation could continue for over three years (Natarajan, 2018; Petersen, 2016; Thenepalli et al., 2019). The combination of slow kinetics and the use of low-grade ore makes the economic viability of this process strongly dependent on the large scale at which the heaps must be run. Due to the large volume of material required to operate a heap, a direct analysis of these leaching processes is a challenging task. Therefore, it is commonplace to perform lab-scale or pilot-scale column leaching experiments to acquire knowledge and enhance the process before building and operating a heap (Van Staden and Petersen, 2021; Winarko et al., 2023).

Column leaching experiments involve packing ore in cylindrical columns of varying diameters and heights. This setting allows for a feed leach solution to be added dropwise from the top of the column, so that it can percolate through the ore within the column and be recovered from the bottom of the system. The leaching solution reacts with the packed ore as it moves downwards, extracting various species. In this way, column leaching resembles heap leaching closely. The main differences between them will be the scale at which the two processes are operated and the possibility of wall effects (preferential flow next to the column walls) occurring if the column is too narrow (Dobson et al., 2017; Fagan, 2013; Ilankoon and Neethling, 2019). Despite these differences, columns are consistently chosen as tools to understand and

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optimise leaching processes before scaling up to heaps due to the convenience and relative low cost of their set-up. It should be noted that lab-scale stirred tank leaching experiments are typically performed before column leaching. These experiments allow for the rapid assessment of the amenability of the ore to leaching, as well as giving an indication of the kinetics of the process (Faraji et al., 2022).

Most studies involving column leaching focus on optimising various parameters experimentally to achieve a higher (Azmayandeh et al., 2017; Moldoveanu and Papangelakis, 2016) or faster (Ai et al., 2019; Ghorbani and Montenegro, 2016; Hernández et al., 2019; Lu et al., 2017) extraction of the target metal, to increase selectivity (Agatzini-Leonardou and Zafiratos, 2004; Williamson et al., 2021), or to decrease the consumption of the leaching agent of choice (Komnitsas et al., 2023, 2019, 2018; Ntakamutshi et al., 2017). Other studies aim to optimise column leaching by obtaining mathematical models that relate leaching efficiency to operating parameters (Flores et al., 2020; Flores and Leiva, 2021; Hoseinian et al., 2017; Leiva et al., 2017; Long et al., 2020). While optimisation studies can provide useful information leading to more efficient leaching processes, they are typically ore-specific and cannot always lead to conclusions that are applicable to other leaching operations.

Many other studies have been centred around comprehending in detail how leaching processes occur in columns. Such studies have aimed to expand the current understanding of general aspects such as permeability (Ai et al., 2022; Lin et al., 2005) or fluid flow within columns (Fagan et al., 2013; Ilankoon and Neethling, 2019, 2016; Odidi et al., 2023). These studies are usually not ore-specific and can provide tools to identify and deal with inefficiencies in the future.

The stationary nature of the solid particles in column leaching causes these systems to exhibit both spatial and temporal heterogeneity. As the solution percolates towards the bottom of the column, its properties and the concentration of leaching agents and other species are expected to change. Due to varying solution conditions, the ores will also respond differently depending on their position within the column. Even though axial changes are mostly responsible for the spatial heterogeneity because of the direction of the flow, wall effects (Ilankoon and Neethling, 2019), and preferential channel formation (Bouffard and Dixon, 2001; Lin et al., 2016a) make radial changes relevant as well. Thus, physical and chemical changes in both the ore and the solution will be a function of time and position.

Understanding the inherent spatiotemporal heterogeneity of column leaching systems is necessary to gain insights into how the process will behave after scale-up to heap leaching. Analysis of the solid residue of the column is typically only performed after the experiments have finished, only providing spatial information *post-mortem* but not throughout the leaching period. Likewise, column characteristics often restrict obtaining temporal information from the solutions at locations other than the outlet of the system, disregarding where or how the changes in the solution are taking place. Therefore, leaching columns typically operate as temporal black boxes from the perspective of the solid particles and as spatial black boxes from the perspective of the leach solution.

Numerous reviews covering a broad array of topics have been published within the field of leaching. These reviews have addressed both general issues that apply to most leaching processes and others which are specific to certain ore-leaching agent systems. Many of these reviews have focused on leaching kinetics and mechanisms (Faraji et al., 2022; Li et al., 2013; Senanayake, 2006), while others have discussed strategies to increase the leaching efficiency of specific systems (Córdoba et al., 2008a, 2008b; Li and Miller, 2006; Muir, 2011; Ng et al., 2020; Owusu-Fordjour and Yang, 2023; Sun et al., 2020; Wang et al., 2021b). In terms of scale of operation, earlier reviews have focused on scale-up studies leading to enhanced heap leaching operations, considering pilot scale testing in leaching columns (Ghorbani et al., 2016; Panda et al., 2015). However, to the authors' knowledge, no article reviewing strategies to unlock spatiotemporal insights from column leaching experiments has been published.

Some efforts have been placed on unlocking these spatiotemporal black boxes by modifying traditional column leaching tests or by incorporating novel techniques to the conventional column leaching experiment workflow. This review has two main objectives: (1) to discuss the techniques, strategies, or methods developed for assessing the spatiotemporal heterogeneity of column leaching systems, and (2) to assess other techniques that have not yet been applied to column leaching experiments but that could have a potential impact if applied in this field in the future.

This review begins with a structured overview of the techniques and methods that are relevant to studying column leaching experiments. Three sections that cover the study of the spatiotemporal heterogeneity of solid particles follow: structural assessment, mineralogical quantification, and surface-level analysis. The review then focuses on the study of the spatiotemporal heterogeneity of the leaching solution, which is subdivided into liquid holdup and distribution assessments, and the application of 3D imaging techniques to investigate fluid flow. Finally, the review closes with a perspective section that discusses the main findings of the study and outlines the identified knowledge gaps that can be addressed in future research.

2. Overview of techniques and methods

This section introduces the main techniques and methods that have been used by researchers to unlock spatiotemporal insights into column leaching experiments or that have been used in other leaching set-ups but could be applied to column leaching. The descriptions presented here aim to briefly introduce the reader to the techniques whose specific applications will be detailed in the next sections. Fig. 1 illustrates five key aspects that govern column leaching performance: structural features, surface-level characteristics, mineralogy, hydrodynamics, and solution properties. This review will focus on techniques to study the first four aspects. These aspects are essential for a full understanding of the spatiotemporal changes that occur during column leaching. As a result, if a study's aim is to understand the process, they should all be considered when designing column leaching experiments. However, some of these aspects may not be relevant as part of optimisation or upscaling experiments. The fifth aspect is not discussed further in this review as it usually relies on measurements that, while essential, have a limited contribution to understanding the spatiotemporal heterogeneity of column leaching experiments.

It should be noted that some of these aspects may be relevant to experiments associated with other processes that involve liquid flow through packed beds, such as heterogeneous catalysis in packed bed reactors (Das et al., 2022) or the use of permeable reactive barriers to remove ions from mine effluents (Bartzas and Komnitsas, 2010; Komnitsas et al., 2006) or contaminated groundwater (Abedin et al., 2011; Sakr et al., 2023). As such, the techniques presented in this review may be useful for the assessment of processes other than leaching.

2.1. Surface-level assessment techniques

Although leaching occurs at a volumetric level, this process is heavily dependent on the characteristics of ore surfaces: the first point of contact between the solution and the ore occurs at the surface. Assessing the surface of the ore both before and during leaching is thus essential to identify factors that could be favouring or hindering the process. Surface-level phenomena are commonly studied as part of batch leaching experiments, but they are rarely the focus of column leaching experiments.

Surface processes during batch leaching experiments have been followed in the past using Scanning Electron Microscopy with Energy Dispersive Spectroscopy (SEM-EDS), X-ray Photoelectron Spectroscopy (XPS), and Time-of-Flight Secondary Ion Mass Spectrometry (ToF-SIMS). However, to the authors' knowledge, the latter two techniques

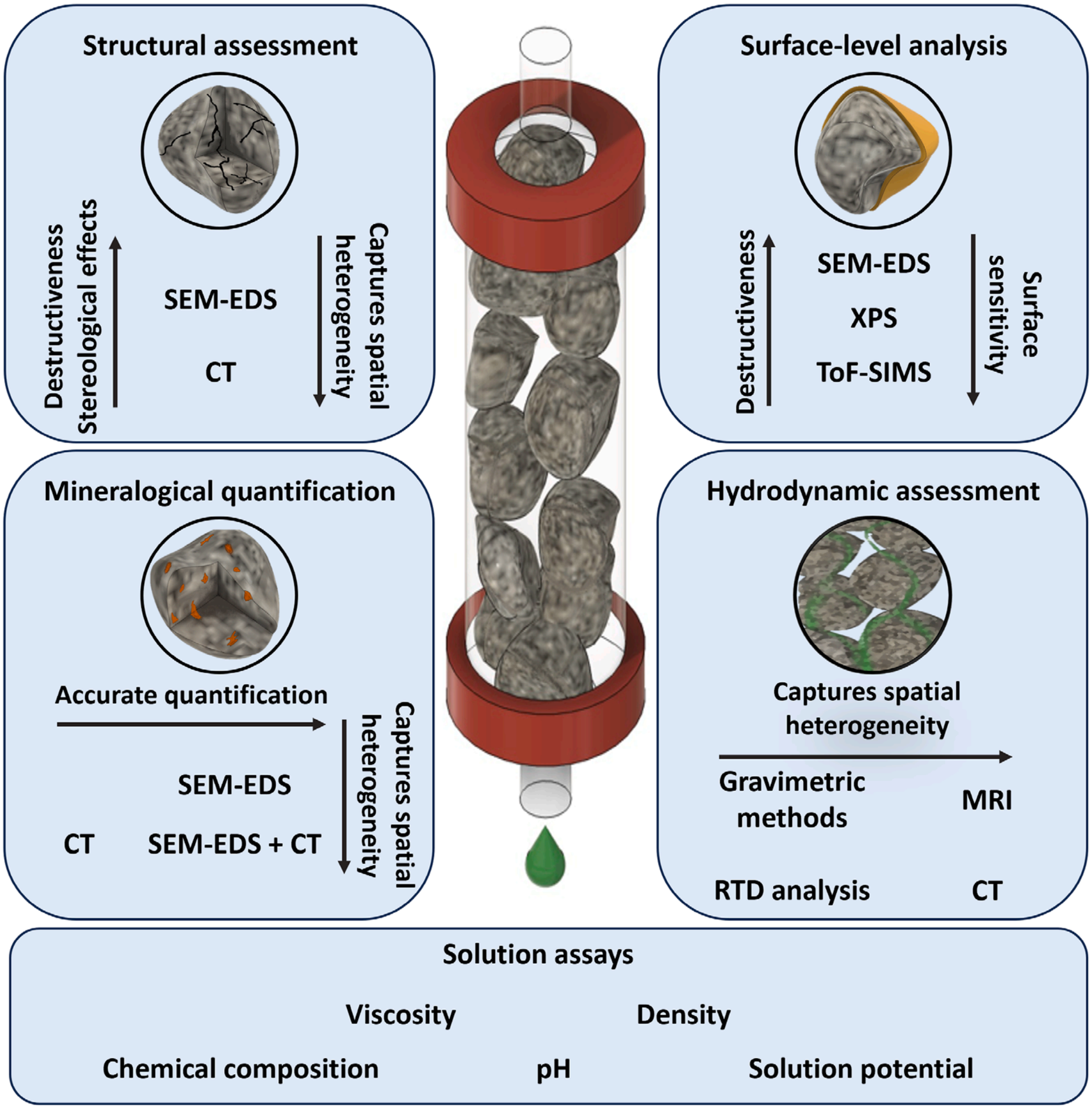

Fig. 1. Schematic overview of the solid particle and solution assessment techniques and methods that have been reviewed as tools for accessing spatiotemporal insights into column leaching experiments. The dimensions of the column and the size of the particles shown in the figure are only for illustrative purposes.

have not been applied to column leaching set-ups yet, although they have all been applied to other leaching set-ups. The following subsections will describe and compare these tools as they all have the potential to provide insights leading to a more thorough understanding of the spatiotemporal heterogeneity of surface processes during leaching. A summary of the comparison of these techniques is presented in Fig. 2. The indices used for the comparison presented in Fig. 2 are explained in detail in Table 1. The references that support this comparison are detailed in subsections 2.1.1, 2.1.2, and 2.1.3.

