

Petrographical and geochemical study of the Eboundja nepheline syenite-derived regolith (Southwest Cameroon): evidence of changes at mineral scale during tropical weathering

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ABSTRACT

Weathering in tropical environments leads to significant changes in the chemistry and texture of primary rocks detectable at both macroscopic and microscopic scales in the resulting weathered materials. This study examines the petrography and geochemistry of nepheline syenite and its derived regolith in Southwest Cameroon, focusing on the mineral-scale responses to tropical weathering. The nepheline syenite protolith consists of microcline, nepheline, plagioclase, amphibole, zircon, magnetite, ilmenite, allanite, pyrochlore, and biotite. Key weathering mechanisms include dissolution, oxidation, chemical replacement, and microfracturing. These processes interact such that all primary minerals, except zircon, are transformed into secondary minerals in the pedolith. Due to its chemical stability, zircon is primarily affected by physical weathering processes such as micro-fracturing and abrasion. Microfracturing plays a crucial role in accelerating weathering intensity in the early stage. Notably, it facilitates the alteration of pyrochlore, the principal primary Nb-bearing mineral, from Ca-Na-F-rich to Ca-Na-F-poor and Sr-rich variants. In the weathered materials, Ti- and Fe-Ti-oxides (ilmenite and leucoxene) are identified as the major Nb carriers, potentially hosting the Nb released from complete dissolution of pyrochlore grains. Niobium is further enriched in weathered ilmenite through oxidative weathering. The humid, high water activity condition also causes direct transformation of biotite and chlorite to kaolinite, gibbsite and goethite. Overall, this study highlights the complex mineralogical and geochemical process, potentially facilitating Nb mineralization, during tropical weathering.

1. Introduction

Rock weathering refers to the chemical and physical degradation of rocks over time (Garczynski et al., 2024). Weathering is intense in tropical environments and gives rise to significant textural, mineralogical and chemical changes of parent rocks (Braun et al., 1990; Etame et al., 2009a; 2009b; Babechuk et al., 2014; Perri et al., 2016; da Silva et al., 2020; Liu et al., 2022; Ntomb et al., 2024). Textural changes include the formation of microfractures, dissolution features, and the surface microtopography that develops as the rock disintegrates and decomposes (Braun, 1991; Navarre-Sitchler et al., 2013). Mineralogical changes include mineral replacements and deposition of secondary minerals from aqueous solutions in cracks and as surface coatings

(Garczynski et al., 2024). Chemical changes are related to the departure of mobile elements and the concentration of immobile elements (Braun et al., 1993; Etame et al., 2009b; Babechuk et al., 2014; Wang et al., 2024; Ntomb et al., 2024). These processes play a crucial role in soil formation, biogeochemical cycles of elements and nutrient availability for plants.

In tropical regions, chemical weathering produces abundant secondary minerals (Etame et al., 2009a; Tsozué et al., 2020; da Silva et al., 2020) which result from the breakdown of primary minerals, mobilization and re-distribution, and precipitation of elements. Goethite, hematite, kaolinite, halloysite and gibbsite are common examples of secondary minerals. These minerals are rarely pure, they often contain trace contents of economically significant elements (e.g. Nb, Zn, Pb, Co,