2.1.1. Scanning electron microscopy with energy dispersive spectroscopy (SEM-EDS)

SEM-EDS has been extensively utilised as part of leaching experiments to acquire micrographs of solid particle samples (SEM) and their associated elemental maps (EDS). SEM images are obtained by detecting secondary electrons (SE) or backscattered electrons (BSE) that are emitted by the sample when irradiated with a primary electron beam. SE are used preferentially when the textural characteristics of the samples are relevant, while BSE are more useful to distinguish between phases. The depth from which the technique can extract information varies greatly and depends on the selected settings and in the type of electrons that are being detected: SE detection is typically limited to the first few nanometres (<10 nm), while BSE can provide information from depths in the order of hundreds to thousands of nanometres (Goldstein et al., 2018). Depth profiling is possible using focused ion beam technology (FIB-SEM), which allows generating 3D maps of the sample by performing multiple 2D scans at different depths by removing the outermost sample layers. It should be noted that FIB-SEM is destructive (Gu et al., 2020; Xu et al., 2017). SEM-EDS can also provide mineralogical quantification if coupled with mineral mapping software (e.g., Mineral Liberation Analysis – MLA, Quantitative Evaluation of Minerals by Scanning Electron Microscopy – QEMSCAN, and TESCAN Integrated Mineral Analyzer – TIMA, among others) (Warlo et al., 2019).

Despite the usefulness of this technique, its full potential has not been applied extensively in column leaching studies. With the exception of one study that employed this technique to follow changes in the surface of a sample (Van der Meer et al., 2009), the use of SEM-EDS in column leaching has been limited to characterising the solids before and after leaching (Ai et al., 2022; Komnitsas et al., 2019; Phyo et al., 2020), thus not yielding insights into the temporal heterogeneity of the particles. One of the limitations of this technique is that its accuracy is higher when the surface to be studied is flat, for which grinding and polishing

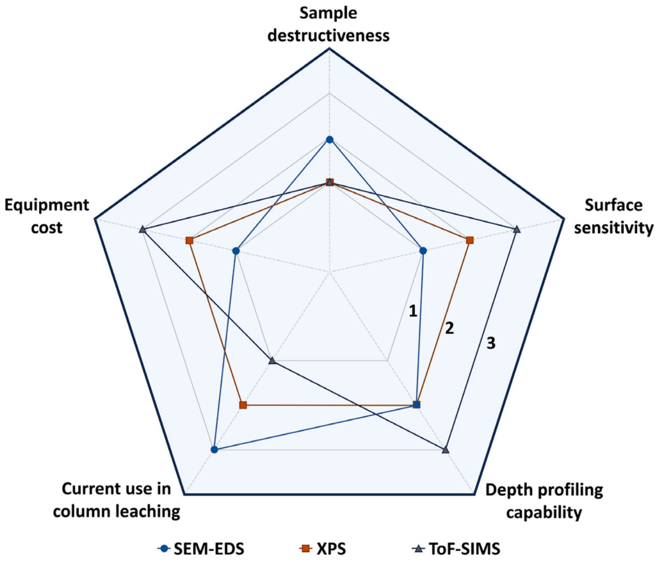

Fig. 2. Comparison of the surface-level techniques that are considered in this review as tools or potential tools to unlock spatiotemporal insights in column leaching experiments.

may be needed (Kjellsen et al., 2003). Moreover, sputter coating the surface of the sample may be necessary if the sample is non-conducting (Echlin, 2009), which increases the sample destructiveness of the technique.

A major advantage of SEM-EDS over other surface-sensitive techniques is that it is already a conventional tool within the hydrometallurgical industry. This implies that adapting its use to follow surface-level processes would be simpler and less expensive than acquiring the equipment and expertise required to use other surface-sensitive techniques. While prices may vary depending on their characteristics and capabilities (Wilkinson et al., 2011), they are usually in the range between US$ 50,000 and US$ 500,000 according to various suppliers (the prices depend greatly on whether it is a tabletop or a floor model instrument). This makes SEM-EDS instruments typically less expensive than other instruments or techniques such as XPS (US$ 500,000–1,000,000) or ToF-SIMS (US$ ~1,000,000) (Cushman et al., 2015; Grant, 2023). Additional information about SEM-EDS, its applications, and limitations can be found in the literature (Goldstein et al., 2018; Ul-Hamid, 2018; Zhou and Wang, 2007).

2.1.2. *X-ray Photoelectron Spectroscopy (XPS)*

XPS is a surface-sensitive analytical technique capable of providing chemical information about the first few nanometres (<10 nm) of a sample (Krishna and Philip, 2022). This technique can quantify the elemental composition of most of the elements present on the surface of a sample. In addition to this, it can distinguish specific oxidation states for each target element (Descostes et al., 2000; Fantauzzi et al., 2015). XPS is a tool capable of following surface processes: it can detect changes in composition or in the chemical state of samples. Since these processes are essential during leaching, XPS can provide insights into them that could not be obtained from other techniques like SEM-EDS or chemical assays, just to name a few (Abdel-Samad and Watson, 1998; Silva-Quiñones et al., 2018).

While the technique itself is non-destructive, some samples may require initial preparation to provide flat surfaces for analysis (Silva-Quiñones et al., 2018), which may result in a partial loss of the non-destructive nature of XPS. Depth profiling is also possible with XPS through argon sputter etching of the surface between measurements. However, surface etching is destructive (Greczynski and Hultman, 2021; Oswald et al., 2020). The reader is referred to the literature for more information on the fundamentals of the technique, the interpretation of XPS data and the various applications of the technique (Moulder et al., 1992; Suga et al., 2021).

2.1.3. *Time-of-Flight secondary ion mass spectrometry (ToF-SIMS)*

ToF-SIMS is a highly surface-sensitive mass spectrometry technique that can be applied to assessing leaching processes (Ren et al., 2023, 2022; Zuo et al., 2023), as it is limited to the analysis of the topmost 2–3 atom layers of samples. This means that ToF-SIMS offers a finer surface-depth resolution than XPS or SEM-EDS. ToF-SIMS excels at quantifying the elemental or molecular composition of surface species (Fearn, 2015; Rinnen et al., 2015). As a result, this technique enables tracking surface-level transformations, thus supplying insights into surface alterations in composition due to the progress of leaching reactions. Surface sputtering is frequently combined with ToF-SIMS analysis to create depth profiles, as the sputtering enables accessing deeper layers within the sample (Fearn, 2015). However, sputtering is destructive as the original surfaces are removed during the process. As in the case of SEM-EDS or XPS, sample preparation may be necessary in some cases, increasing the sample destructiveness of ToF-SIMS. Further information on the fundamentals, limitations, experimental methodologies, and applications of this technique is available in the literature (Vickerman and Briggs, 2013).

2.2. *3D imaging techniques*

3D imaging techniques have been used in the context of leaching experiments to assess fluid flow within columns or changes in the particles at a volumetric scale. The non-destructive nature of these techniques allows extracting information from the column without interfering with the ongoing leaching process or disrupting the packed ore bed, thus enabling tracking changes or processes over time. The most common 3D imaging techniques used by researchers in the field are X-ray Computed Tomography (CT) (Lin et al., 2016a; Phyo et al., 2020; Reyes et al., 2017; Salinas-Farran et al., 2022) and Magnetic Resonance Imaging (MRI) (Fagan et al., 2013; Videla et al., 2017; Yu and Gao, 2020).

Both techniques can produce 3D reconstructions of specific volumes of interest by combining acquired sets of 2D images, which can then be

Table 1
Comparison metrics for surface-sensitive techniques: SEM-EDS, XPS, and ToF-SIMS.

Index	1	2	3
Sample destructiveness	Surface grinding during sample preparation	Surface grinding and sputter coating during sample preparation	Fully destructive (sample is digested or significantly changed during or before analysis)
Surface sensitivity	100–1000 nm surface sensitivity	<10 nm surface sensitivity	2–3 atoms surface sensitivity
Depth profiling capability	Cannot perform depth profiling	Can be applied to depth profiling	Dedicated to depth profiling
Current use in column leaching	Rarely used in any leaching set-ups	Frequently used in some leaching set-ups	Has been used in column leaching set-ups
Equipment cost	< $500,000	$500,000 – $1,000,000	> $1,000,000

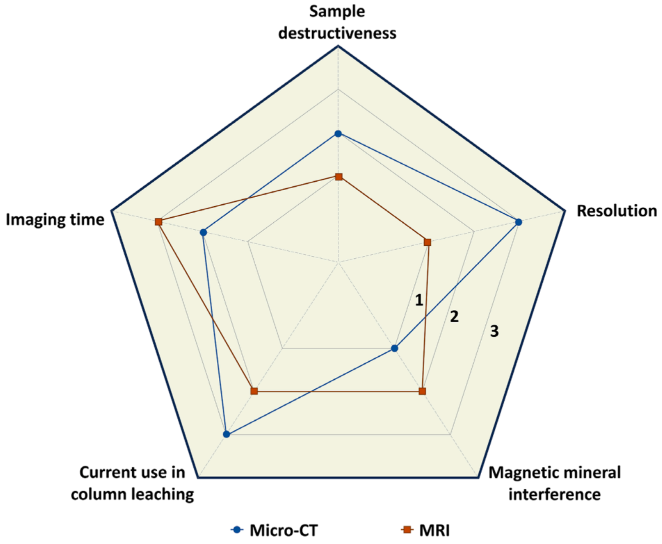

Fig. 3. Comparison of the 3D imaging techniques that are considered in this review as tools to access spatiotemporal insights in column leaching experiments. Micro-CT was selected for the comparison as it is the most widely used CT scale in column leaching applications. The information for both micro-CT and MRI are based on the column leaching applications found in the literature.

processed for different purposes. Sections 2.2.1 and 2.2.2 will describe and compare CT and MRI. Fig. 3 summarises the comparison between the two techniques. The indices used to compare the 3D imaging techniques are explained in detail in Table 2. The references that were used to establish the metrics for the comparison are detailed in subsections 2.2.1 and 2.2.2.

2.2.1. *Computed tomography (CT)*

CT has received attention as a tool to assess heap and column leaching performance over the last two decades. Studies have used this technique to analyse factors that can affect leaching processes, including the modal mineralogy of the samples (when CT is coupled with 2D mineral maps produced with other techniques), the exposure of minerals of interest, and the size distribution of particles or grains (Ghadiri et al., 2020; Miller et al., 2003b; Salinas-Farran et al., 2022). Moreover, CT studies can follow how these factors change over time due to leaching. CT can also be used to study aspects related to the packing of the ore within the column or to structurally characterise the particles (Lin et al., 2005; Phyo et al., 2020; Salinas-Farran et al., 2024). For instance, this technique can provide images of the pore network, leading to estimations on the porosity of the packed system and providing valuable information for fluid flow modelling purposes (Dhawan et al., 2012; Guntoro et al., 2019; Wang and Miller, 2020).

Depending on the spatial resolution of the CT scanner (i.e., the voxel size of the obtained image), the technique can be classified as milli-CT (millimetres), micro-CT (micrometres), or nano-CT (nanometres) (Wang and Miller, 2020). The resolution of the CT scanner can be a limitation as particles or grains that are smaller than the resolution will not be registered. The literature revealed that column leaching studies have favoured the micrometre scale, with most studies over the last decade using micro-CT achieving resolutions ranging between 10 μm and 50 μm, considering an imaging time of 50–120 min (Ai et al., 2022; Charikinya et al., 2015; Fagan-Endres et al., 2017; Ghadiri et al., 2020; Lin et al., 2016b; Miao et al., 2017; Reyes-Leiva, 2018; Salinas-Farran et al., 2022).

The selection of the correct equipment for a specific application is essential, as each scanner will have limitations in terms of the size of the samples that it can analyse (i.e., samples cannot be larger than the scanning chamber). Detailed information regarding the physics behind the technique, the fundamentals of image reconstruction and processing, and the applications of CT in various fields can be found in the literature (Orhan, 2020; Stock, 2019; Wang, 2022).

The authors acknowledge the recent development of modified micro-CT equipment and software for 3D image processing based on deep learning algorithms by various companies. As advertised on technical brochures, these techniques enable producing 3D mineral maps and assessing mineral liberation non-destructively. As such, they have the potential to be applied on future column leaching studies. To the authors' knowledge, independent scientific publications using these techniques are not yet available in the field, so they will not be described further in this review.

2.2.1.1. *Spectral Computed Tomography (sp-CT).*

One of the limitations of conventional CT is that this technique is unable to directly characterise the elemental or mineralogical composition of samples (Godinho et al., 2023). Recent studies have focused on targeting this issue by using sp-CT. In addition to counting the total amount of X-ray photons that are transmitted through the sample and reach the detector (conventional CT), sp-CT separates these photons into discrete bins based on their energy (Sittner et al., 2021). This allows quantifying how many photons are reaching the detector and how much energy each of them has. By doing this, a more accurate elemental composition can be obtained as each element preferentially attenuates X-rays of specific energies (K-absorption edge), which sp-CT can identify (Egan et al., 2017, 2015).

However, the technique currently suffers from two key limitations which may be addressed in the future: (1) it can only quantify heavy elements (silver and heavier elements) and (2) scanning times are significantly higher for sp-CT (days) than for CT (hours) (Godinho et al., 2021; Sittner et al., 2021). This technology will not be described in further detail in this review as very few studies within the minerals processing field have used it and it has not been applied to leaching experiments yet. However, sp-CT has a significant potential to revolutionise the 3D elemental characterisation of samples in minerals processing due to its advantages over 2D techniques such as SEM-EDS: sp-CT is non-destructive and has no stereological bias (Godinho et al., 2021). Additional information about the technique is available in the literature (Greffier et al., 2023; Taguchi et al., 2020).

Table 2
Comparison metrics for 3D imaging techniques: micro-CT and MRI.

Index	1	2	3
Sample destructiveness	Fully non-destructive (technique does not affect the sample)	May be destructive in specific cases (e.g., sterilisation of bioleaching columns)	Fully destructive (sample is digested or significantly changed during or before imaging)
Resolution	>800 μm	50–800 μm	<50 μm
Magnetic mineral interference	Does not occur	Can occur but is not prohibitive	Can occur and is prohibitive
Current use in column leaching	Rarely used in any leaching set-ups	Occasionally used in column leaching set-ups	Frequently used in column leaching set-ups
Imaging time	<1h	1–2 h	>2h

2.2.2. Magnetic resonance imaging (MRI)

MRI has been used less extensively than CT as a tool to obtain information from column leaching experiments. However, MRI is preferable to CT in some cases: for instance, MRI is recommended when the process under study involves a microbial population. This is because CT may sterilise bioleaching columns due to X-ray radiation, thus becoming a destructive technique in specific cases. Additionally, MRI can provide a clearer contrast between liquid and gaseous phases when high density solids are present, which can be useful to estimate solid–liquid and solid–gas interface areas (Fagan et al., 2013). Nonetheless, MRI has certain limitations when dealing with particles with a high content of paramagnetic and ferromagnetic materials, which can cause image distortions (Fagan et al., 2012; Li et al., 2018). However, these limitations are not prohibitive, and MRI can still be used to obtain information from particles with magnetic properties (Fagan et al., 2014, 2012).

As in the case of CT, the desired resolution and sample size must be considered when selecting the correct MRI equipment for a specific application. To the authors' knowledge, MRI-based studies involving leaching columns have been limited to resolutions between 0.8 mm and 2.5 mm, associated to acquisition periods of 3–13 h (Fagan-Endres et al., 2015; Fagan et al., 2014, 2013, 2012; Videla et al., 2017). Although prices may vary depending on each equipment's characteristics, MRI scanners are generally more expensive than CT scanners (Kim et al., 2022; Vásárhelyi et al., 2020), which constitutes a limitation for the widespread application of MRI in column leaching experiments. The reader is referred to the literature for additional information on the fundamentals of MRI, the processing of images obtained using this technique, and some of its applications (Akçakaya et al., 2022; Constantinides, 2014).

2.3. Liquid holdup and distribution assessment methods

Liquid holdup in column and heap leaching systems is an important hydrodynamic parameter capable of influencing leaching performance because it affects mass transfer processes occurring in these systems (de Andrade Lima, 2006; Ilankoon, 2012). Characterising liquid holdup and how the leaching solution distributes within packed bed systems is thus an important field of study, as it can lead to identifying hydrodynamic factors that may be negatively affecting the leaching process, such as the formation of preferential flow channels (Wang et al., 2021a).

Different approaches have been used to understand liquid holdup and distribution in the context of column leaching. In addition to using 3D imaging techniques (see section 2.2.2) to study solid–liquid interfaces (Fagan et al., 2013), certain researchers have performed gravimetric liquid holdup assessments (Ilankoon and Neethling, 2012; Wang et al., 2021a) and Residence Time Distribution (RTD) analyses (de Andrade Lima, 2006; Odidi et al., 2023) to gain insights into how the solution accumulates and is distributed inside the columns. The data obtained using these methods can then be used as an input for modelling liquid holdup and distribution in column or heap leaching systems (McBride et al., 2017). The main characteristics and capabilities of these two liquid holdup and distribution assessment methods are summarised in Table 3. Details on this information are provided in sections 2.3.1, 2.3.2, and 6.1.

Table 3
Comparison of the characteristics and capabilities of gravimetric liquid holdup assessment methods and RTD analyses as tools to study liquid holdup and distribution.

Characteristics	Gravimetric liquid holdup assessment	RTD analysis
Measured variable	Weight of system	Solution properties
Requires tracer addition	No	Yes
Types of operation it has been used for	Steady state and transient	Steady state
Can identify preferential flow paths	Yes	No
Can measure horizontal dispersion	No †	No
Can measure axial dispersion	No †	Yes
Time resolution of measurements	High	Low ‡
Complexity of set-up	Low	High

† Gravimetric assessment methods can measure axial and horizontal dispersion if they are adapted by including the use of visual tracers or by modifying the set-up (see section 6.1).

‡ The time resolution of column leaching studies with RTD analysis is typically low but can be increased by modifying the data processing procedure (see section 2.3.2).

2.3.1. Gravimetric liquid holdup assessment

Some efforts have been made to quantify the liquid holdup in column leaching systems by weighing the columns throughout the irrigation period. This assessment approach has been performed both during steady state (Ilankoon and Neethling, 2013) and transient operation periods (Ilankoon and Neethling, 2014), which provides a more complete understanding of the whole process, from the initial irrigation to the final stages of draining. Some of these studies have used a porous packed bed, while others have used non-porous materials, which allows decoupling interparticle and intraparticle liquid holdup and understanding hysteresis effects.

This type of study can help understand how liquid accumulates in the columns and can supply insights for process optimisation (Wang et al., 2021a) or for modelling this phenomenon (McBride et al., 2017; Mostaghimi et al., 2014a). Since gravimetric methods are based strictly on measuring the weight of column leaching systems, a significant amount of information is not captured by these methods. Due to this, some studies have used more complex set-ups to visually detect how liquid distributes in packed beds on top of quantifying the liquid holdup in these systems, which increases the understanding of the spatial heterogeneity of the experimental system (Ilankoon and Neethling, 2019, 2016).

2.3.2. Residence time distribution (RTD) analysis

Residence Time Distribution (RTD) analyses can be used to assess the hydrodynamics of column leaching by characterising the time-dependent response of these systems to controlled stimuli. To do so, the method requires dosing the feed solution with tracers whose concentrations can be measured (e.g., through pH or conductivity measurements) (Bouffard and West-Sells, 2009; de Andrade Lima, 2006; Fagan-Endres et al., 2023). By measuring the concentration of these tracers in the input and output streams of the column during a specific period, this method allows detecting how input changes can affect the output stream as a function of time. In addition to providing insights into liquid holdup and fluid dispersion in packed bed systems (Bouffard and Dixon, 2001; Saroha et al., 1998), this assessment method can be used to optimise leaching column operation by supplying key parameters for modelling and simulating hydrodynamic phenomena (Bouffard and West-Sells, 2009; de Andrade Lima, 2006). This could enable researchers to predict and improve the performance of leaching processes, addressing issues like preferential flow channel formation or poor lateral column wetting (Odidi et al., 2023).

One of the limitations of this analysis method is that any measurements will have a low time resolution: the effluent solution must accumulate over time to allow measuring tracer concentrations. The low volumetric flow rates characteristic of column leaching experiments exacerbate this issue, meaning that RTD analyses applied to column leaching experiments will not capture the temporal heterogeneity of the systems fully. However, it is possible to work around this issue by following a methodology proposed by Fagan-Endres et al. (2023). This methodology allows estimating true column output tracer concentrations as a function of measured tracer concentrations in output solutions that have accumulated over a known period. To achieve this, the transport, accumulation, and mixing of the output solution are modelled as a plug flow reactor in series with a continuous stirred tank reactor (Fagan-Endres et al., 2023).