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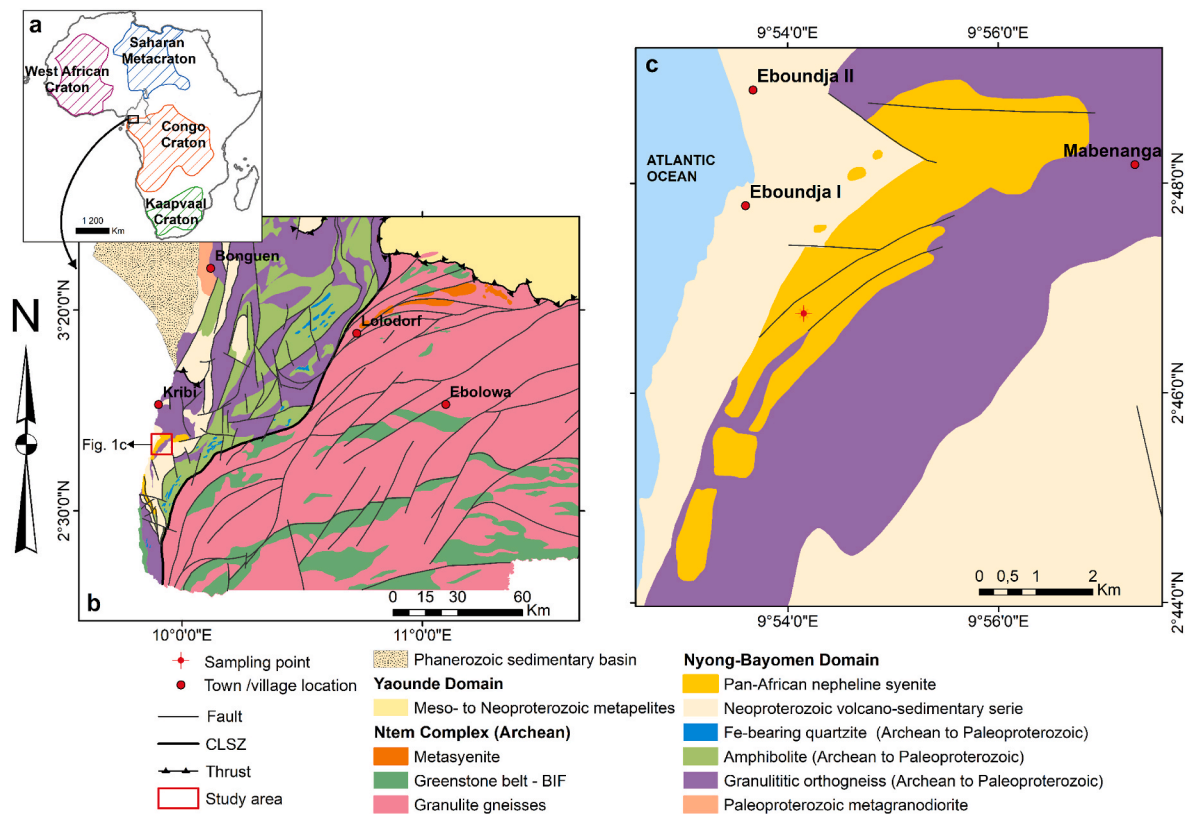


Fig. 1. (a) Main cratonic blocks in Africa (after Haissen et al., 2017); (b) Geological sketch map of Southwest Cameroon (modified after Noel and Roubichou, 1986; Maurizot, 2000; Lerouge et al., 2006; Owona et al., 2021; Delor et al., 2021); and (c) Simplified geological map of the Eboudja nepheline syenite with sampling point location (modified after Noel and Roubichou, 1986; Delor et al., 2021).

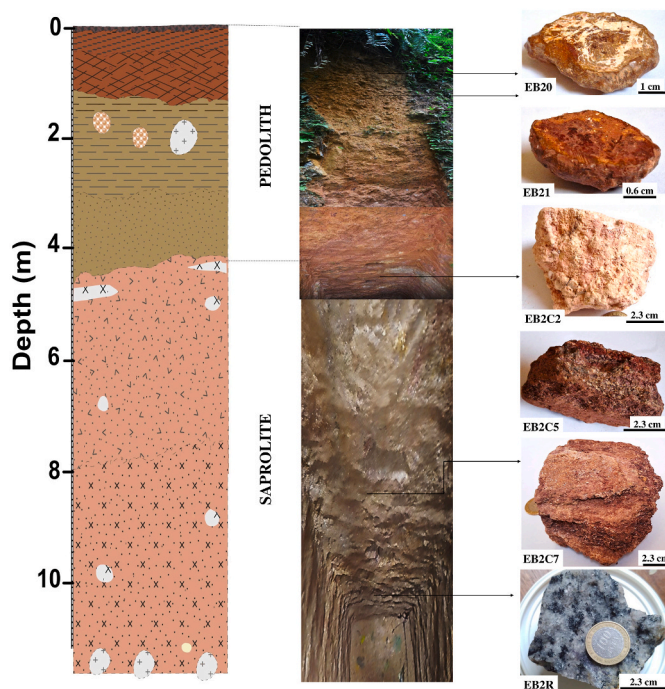


Fig. 2. A schematic weathering profile of the studied site and occurrence of regoliths in the profile.