2.4. *Other techniques with currently limited applications*

Numerous other techniques are part of the usual leaching toolkit and have been used as part of column leaching experiments. However, to the authors' knowledge, the techniques listed in this section have only been used to characterise samples before and after experimenting on them and not to follow changes during the leaching process. While these tools have potential to be used to unlock spatiotemporal insights, they have not been applied to fulfil that purpose yet. This situation is caused by limitations that are inherent (but not necessarily prohibitive) to the techniques or to the complexity associated to extracting and replacing solid samples from a column leaching experimental system. These tools include optical microscopy imaging techniques (including transmitted and reflected light microscopies, with or without polarised light) (Agatzini-Leonardou et al., 2021; Quezada et al., 2021), particle size distribution assessment methods (Chen et al., 2020), Mercury Intrusion Porosimetry (Hoummady et al., 2018; Laurent et al., 2019), X-ray Diffraction analysis (Rodrigues et al., 2018; Shi et al., 2022), and X-ray Fluorescence analysis (Ghorbani and Montenegro, 2016; Komnitsas et al., 2019).

3. Structural assessment

As detailed in the following two subsections, the structural assessment of particles and packed beds in studies focusing on column leaching or on stages leading to column leaching has received a significant amount of attention over the last two decades. This is because the macrostructure and microstructure of these particulate systems can often determine the kinetics governing solution transport and the extent to which specific grains can be accessed by the solution. This section will describe the application of techniques to assess the spatiotemporal heterogeneity of the physical structure of packed beds. Most of the reviewed studies have used CT to achieve this task, but some studies have relied on complementary methods, such as using SEM or strategies to estimate the permeability of the packed beds. Likewise, some studies have focused on using experimentally collected structural information to validate models describing phenomena that are characteristic of column curing and leaching processes.

3.1. *Structural assessment during column leaching*

Several efforts have been placed on assessing the microstructure (porous network) and macrostructure (column packing) of particles in leaching columns and how these aspects change during the process. To do so, researchers have used CT, due to its non-destructive nature and to its ability to produce 3D images of the packed beds. However, a common limitation in these studies is that the resolution of the instrument will impact any assessment of the microstructure of particles: any feature smaller than the instrument's resolution cannot be quantified appropriately (Guntoro et al., 2019; Miao et al., 2017).

The study by Lin et al. (2005) was the first to use CT imaging to obtain a 3D reconstruction of a leaching column before and after bioleaching copper-containing agglomerates. The authors used a milli-CT to obtain information on the microstructure and on the pore network of the packed column. The porosity of the packed bed was followed spatially (at different heights) and temporally (before and after leaching). It was found that porosity is lower in the bottom of the column, due to loading effects. Likewise, it was found that overall porosity decreases significantly during leaching because of fine particle migration towards the bottom of the column and compaction (Lin et al., 2005). These observations contribute to the spatiotemporal understanding of the leaching process and may help troubleshoot solution flow issues that may arise during heap leaching operation.

Yang et al. (2014) employed a medical CT scanner to study the porous network of nine different copper sulphide ores, each of which was separated in narrow size classes. These classes were used to pack columns sequentially, going from finer to coarser particles from the bottom to the top of the column. Particle segmentation was performed to distinguish solids from air (in pores or between particles). This generated a 3D binary volume, which allowed quantifying the pore size distribution, the fraction of the total volume occupied by pores, and the pore connectivity degree of each sample. While this study did not use CT on a running column leaching experiment, it proved that this technique can be used to study the characteristics of porous networks and, in particular, that it can be used to assess pore connectivity, although the resolution used for the scans did not allow the analysis of features under 180 μm in diameter (Yang et al., 2014). Quantifying this aspect is essential for column and heap leaching as an interconnected porous network will facilitate the transport of the leaching solution towards target grains.

Taking advantage of its non-destructive nature, micro-CT has also been used to follow structural changes during column leaching of copper ore. The goal of these studies is to obtain visual representations of the pore structure of the ores and how they can change throughout the leaching process (Ai et al., 2022; Phyo et al., 2020; Salinas-Farran et al., 2024). This type of assessment is relevant as leaching induces physical changes in the pore structure over time. Leaching can open or close pore channels, thus altering the solution's ability to reach certain grains. Phyo et al. (2020) assessed pore network changes during leaching of a chalcocite ore by using micro-CT. This study showed that leaching could generate cracks and modify the porous structure of the ore particles. These results (which may be ore-specific) imply that particle breakage occurs during column leaching: the particle structure changes from compact to porous and fractured (Phyo et al., 2020).

Salinas-Farran et al. (2024) investigated changes in the porosity of chalcopyrite-containing agglomerates that were processed through column leaching. The study identified a decrease in the interparticle porosity of the agglomerates over the leaching period for the two different leaching feed solutions that were tested. It was shown that the changes in the measured porosity did not impact the copper leaching extraction. However, the authors stated that this observation was limited by the resolution (~17 μm) of the micro-CT images used in the study. They suggested that variations in microporosity may influence the leaching performance (Salinas-Farran et al., 2024).

Ai et al. (2022) evaluated the effect of adding a surfactant (sodium dodecyl sulphate) during leaching on the pore structure of particles located at different axial positions within the column. To do so, they ran two different column leaching experiments (with and without addition of surfactant) and each column was divided into five different sections based on their vertical positions. Micro-CT results suggested that leaching increases the average porosity in the upper sections of the columns during leaching, while it had the opposite effect in the lower sections. This general behaviour was seen with or without surfactant addition. Nevertheless, the presence of the surfactant accentuated the changes, particularly in the uppermost sections of the column. This micro-CT assessment was coupled with using a rig to study the permeability coefficient in each of the leaching columns. This rig allowed determining how long it took for the inlet solution to percolate through the column. The permeability coefficient was estimated from the combination of this information with some of the columns' geometric parameters. Regardless of the selected leach solution, the permeability coefficient decreased during leaching in the bottom of the column, while rising in the uppermost sections of the column. However, higher permeability coefficients after leaching were reported for the case in which surfactant was added (Ai et al., 2022).

The studies undertaken by Phyo et al. (2020), Salinas-Farran et al. (2024), and Ai et al. (2022) demonstrate that micro-CT can be used to track structural changes over time *in situ*. Furthering the understanding of how contact with the leaching solution can affect the porosity and permeability of leaching columns is essential: structural changes resulting in reduced permeability may reduce metal recoveries and could lead to the techno-economic failure of a leaching heap (Wang et al., 2021b).

In addition to tracking structural changes, micro-CT has been used to provide accurate structural information to leaching models (Lin et al., 2016a), aiming to relax premises from the shrinking core model (Levenspiel, 1972) about particle shape, grain size, and grain spatial distribution. These calibrated models can then be used to study, predict, and optimise leaching performance for specific cases in a more reliable way by reducing the number of assumptions required to run simulations from these models. Lin et al. (2016a) used micro-CT to develop a leaching kinetic model at the particle scale. This model was fed with information from the first 53 days of leaching. Data from the remaining 115 days of leaching was then used to successfully validate the model's predictions (Lin et al., 2016a). Further research on this subject is still required, but the results from this study suggest that it is feasible to run shorter column leaching experiments and use the CT-adjusted models to extrapolate the results. This would save resources and shorten optimisation cycles.

3.2. *Structural assessment of stages prior to leaching*

Micro-CT has also been used to evaluate the effect of applying comminution strategies to ores that will then be subjected to column leaching. The study by Kodali et al. (2011) used micro-CT as a tool to compare the effect of two different crushing methods on the subsequent leaching of copper-containing ore. Both columns were packed with particles of a comparable size. Particle reduction was performed using high-pressure grinding rolls (HPGR) for one column and jaw crushing for the other. The authors evaluated the progress of leaching by focusing on the extent of dissolution of specific mineral grains from micro-CT images from each column. This helped them assert that the particles obtained from HPGR were more responsive to column leaching than those obtained from jaw crushing (Kodali et al., 2011).

A similar finding was obtained as part of another study that compared the effects of using HPGR or cone crushing technologies for sphalerite particle size reduction. Although this study did not perform column leaching as part of the study, micro-CT demonstrated that HPGR produced a more extensive microcrack network, (Ghorbani et al., 2011) which was found to have a positive effect on bioleaching after comminution (Ghorbani et al., 2013, 2012). A similar finding was obtained in a study by Zhong et al. (2023), in which SEM and column leaching tests were used to evaluate the effect of different crushing methods on the microcrack network of a low-grade copper sulphide ore. SEM micrographs revealed that HPGR yielded particles with larger and more abundant microcracks than those obtained using a jaw crusher or a roller crusher. Column leaching tests confirmed that this increase in particle porosity led to a higher copper extraction rate (Zhong et al., 2023). It should be noted that SEM-based estimations (using mineral mapping software) of porosity are expected to differ from those based on micro-CT due to stereological effects, even if both techniques are being applied with the same resolution (Ghorbani et al., 2011; Reyes et al., 2017).

The study by Nwaila et al. (2013) used micro-CT to evaluate the use of HPGR before performing cyanidation of a sulphide-rich gold-containing ore. The authors used three different pressure settings (60 bar, 90 bar, and 120 bar) for the HPGR and separated the HPGR products into two size fractions. The authors identified that cyanidation efficiency was higher for smaller particles obtained by using a 90 bar pressure setting for the HPGR. This hydrometallurgical result agrees well with the results obtained from micro-CT: small particles obtained from a 90 bar pressure setting for HPGR have the highest porosity and the widest cracks, which promotes a more thorough cyanide penetration into the particles (Nwaila et al., 2013). Charikinya et al. (2015) used micro-CT to evaluate the effect of performing a microwave treatment on a crushed sphalerite ore. These particles were pretreated using either an HPGR or a cone crusher. This study found that a microwave treatment could increase the porous network significantly, by generating both grain boundary and *trans*-granular microcracks. The study also identified that the effect of the microwave treatment is independent of the crushing method used in the previous stage (Charikinya et al., 2015).

The studies conducted by Kodali et al. (2011), Ghorbani et al. (2011, 2012, 2013), Zhong et al. (2023), Nwaila et al. (2013), and Charikinya et al. (2015) outline how micro-CT can be used to study structural changes induced by pretreatments or comminution stages before column leaching. Coupled with its capacity to follow structural changes during column leaching, as demonstrated by Phyo et al. (2020) and Ai et al. (2022), micro-CT can be used to track the entire metal liberation and extraction process. As discussed earlier, this structural information is valuable as it constitutes a key factor in determining whether the leaching agent will be able to reach the target minerals during heap operation. Moreover, the research produced by these authors is valuable as it demonstrates that a single 3D imaging technique can produce structural information throughout subsequent stages of the beneficiation process.

Agglomeration and curing are often considered before column or heap leaching. However, limited information is available about the curing stage, due to the lack of models and experimental information focusing exclusively on understanding this process and not only on its effects on subsequent leaching. To bridge this knowledge gap, Salinas-Farran and Neethling (2023) cured agglomerates in columns for 65 days. During this period, the authors scanned the columns using micro-CT to obtain spatiotemporal information on the structural, physical, and chemical processes occurring as part of the curing stage. The 3D images were then used to validate a mathematical model that was developed to consider many of the subprocesses that occur during the curing of agglomerates (i.e., mineral dissolution, transport of species, loss of moisture through evaporation, and reprecipitation of species within the structure of the agglomerates). The results from this study demonstrated that the phenomena that occur during curing of agglomerates can be modelled successfully, which opens the possibility of reducing the number of experiments required to assess and optimise curing stages through the use of simulations (Salinas-Farran and Neethling, 2023).

4. Mineralogical quantification

Microscopy techniques (e.g., polarisation microscopy or SEM-EDS coupled with mineral mapping software) are often used to study the liberation of valuable mineral grains in samples and to quantify their mineralogical composition. To perform these analyses, the samples usually need to be mounted in resin before creating polished flat cross-sections, which means that it is not possible to undergo *in situ* liberation assessments and mineralogical quantification based on microscopy. However, non-destructive imaging techniques like CT can be used to perform these analyses during leaching without disrupting the packed bed.

Two initial micro-CT studies in the field of mineral processing by Miller et al. (2003a, 2003b) showed that this technique can assess the exposure of copper-containing minerals before column leaching. This could be used to make copper extraction predictions from column leaching experiments. These studies classified sulphide-based and oxide-based copper ore in different size fractions. Each of these fractions was then analysed individually using micro-CT to determine the total copper content and the exposed copper content. This allowed estimating overall copper exposure for each composite by averaging size fraction-based results while considering the particle size distribution. Exposure estimation was achieved by differentiating copper-containing grains from air and gangue based on their distinct linear attenuation coefficients. Column leaching of the composites revealed that the predicted copper extraction based on exposure estimates were slightly lower than the real copper extractions. This proved that micro-CT is a reliable technique for copper exposure estimation (Miller et al., 2003a, 2003b). According to the authors, the difference between the predictions and the extraction results can be attributed to two factors: (1) micro-CT resolution limitations and (2) unexposed grains can become exposed during leaching (Miller et al., 2003b). While this study did not use CT during column leaching, it proved that this technique can be useful for studying the leaching potential of samples before conducting experiments. Beyond the field of column leaching, other studies have also utilised CT to quantify the degree of liberation of specific

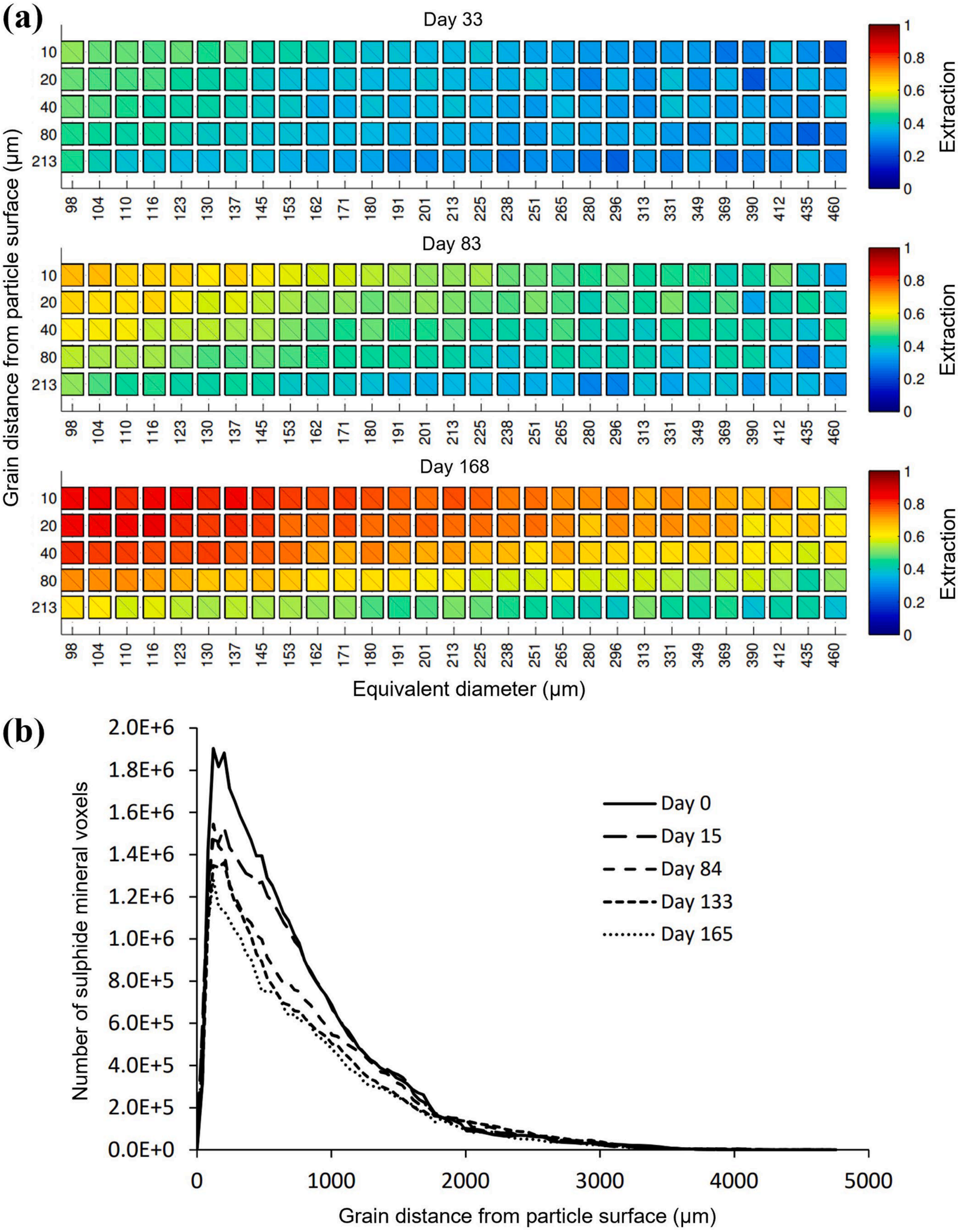

Fig. 4. Position and time dependent metal sulphide grain dissolution tracking as a way of unlocking spatiotemporal insights in copper column leaching experiments. (a) Average metal sulphide dissolution in categories based on grain diameter and grain distance to particle surface after 33, 83, and 168 days of leaching. Figure reprinted (minor adaptations) from Lin et al. (2016b, Elsevier, Creative Commons CC-BY). (b) Sulphide content (by number of sulphide-containing voxels) vs distance from grain to particle surface after 15, 84, 133, and 165 days of leaching. Figure reprinted (minor adaptations) from Ghadiri et al. (2020, MDPI, Creative commons CC-BY).

mineral grains before other mineral processing operations (Miller et al., 2009; Reyes et al., 2018; Wang et al., 2017).

In situ liberation and mineralogical quantification assessments as part of column leaching experiments have been performed using CT. Lin et al. (2016b) used micro-CT to evaluate and model column leaching kinetics of copper sulphides at both grain and particle scale. Micro-CT was used to follow changes in the solids at these two scales during leaching by segmenting and tracking particles and grains over time, even if they move during this period. This approach facilitated the assessment of the factors that may influence column leaching. The authors found that the location of a particle in a column had a weak effect on its individual leaching performance. The study then grouped the imaged grains based on their size and location relative to the surface of the particles. The averaged leaching progress (extraction) for each group was then estimated based on the progress of sulphide dissolution for each grain, as shown in Fig. 4a. Among other findings, micro-CT demonstrated that the size of the grains and their distance from the surface of particles have an inverse effect on leaching performance. While this finding was expected, it was also identified that the effect of grain size is reduced as leaching progresses (Lin, 2015; Lin et al., 2016b).

Ghadiri et al. (2020) used micro-CT to quantify changes in the volume occupied by metal sulphides during column leaching of three different ores (malachite, chalcopyrite, and pyrite). These results were used to calculate the sulphide leaching progress as a function of time. These estimates agreed with the conventional estimation of leaching extent based on solution assays. In addition to this, following grains located at different distances from the surface (Fig. 4b) provided insight into the reaction mechanism that controls the leaching of each sample (Ghadiri et al., 2020). The two panels shown in Fig. 4 reveal the importance of spatiotemporally tracking grain dissolution progress, as the leaching kinetics of each grain are affected by their distance to the surface of the particles.

Similarly, Fagan-Endres et al. (2017) used micro-CT to track the progress of column leaching in chalcopyrite ore as a function of the axial and radial position of the particles in the column. The authors also tracked the leaching progress within each individual particle, focusing on the effect of the distance between target grains and the surface of the particles, which had a low porosity. It was identified that there was a 2 mm penetration limit from the surface for the leaching agent: grains located beyond this depth were not leached (Fagan-Endres et al., 2017). Other studies tracking the effect of grain location relative to a particle's surface are available in the literature (Reyes-Leiva, 2018; Salinas-Farran et al., 2022). These studies are essential, as understanding the spatial heterogeneity governing leaching extent can be useful to determine whether additional strategies (e.g., comminution or chemical pre-treatments) may be required to deal with diffusion-related issues and increase the overall extraction.

Distinguishing between different sulphides from micro-CT images is a challenging task due to them having similar linear attenuation coefficients (Ghadiri et al., 2020; Lin et al., 2016a, 2016b). Enabling this distinction would permit extracting key information from micro-CT images, as some sulphide minerals contain valuable metals (e.g., copper sulphides) while others do not. In contrast, SEM-EDS (when coupled with mineral mapping software) excels at identifying specific minerals, including sulphides. However, this technique is limited to providing 2D information and is typically destructive. The integration of micro-CT and SEM-EDS results enables the distinction between sulphide minerals without losing the advantages associated with 3D imaging (Reyes-Leiva, 2018; Reyes et al., 2017; Salinas-Farran et al., 2022). Consequently, the combination of these techniques constitutes a powerful tool for assessing and quantifying leaching performance at a grain scale.

Research on this topic has focused on developing a methodology to combine the information provided by these techniques at a grain scale. This methodology involves matching specific grains between 3D micro-CT images and 2D SEM-EDS images (processed with mineral mapping software). After this, mineralogical identification information from these grains is provided as an input for the segmentation of grains in the 3D images. An initial test of this methodology was capable of distinguishing between copper sulphides, pyrite (FeS_2), other gangue, and air (porosity) in ore samples. However, distinguishing between the different copper sulphide minerals was not possible with this methodology (Reyes et al., 2017). A second study on this subject demonstrated that this methodology can be refined to identify specific minerals in micro-CT images of agglomerates during curing and leaching. The added complexity of applying the methodology to agglomerates is that they can break, shrink, or deform during curing and leaching, behaviours that would not be expected from ore particles (Salinas-Farran et al., 2022).

Micro-CT has proven to be a critical source of information for column leaching studies focusing on the non-destructive and non-disruptive *in situ* quantification of liberation and mineralogical changes. This application of micro-CT represents a significant advancement in achieving a comprehensive understanding of the spatiotemporal heterogeneity of column leaching experiments. Moreover, this tool has shown remarkable versatility in terms of the information that can be extracted from it directly, as well as due to the potential of combining its data with that of 2D mineral maps, leading to the creation of 3D mineral maps, capable of distinguishing between minerals with similar linear attenuation coefficients.

5. Surface-level analysis

The most extensively studied surface-level phenomenon during leaching is passivation (Li et al., 2013; O'Connor and Eksteen, 2020). However, other surface processes have also received attention, including preg-robbing, and the depletion or enrichment of species at the surface of ores (Larrabure and Rodríguez-Reyes, 2021; Senanayake, 2008). Passivation is a phenomenon in which a layer that is mineralogically different to the rest of the ore may form over its surface during leaching. By covering the target minerals, these surfaces may hinder the leaching reaction, as the leaching agent would now need to diffuse through the passivation layer (Córdoba et al., 2008b; Klauber, 2008). Depending on the nature of the layer and of the leaching system, passivation can negatively affect the kinetics of the process to different extents. Many different leaching systems are known to be affected by passivation phenomena, including cyanide leaching of gold and silver (Bas et al., 2018; Larrabure et al., 2021; Zia et al., 2019), sulphuric acid leaching of chalcopyrite (Peng et al., 2019; Zhang et al., 2019; Zhong and Li, 2019), or leaching of sphalerite using different media (Da Silva et al., 2003; Nikkhou et al., 2019). Understanding the causes of surface passivation and the characteristics of this layer can provide insight into strategies for enhancing leaching processes.