REE, Sc, G, Ge, V; Kundsén et al., 2015; Ram et al., 2019; Li and Zhou, 2020, 2023) as well as deleterious elements (Hg, Cd, Cr, As; Marques et al., 2014; Hu et al., 2022; Santoro et al., 2020, 2022). Process

involved in the scavenging of trace elements in secondary minerals includes lattice substitution, adsorption, and colloidal binding (Kundsén et al., 2015; Wu et al., 2023). Previous studies show that, the behaviour of trace elements in soils is primary dependent of the nature of the bedrock and its mineralogy (e.g., Giovannini, 2013; Giovannini et al., 2020; Fu et al., 2019; da Silva et al., 2020). Thus, understanding the mineralogical process for primary mineral breakdown and secondary mineral neoformation during weathering would be the key to explain the supergene mobilization and enrichment of the abovementioned economic or hazardous trace elements. Nonetheless, few works have been done on a mineral scale (Ojanuga, 1973; Braun, 1991; Wall et al., 1996; Santoro et al., 2022; Machado et al., 2021), though there have been studies on weathering of igneous rocks under tropical climate (Braun et al., 1990; Nguetkam et al., 2007; Sababa et al., 2021; Ntomb et al., 2024). Among different trace elements, niobium (Nb) is of particular interest both for its high economic value, and for understanding geological processes, since it is generally immobile during supergene weathering. The latter results from the resistance of common Nb-bearing minerals (such as pyrochlore group minerals) to alteration and its low solubility in fluids (Nahon and Merino, 1996; Kurtz et al., 2000; Peiffert et al., 2010; Guinoiseau et al., 2021). Tropical weathering of carbonatites and silica-undersaturated rocks could lead to the formation of secondary or supergene Nb deposits, often by an enrichment factor of 10 times of more, through residual accumulation of pyrochlore group minerals in the overlying laterites (Mitchell, 2015), forming some of the world's largest Nb deposits, e.g., the Araxá deposit in Brazil (Issa Filho et al., 2013; Traversa et al., 2001), and the Lueshe deposit in Democratic Republic of Congo (DRC; Maravic et al., 1989; Wall et al., 1996). Primary Nb hosts often are pyrochlore, perovskite group minerals, niobates, and Nb-bearing silicates (i.e., titanite), as well as Ti-oxides (rutile, brookite) and ilmenite (Cerný and Ercit, 1989; Mitchell, 2015; Bollaert et al., 2023b) that could contain minor amounts