Despite the importance of understanding surface passivation, to the authors' knowledge only one study has assessed this phenomenon during column leaching. This study focused on the bioleaching of chalcopyrite ore in columns. The columns used in this study were designed with removable chambers to extract solid samples from the leaching system for analysis. The removed samples were then chemically (inductively coupled plasma – atomic emission spectroscopy – ICP-AES) and mineralogically (SEM-EDS and optical microscopy) characterised. Solid characterisation was performed before bioleaching and at three points during the leaching period. The study found that several surface-level processes occurred simultaneously, resulting in the formation of layers that act as diffusion barriers. The main components of these layers were identified as secondary iron oxides, covellite, and mixed polysulphides, all of which were either formed at the surface or precipitated over it during leaching (Van der Meer et al., 2009).

Surface passivation in column leaching experiments has received limited attention. However, numerous studies have focused on evaluating it using other leaching set-ups, with lab-scale batch reactor leaching being most common. While these studies are not dedicated to column leaching, they will also be covered in this section, as the techniques used in them could be applied to column leaching experiments in the future.

Parker et al. (2003) used XPS during chalcopyrite oxidative acid leaching to gain insight into the leaching and passivation mechanisms of the process. XPS detected that elemental sulphur, a pyritic-like disulphide, and ferric sulphate form over the surface during distinct stages of the leaching mechanism. The study also found that the ferric sulphate formed during leaching contributes to the formation of jarosite, which forms part of the passivation layer (Parker et al., 2003). Another study using XPS also identified jarosite phases as constituents of the passivation layer formed during chemical leaching and bioleaching of chalcopyrite (Sandstrom et al., 2005). However, Khoshkhoo et al. (2014) were unable to link passivation with jarosite. Instead, they used XPS to identify that the main components of the passivation layer were elemental sulphur and iron oxyhydroxides (Khoshkhoo et al., 2014). Another XPS study found that while both elemental sulphur and polysulphides build up on the surface of chalcopyrite during leaching, they do not cause surface passivation (Harmer et al., 2006).

The results from the previous studies appear to be contradictory, but this is due to the uniqueness of each ore and to differences in the leaching procedure, which highlights the usefulness of using XPS to study specific leaching systems: this information can then be used to devise strategies to enhance leaching processes. In addition to the previously discussed studies, others have also used XPS alone or combined with other techniques to assess surface passivation during leaching of chalcopyrite (Fu et al., 2012; Hackl et al., 1995; Hua et al., 2018; Kartal et al., 2020; Klauber et al., 2001; Li et al., 2016; Mikhlin et al., 2004; Wang et al., 2016) or of other ores (Hashemzadeh and Liu, 2020; Liu et al., 2017; Masaki et al., 2018; Mwase and Petersen, 2017).

Beyond assessing passivation, XPS has received attention as a technique to study other surface-specific processes that occur during leaching or leaching pretreatments. For example, XPS has provided inputs to elucidate reactions leading to the oxidation, reduction, or dissolution of species located at the surface, as well as the precipitation of previously leached species over the surface of the ore (Cao et al., 2020; Deschênes et al., 2012, 2006, 2000; Larrabure et al., 2023, 2021; Li et al., 2023; Mikhlin and Romanchenko, 2007; Portilla et al., 2020; Silva-Quiñones et al., 2018; Tan et al., 2005). These surface processes may or may not have an effect (beneficial or detrimental) on leaching performance. However, understanding them can help explain leaching mechanisms and provide insight leading to the enhancement of leaching processes.

ToF-SIMS has received less attention than XPS as a tool to study surface processes as part of leaching experiments. However, this technique can produce elemental depth profiles, which can provide insight into the composition and thickness of surface layers on samples (Ren et al., 2022; Zuo et al., 2023). For example, Ren et al. (2022) used ToF-SIMS to study the mechanism of chalcopyrite passivation by estimating depth-dependent changes in the copper to iron ratio before and after performing oxidative acid bioleaching on the first 65 nm from the surface of samples. This assessment showed that the surface of the leached sample had a higher copper to iron ratio than the unprocessed sample, which was attributed to the formation of a copper rich layer similar to covellite. This layer was found to alter the electrochemical environment of the system, resulting in slower leaching kinetics at a volumetric level (Ren et al., 2022). In a later study, Ren et al. (2023) used ToF-SIMS to study the addition of ethylene thiourea to suppress the formation of the covellite-like passivation layer during oxidative acid bioleaching. ToF-SIMS revealed that the resulting passivation layer was thinner when ethylene thiourea is present in the leaching solution than when it is not. This was attributed to ethylene thiourea partially dissolving the passivation layer, thus exposing fresh chalcopyrite surfaces and favouring leaching kinetics (Ren et al., 2023).

Surface-level processes are essential to expand the understanding of leaching mechanisms and support decision-making, enhancing process optimisation. Surface assessments have generally been neglected as part of column leaching experiments, though they are necessary to fully characterise how ore particles react during scaling up to industrial heap operations. The studies based on XPS or ToF-SIMS presented here are all associated with batch leaching experiments: these techniques have not been used as part of column leaching experiments to the authors' knowledge. Despite that, these studies support the usefulness and applications of the techniques, whose use could be adapted to column set-ups in the future. To achieve this, modified column leaching set-ups that consider sampling chambers would be needed, such as the one proposed by Van der Meer et al. (2009).

6. Hydrodynamic assessment

6.1. *Liquid holdup and distribution assessment*

Numerous studies have aimed to quantify liquid holdup and assess liquid distribution within column leaching systems. Research on this topic is relevant as leaching will only occur if the leaching agent contacts the target mineral. In that sense, the leaching extent will be limited by the ability of the leaching solution to reach the location of the minerals of interest. The main two assessment methods identified in the literature are gravimetric methods and RTD analysis, as described in Section 2.3. The various applications of these methods will be described in this section.

Ilankoon and Neethling (2012) introduced spherical glass beads to a column set-up capable of quantifying liquid holdup gravimetrically. The use of these non-porous beads proved that interparticle liquid holdup in columns during steady state operation shows hysteresis: holdup depends not only on current flow rate but also on flow history. The authors identified that this dependency can be explained mainly by changes in the amount of flow paths and not by changes in the shape of the rivulets (Ilankoon and Neethling, 2012). A second study from the same authors compared the effect of porous (copper ore) and non-porous (spherical glass beads) particles on liquid holdup during steady state operation. The study found that porous particles could retain a significant amount of water, which can impact liquid holdup in ways that cannot be explained by the same mechanism that characterises liquid holdup in non-porous systems. However, decoupling liquid holdup within particles by saturating the porous ore before beginning the experiment causes both systems to exhibit the same liquid holdup mechanism, regardless of their porosity (Ilankoon and Neethling, 2013). A subsequent study used the same experimental set-up to model transient liquid holdup during the initial irrigation and drainage periods of porous and non-porous packed columns. The model presented in this study and the experimental procedure to obtain its parameters were found to be capable of predicting the liquid holdup in packed beds during both steady state and transient operation (Ilankoon and Neethling, 2014).

Wang et al. (2021a) employed a similar set-up to assess the effect on the liquid holdup in an unsaturated leaching column when using stepwise irrigation (flow rate is increased in discrete steps or cycles towards a target value, but irrigation is stopped after the operational holdup stabilises and is then resumed once a non-operational residual holdup stable value is achieved). The authors showed that, for the same target irrigation flow rate, a higher liquid holdup can be achieved if stepwise irrigation is used than if uniform irrigation is considered. These results were attributed to the pre-wetting of the bed during the cycles, which facilitates the access of liquid to previously unwetted pores during subsequent cycles. Ultimately, these results are also linked to the liquid holdup hysteresis behaviour of unsaturated porous bed systems, as residual liquid is always retained in interparticle and intraparticle pores, even after irrigation is stopped (Wang et al., 2021a).

Mostaghimi et al. (2014a, 2014b) also used a gravimetric approach to validate fluid flow results from a control volume finite element method simulation with mesh adaptivity (Mostaghimi et al., 2014a, 2014b). Two different packed beds were used as test cases: (1) a homogeneous 2 mm glass spheres bed, and (2) a bed holding 2 mm and 10 mm glass spheres separated in two vertically contacting regions. This study found good agreement between the experimental liquid holdup and the results from the simulations for the two test cases (Mostaghimi et al., 2014a). These studies provide insight into the temporal heterogeneity associated with the liquid holdup phenomenon in leaching column systems. However, by considering only the total weight variations in the column due to liquid holdup, this type of set-up is unable to identify if the system may exhibit spatial heterogeneity (i.e., holdup occurring in preferential regions within the column).

A different liquid holdup and distribution assessment set-up was devised by Ilankoon and Neethling (2016) to gain insight into the liquid spread mechanisms that occur in packed beds. The experimental rig consisted of a transparent, narrow, and rectangular column (800 mm × 600 mm × 100 mm) that had solution emitters at the top and solution collection points at the bottom, both of which were linearly spread out. While this set-up differs from the traditional cylindrical shape of leaching columns, its characteristics allow it to yield valuable spatiotemporal information that cannot be accessed using a regular column. The shape and transparency of this set-up allows observing how the liquid spreads out as it percolates vertically (rapid gravity induced flow) or horizontally (slow capillary flow), as shown in Fig. 5a. Likewise, having multiple collection points allows identifying the formation of preferential channels (Ilankoon and Neethling, 2016). Results from this study were then used to validate the simulations produced by a computational fluid dynamics (CFD) model. The experimental results agreed with the simulation if the model accounts for packing variations leading to the presence of local preferential flow paths (McBride et al., 2017).

Ilankoon and Neethling (2019) then refined their previous methodology to produce information leading to a more detailed spatiotemporal understanding of the liquid spread mechanisms in packed beds. The authors irrigated the rectangular column with a UV fluorescent dye tracer (2.5 g/L sodium fluorescein). Under normal light, dry areas can be distinguished from wet areas, but UV light reveals specific areas where the solution flow is more active (rivulets), as shown in Fig. 5b. The authors recognised that wall effects due to using a narrow column can affect the results, as wall flow was detected in the system. The authors indicate that further experiments to decouple wall flow from characteristic flow mechanisms are required (Ilankoon and Neethling, 2019).

Liquid distribution and liquid holdup in column leaching systems have also been studied through residence time distribution (RTD) analyses using tracers (Bouffard and Dixon, 2001; de Andrade Lima, 2006; Fagan-Endres et al., 2023; Odidi et al., 2023). Bouffard and Dixon (2001) used a sodium nitrate solution as a conductivity tracer to supply parameters (including the ratio of stagnant liquid holdup to flowing liquid holdup) to three diffusion models that describe fluid flow through columns packed with agglomerated or non-agglomerated ore. The simulations based on these models were then compared with experimental data to identify the best-fitting model for the specific leaching system under study. Further experiments then proved that the models validated using small columns can also be representative of larger systems. However, the authors recognised that their study was limited to a short time period, so the models may not be able to predict the hydrodynamic behaviour of these systems if slow time-dependent processes occur concurrently, such as fine particle migration or particle breakage (Bouffard and Dixon, 2001). De Andrade Lima (2006) performed an RTD analysis using hydrochloric acid as a pH tracer to study the effect of liquid flow rate on liquid holdup and axial dispersion within column leaching systems. The occurrence of axial dispersion (deviation from plug flow model) had not previously been considered by Bouffard and Dixon (2001) and was found to be a non-negligible phenomenon in the context of hydrodynamic leaching column modelling (de Andrade Lima, 2006).

Odidi et al. (2023) identified four frequently neglected aspects in previous RTD experiments for column leaching: (1) particle shape and particle porosity effects have not been decoupled, (2) the effect of the intrinsic wettability of particles has been ignored, (3) particle sizes have been characterised by narrow distributions or have not considered fine particles, and (4) changes in fluid viscosity have not been assessed. To address these issues, a set of thirty RTD experiments using a potassium chloride solution as a conductivity tracer were performed. To ensure a better time resolution, their workflow incorporated the modelling approach proposed by Fagan-Endres et al. (2023) and described in Section 2.3.2. These experiments tested five different solution viscosities (adjusted using viscosity modifiers) and four different particle types (with differences in porosity, shape, wettability, and size distribution). Among the non-ore-specific conclusions of this study, particle porosity was found to affect flow profiles more than the other factors, with higher porosities resulting in an increase in wettability (Odidi et al., 2023). Other studies have also used RTD analyses using tracers to study liquid holdup and distribution in similar settings (Bouffard and West-Sells, 2009; Decker and Tyler, 1999; Petersen and Petrie, 2000).

This section has presented the main strategies that have been applied by researchers to assess the liquid holdup and distribution in column leaching systems. Gravimetric liquid holdup experiments and RTD analyses have been recognised as tools to comprehend the hydrodynamic phenomena occurring within leaching columns. While these assessments can provide insights into the temporal heterogeneity of these systems to a certain extent, most of them still have an important limitation: they rely on input–output measurements or in whole-volume liquid holdup results, neglecting partially or totally the spatial heterogeneity of the column leaching system.

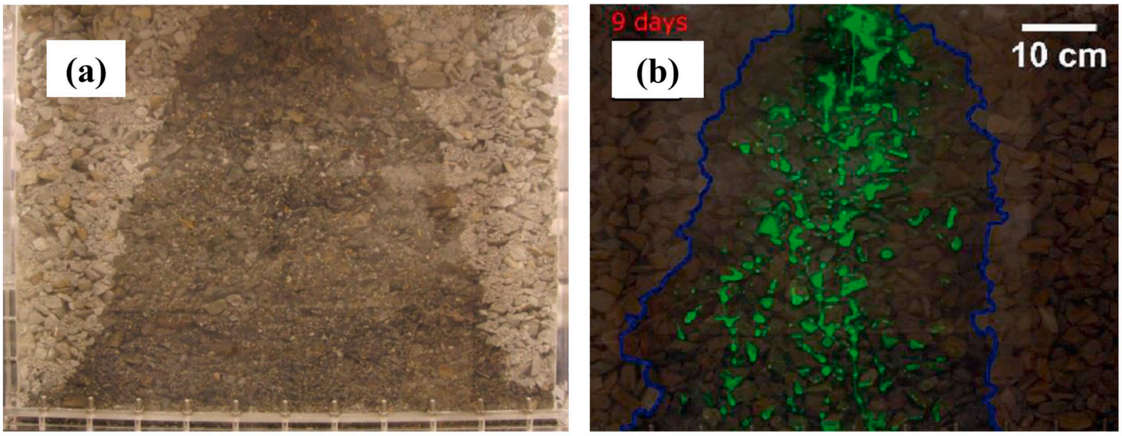

Fig. 5. Liquid spread mechanism assessment methods in rectangular columns packed with ore. (a) Changes in colour allow identifying the distribution or spread of liquid over time. Figure reprinted from Ilankoon and Neethling (2016), with permission from Elsevier and (b) a fluorescent dye tracer can be used to distinguish between wet regions with different levels of flow activity. Figure reprinted from Ilankoon and Neethling (2019), with permission from Elsevier.

6.2. *Application of 3D imaging techniques for fluid flow assessment*

The use of 3D imaging techniques to obtain insights into the hydrodynamic spatiotemporal heterogeneity of column leaching systems has been considered by researchers. For this application, the focus has been placed on both CT and MRI. These techniques can overcome the limitation posed by the assessment methods described in the previous section: 3D imaging can track the location of the solution over time accurately and without causing major disturbances in the system.

For instance, researchers have focused on obtaining experimental information to model fluid flow and how it distributes inside columns. Lin et al. (2005) used milli-CT to supply geometrical and structural information from column leaching for fluid flow simulations based on the Lattice-Boltzmann method. These simulations revealed that most of the fluid flow was constrained in a small percentage of the available pore network. Likewise, the simulation provided permeability parameters, which agreed well with calculations based on the porosity of the sample (Lin et al., 2005). A study with a similar aim by Videla et al. (2008) used micro-CT instead of milli-CT to supply information for Lattice-Boltzmann simulations considering a finer resolution. Results from the simulation were then confirmed experimentally. Although this study did not focus on a column leaching setting, it further demonstrated that CT is a powerful tool capable of providing information to refine simulations of fluid flow through packed beds (Videla et al., 2008). Miao et al. (2017) focused on using micro-CT images to model fluid flow in column leaching both at an interparticle scale (free channel flow, governed by the Navier-Stokes equations) and at an intraparticle scale (porous media flow through a heterogeneous pore structure, governed by the Brinkman equations). The authors concluded that this two-scale assessment can represent the flow behaviour of the solution in leaching columns more accurately than models that only focus on either one of the two scales (Miao et al., 2017).

MRI has received attention as part of column leaching studies on the distribution of liquid and gas within the packed bed. It is known that MRI can produce distorted images if the scanned objects have ferromagnetic or paramagnetic properties. Acknowledging this limitation is important as materials with these properties are expected in a wide variety of ores. This limitation prompted a study to use spheroidal agar beads of varied sizes instead of real ore particles to study the saturated flow velocity characteristics in leaching columns (Li et al., 2018). However, it is possible to avoid these image distortions by using special MRI acquisition sequences. On that matter, Fagan et al. (2012) evaluated different MRI techniques to obtain images of fluid flow through a low-grade copper ore. The assessed techniques were spin echo frequency encoded (SEFE) imaging, single point imaging multiple point acquisition (SPI-MPA) imaging, and spin echo single point imaging (SESPI). The images obtained by these methods were compared with equivalent CT images, which led to identifying that SEFE imaging techniques were unable to provide an accurate representation of the sample. SPI-MPA and SESPI techniques achieved better results, but the SESPI technique provided images with better defined particle edges and with a higher signal to noise ratio for the same acquisition time (Fagan et al., 2012).

Fagan et al. (2013) performed MRI of a flooded (saturated) column using the SESPI imaging acquisition sequence. The images allowed mapping the voids that were reachable by water (interparticle and intraparticle pores). After desaturating the column, an unsaturated experiment was performed to map the positions where liquid flows during leaching. Combining these images yielded information about the solid–liquid-gas distribution during leaching, which is shown in Fig. 6a for a single slice and in Fig. 6b for the whole scanned volume. In turn, this led to the estimation of the areas of the solid–liquid, solid–gas, and gas–liquid interfaces. The assessment of the first two parameters can provide an estimation on the quality of the contact between the surface of the particles and the leaching solution (Fagan et al., 2013).

A subsequent study by Fagan-Endres et al. (2015) used MRI to assess the effect of different liquid flowrates on the behaviour of liquid holdup in unsaturated columns. MRI provided evidence that increasing the liquid flowrate decreased the solid–gas interface area and increased the solid–liquid interface area. Likewise, MRI showed that a higher flowrate led to the formation of new liquid paths within the column, as well as to thicker liquid films. However, a second experiment led the authors to acknowledge that this improvement is restricted to columns of a small

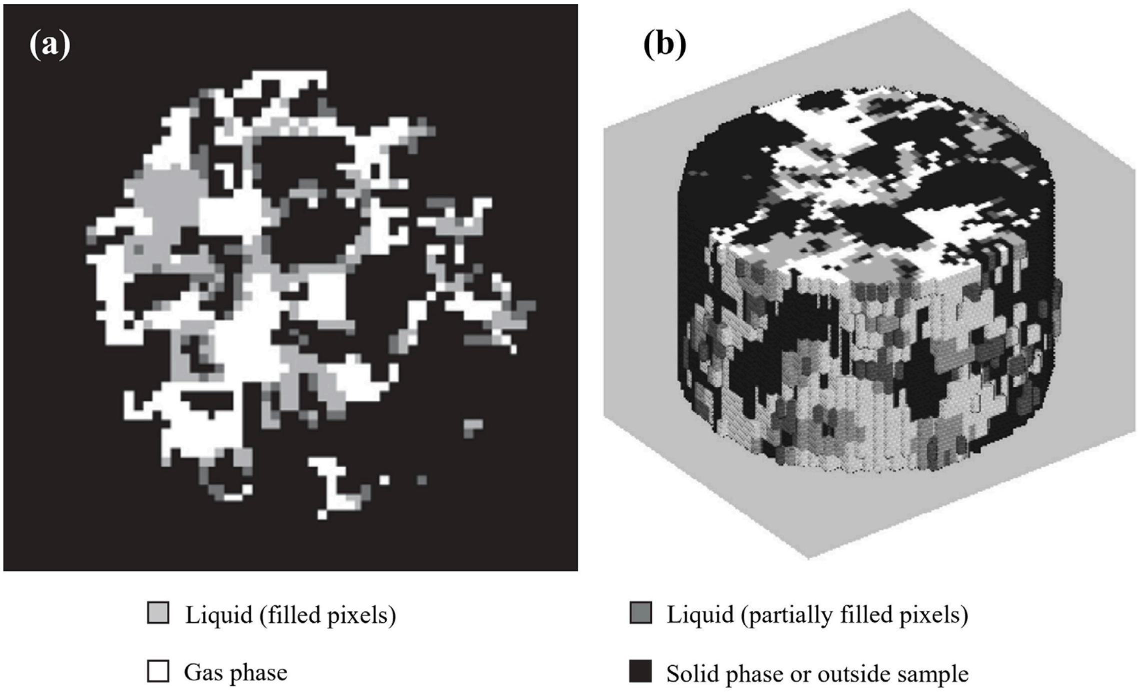

Fig. 6. Solid-liquid–gas phase mapping based on MRI data as a tool to understand the spatial heterogeneity of column leaching hydrodynamics. (a) 2D phase map corresponding to a single slice and (b) 3D phase map corresponding to the full scanned volume during unsaturated low-grade copper ore leaching. Figure reprinted (minor adaptations) from Fagan et al. (2013), with permission from Elsevier.

diameter. The second experiment involved performing MRI during a similar procedure using a different geometrical configuration. This experiment showed that changes in the liquid holdup were restricted to the volume directly beneath the solution input. Thus, increasing liquid flowrate would have a negligible effect on liquid holdup in larger systems, such as those characteristic of heap leaching (Fagan-Endres et al., 2015). Yu and Gao (2020) used MRI to analyse the effect of providing aeration during leaching on the liquid content within columns. MRI showed that aeration promoted a higher liquid content in the columns, leading to a larger solid–liquid interface area during leaching (Yu and Gao, 2020).

As discussed in this section, column leaching hydrodynamic studies involving the use of CT have focused on supplying parameters for modelling and simulation purposes. In contrast, MRI has been used to provide insights on how liquids and gases are distributed around solid particles in unsaturated columns, even when paramagnetic or ferromagnetic materials are present. These MRI studies can display the channels through which liquids percolate through the column and assess the extent of the contact between the liquid and the solid. This information is useful as increasing the liquid–solid interface area can lead to a greater interaction between the leaching agent and the solid particles. It should be noted that these studies used water instead of solutions with leaching agents. Further studies on the use of MRI in systems with solutions with leaching capabilities are still required.

7. Modified destructive assessment methodologies

Studying the temporal heterogeneity of the solids within a leaching column should imply non-destructive assessments throughout the experimental period, so that the process can resume after the analysis. However, most conventional solid analysis techniques associated with column leaching are destructive. Two studies were found to have adapted conventional solid assessment techniques so that they work around the destructive nature of these methods (Govender et al., 2016; Zou et al., 2015). In this way, these methodologies use conventional tools but manage to capture the temporal heterogeneity of solids.

A study involving column leaching of low-grade sulphides evaluated the solid particles within the column at different times. They did this by removing the ores from the column after specific intervals, mixing the particles, extracting samples, and then returning the rest of the ore back to the column to resume the leaching experiment. In this way, representative samples were extracted from the column at different intervals, which were then assayed to determine copper, iron, and sulphur content. No more than 25% of the ore was removed from the columns in total after all the sequential extractions (Zou et al., 2015). Even though the assays were destructive, this methodology can produce information on how metals are removed from the ore at different times before the experiment ends. However, by removing and mixing the ore for sampling before returning it to the column, they were obtaining a homogeneous distribution of solids within the columns before resuming the leaching process. This procedure led to disregarding the spatial heterogeneity of the columns, which could also impact on the characteristics of the pregnant leach solution over the leaching period.

Another study described a methodology to evaluate changes in the microbial population dynamics during the leaching of two groups of columns that were set to two different bioleaching conditions to extract copper. Each group was formed of seven columns that were operated under identical conditions. Every set amount of time, one column from each group was put to an end, and its solids were analysed to study changes in the microbial populations associated with the solid particles within the columns. The sacrifice of columns provides information about what is going on inside each system after specific time periods. Given that the columns were initially identical, the results obtained from the solids after terminating a column in a specific moment should apply to all the remaining columns (Govender et al., 2016). Thus, this methodology enables following the temporal heterogeneity of solid particles. However, a disadvantage of this methodology is that it requires operating many columns in parallel, which can increase the cost of the experiment significantly.

8. Perspective

The aim of this literature review was to comprehensively explore the current trends in column leaching experiments, with an emphasis on strategies that lead to a more thorough understanding of the spatiotemporal heterogeneity of these systems. Learning more about the phenomena that occur as part of column leaching studies is essential as it can help detect process inefficiencies and strategies to optimise leaching operations. By reviewing the existing experimental methodologies from the literature, it was possible to identify procedures and tools that are not ore-specific and can be applied to different column leaching systems to understand each of them individually.

A substantial number of studies were considered for the review, which highlights the continued interest by researchers on topics ranging from structural or hydrodynamic assessments and liberation to mineralogical quantifications in column leaching experiments. These aspects have been studied primarily by using 2D and 3D imaging techniques, as well as by using gravimetric or tracer-based methods to analyse liquid holdup and distribution within packed beds in column systems. However, a key aspect of column leaching experiments has received significantly less attention than others despite its importance: surface-level processes. These processes can be studied by using techniques with different depth spatial resolutions, such as ToF-SIMS, XPS, or SEM-EDS, although only the latter has been applied to column leaching set-ups.

A trend found in the reviewed literature is that authors are using combinations of conventional leaching solution or solid particle assays with strategies involving 2D or 3D imaging, surface-sensitive techniques, or hydrodynamic assessments. These integrated approaches have provided valuable insights into the spatiotemporal heterogeneity of column leaching systems, which otherwise remain black boxes. However, a challenge remains in that most studies in the field have used these approaches to focus on specific aspects of column leaching experiments. These approaches yield results that explain only partially the complexities related to the spatiotemporal heterogeneity. Thus, studies combining the different strategies to thoroughly assess the phenomena that occur simultaneously during column leaching experiments are still needed. This is of particular importance because the same experimental equipment (e.g., CT or MRI) can sometimes be used to study more than one aspect.

The structural assessment of particles in packed beds has received significant attention in the literature. Most studies on this topic used CT due to its non-destructive nature and its ability to produce 3D images of the solids. As such, this technique has been used to supply information about both the microstructure and macrostructure of the packing of the columns, which can be used to obtain key parameters for kinetic (Lin et al., 2016a) or fluid flow (Lin et al., 2005; Miao et al., 2017; Videla et al., 2008) modelling.

Since the structural characteristics of the particles can have an important effect on the overall performance of leaching columns, CT has also been used to study how structural changes occur, both as part of leaching (Ai et al., 2022; Phyo et al., 2020) or as part of pretreatments (Charikinya et al., 2015) or comminution strategies (Ghorbani et al., 2013, 2012, 2011; Kodali et al., 2011; Nwaila et al., 2013; Zhong et al., 2023). However, the impact of some pretreatments or strategies on the structure of particles have not been assessed yet, which may lead to underestimating the effect that these processes may have on leaching efficiency. Additionally, a key limitation identified in structural studies is that the spatial resolution of imaging techniques can restrict their ability to detect cracks or pores in the micron or submicron range. Further technological developments or the incorporation of other existing techniques into the column leaching toolbox is expected to allow future studies to produce images with higher resolutions. This should enable the production of more detailed information that may explain how leaching performance is affected by structural features, such as pore network changes.

Two recurring lines of research are liberation and mineralogical quantifications *in situ*. While 2D imaging strategies are commonplace for these types of measurements, sample preparations cause them to be destructive and do not allow them to be used *in situ* without disrupting the packed bed. Due to this and to the stereological effects associated with 2D imaging, the focus is shifting towards 3D imaging. CT can study the liberation or exposure of target grains before leaching begins, allowing predictions of copper extraction during leaching (Miller et al., 2003a, 2003b). Likewise, CT can also be used throughout the leaching process, to track the leaching progress of individual grains and to measure the effect of size or distance from exposed surfaces on individual grain leaching performance (Fagan-Endres et al., 2017; Ghadiri et al., 2020; Lin et al., 2016b). Applying CT for mineralogical quantification used to be limited to differentiating between minerals with very distinct linear attenuation coefficients. Recent multimodal studies have combined information from 2D mineral maps, therefore producing more comprehensive 3D mineral maps where minerals with similar linear attenuation coefficients can be differentiated (Reyes et al., 2017; Salinas-Farran et al., 2022). Although sp-CT has not been applied to column leaching studies yet, it has significant potential as a tool to non-destructively create 3D mineral maps (Godinho et al., 2021; Sittner et al., 2021).

Although surface-level phenomena are essential in leaching processes, they are rarely studied when leaching is performed using a column set-up. Instead, these assessments are commonly limited to laboratory scale batch leaching experiments. Based on the current literature, the main challenge for surface-level assessments is that of producing samples that can be analysed non-destructively in a recurring way throughout the leaching period. Indeed, only one study was found to focus on surface-level processes as part of column leaching experiments. Van der Meer et al. (2009) proved that examining the surface of samples during leaching without disrupting the packed bed was possible. The authors used SEM-EDS for this task (Van der Meer et al., 2009), which is a moderately surface-sensitive technique. However, their set-up would allow using other more surface-sensitive techniques if desired, like XPS or ToF-SIMS. It is suggested that future research into column leaching experiments should follow surface-level phenomena by using one or more of these three techniques. This would help fill an important knowledge gap in column leaching experiments as these techniques can provide information about the surface that cannot be currently achieved in other ways.

The hydrodynamic characteristics of column leaching experiments are relevant as they establish the quality of contact that may occur between the liquid and solid phases and are a valuable source of insight leading to the optimisation of leaching processes. As a result, many efforts have been placed in studying key aspects like liquid holdup and distribution in leaching column set-ups. A sizable fraction of the studies on this topic used gravimetric liquid holdup assessments (Ilankoon and Neethling, 2016, 2014, 2013, 2012; Mostaghimi et al., 2014a; Wang et al., 2021a) and residence time distribution analyses using tracers (Bouffard and Dixon, 2001; de Andrade Lima, 2006; Fagan-Endres et al., 2023; Odidi et al., 2023) to study how the leaching solution percolates towards the bottom of the columns. However, the insights into the spatial heterogeneity that these methods can produce is often limited as they either focus strictly on the solution inputs and outputs of the columns or on weight measurements, disregarding changes that may be dependent on the position within the column.

Alternatively, MRI can be used to produce 3D images that visually show how the solution distributes or accumulates in the volume. This technique can also be used to quantify the contact between solid surfaces and the solution (Fagan-Endres et al., 2015; Fagan et al., 2013, 2012; Yu and Gao, 2020), although spatial resolution limitations might cause underestimations or overestimations. Cross-validation between the results of gravimetric or residence time distribution analyses and 3D imaging techniques has not been found in the literature, which is an important topic that should be addressed by future studies.

A key research topic that has not been identified in the available literature is that of chemically characterising the leaching solution in a way that allows understanding how factors like pH, oxidation–reduction potential or concentration of dissolved ions change both in the axial and in the radial directions. Conventional column leaching set-ups only study these properties at the input and output of the column, not capturing the chemical spatial heterogeneity of these systems. Studies focusing on the chemical changes in the solution as a function of position would be a valuable addition to the literature. For instance, it would be possible to access this information by setting multiple short columns in series instead of a single long one. This would allow solution aliquots to be extracted from different heights in the column, thus increasing the spatial resolution of the experiment.

This literature review identified that numerous efforts to acquire spatiotemporal insights into column leaching experiments have been made. These efforts have aimed to assess various aspects pertaining both the solid particles and the leaching solution. However, equipment limitations (e.g., insufficient resolution, material restrictions, sample destructiveness) and lack of focus in specific areas have led to some knowledge gaps, which should be addressed as part of future research. In particular, the two topics that require the most attention are the application of surface-sensitive techniques to column leaching experiments and performing chemical solution assays in a way that allows the production of position-dependent information. The development or application of techniques and methodologies capable of addressing these gaps would significantly enhance the current understanding of the spatiotemporal heterogeneity of column leaching. Ultimately, furthering this knowledge has potential economic and environmental benefits, as optimisation and upscaling experiments could become more resource and time efficient.

CRediT authorship contribution statement

Gonzalo Larrabure: Writing – original draft, Visualization, Conceptualization. **Luis Salinas-Farran:** Writing – review & editing, Supervision, Conceptualization. **Stephen J. Neethling:** Writing – review & editing, Supervision. **Pablo R. Brito-Parada:** Writing – review & editing, Supervision.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

No data was used for the research described in the article.

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