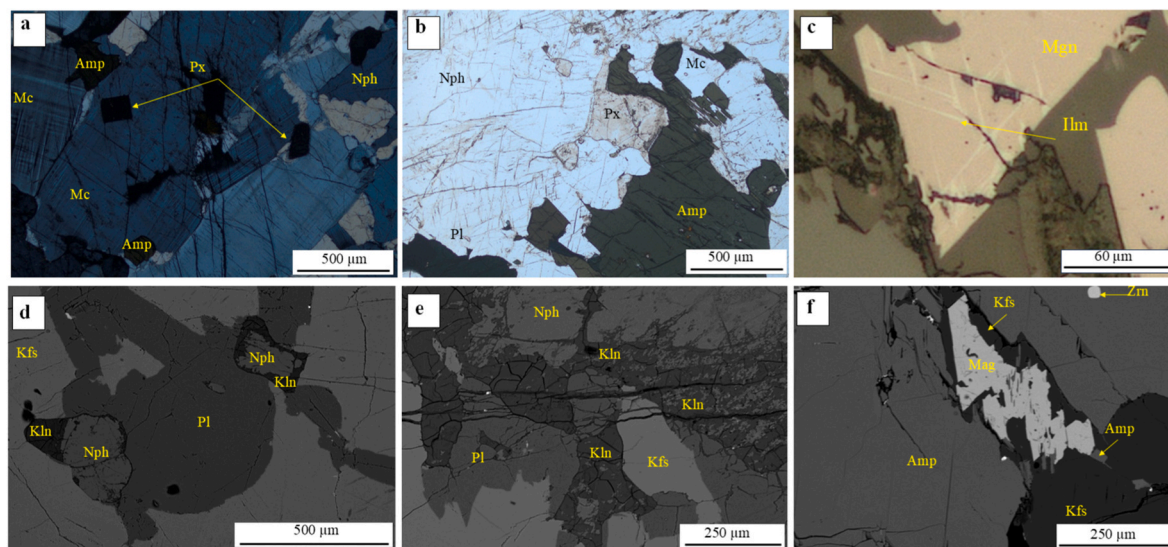


Fig. 3. (a)–(c) Photomicrograms and (d)–(f) SEM-BSE images of the parent rock: (a) Amphibole and pyroxene inclusions in K-feldspar; (b) Euhedral amphibole associated with nepheline, K-feldspar, plagioclase and pyroxene; (c) Magnetite with ilmenite exsolution; (d) Fractured nepheline with kaolinite/halloysite coating; (e) Nepheline, K-feldspar and plagioclase with kaolinite/halloysite filling cracks; and (f) Magnetite, K-feldspar, and zircon inclusions in amphibole. (a) is taken under cross polarizing light; (b) under plane polarizing light; and (c) under reflected light. Abbreviations: Amp: amphibole; Kfs: K-feldspar; Kln: Kaolinite; Mc: microcline; Nph: nepheline; Pl: plagioclase; Px: pyroxene; Zrn: zircon.

Table 1

Semi-quantitative Analyses (EDS) of minerals in the parent rock (EB2R).

	Nepheline		Plagioclase		K-feldspar		Kaolinite		Amphibole		Magnetite		Zircon*		Pyrochlore*	
	wt. %															
SiO ₂	42.55	41.76	68.54	68.84	64.82	64.92	52.50	45.46	36.47	36.62	1.45	0.21	31.72	32.02	5.59	7.77
Na ₂ O	15.02	15.19	11.00	10.99	0.57	0.47			4.08	4.19					5.59	7.64
MgO									0.40							
Al ₂ O ₃	35.01	34.60	20.16	20.27	19.03	18.88	39.24	45.20	14.04	14.19	1.49					7.41
K ₂ O	6.70	6.70			15.80	15.96	0.39	0.40	2.72	2.69	0.08					1.40
CaO		0.27					0.46		6.49	6.56					19.52	13.88
TiO ₂									1.28	1.30	4.44	3.87			7.34	5.09
V ₂ O ₅						0.41										
MnO									1.77	1.66	0.81					
Fe ₂ O ₃									32.37	32.98	91.29	94.05	0.81			
Y ₂ O ₃																
ZrO ₂													65.55	66.05		
Nb ₂ O ₅															59.21	44.42
HfO ₂													1.40	1.72		
Ta ₂ O ₅															1.12	0.78
UO ₂															1.71	1.12
Cl									0.07							
F															5.59	4.07
Total	99.28	98.51	99.70	100.10	100.22	100.64	92.59	91.05	99.70	100.20	99.56	98.13	99.50	99.80	105.68	93.57

Element concentrations are in wt. %. * Quantitative EPMA analyses in Tables 2 and 5

of Nb. However, under prolonged lateritization processes, these minerals, particularly pyrochlore which is presumably resistant, could decompose to Ti-oxides (Bollaert et al., 2023a, 2023b), Nb-bearing hydroxides (Mariano, 1989) and Fe-hydroxides (goethite; Wall et al., 1996). This calls for more detailed investigations down to a mineral scale to study the Nb behaviour during tropical weathering.

This study focuses on weathered materials of a Nb-REE-bearing nepheline syenite at Eboudja in Southwest Cameroon. The aim is to characterize petrographic and mineralogical changes during weathering of the nepheline syenite for the mobilization and enrichment of Nb and other trace elements under tropical climatic conditions.

2. Geological and geographic setting

The investigated regolith profile is located on a hillslope at an altitude of approximately 80 m a.s.l. in Eboudja, Southwest Cameroon, Central Africa (Fig. 1). The locality is characterized by an annual rainfall of 2900 mm and a mean annual temperature of 26 °C, indicative of an equatorial climate. The vegetation consists of evergreen equatorial forest, and the soils in the study area are classified as ferralsols (Temga et al., 2021). This profile developed over the Eboudja nepheline syenite intrusion (Fig. 1c) through intense chemical weathering. Previous studies indicate that the Eboudja syenitic intrusion is Pan-African in age, dated at approximately 590 ± 19 Ma (Lerouge et al., 2006; Nsifa

Table 2

Quantitative analyses (EPMA) of Zircon in parent rock (EB2R).

	EB2R				
	n. 26				
	Average	Max	Min	Median	SD
Na ₂ O	0.02	0.10	0.00	0.00	0.03
Pr ₂ O ₃	0.05	0.21	0.00	0.00	0.06
Cl	0.00	0.02	0.00	0.00	0.01
MgO	0.01	0.05	0.00	0.01	0.02
ZrO ₂	64.17	65.43	62.51	64.19	0.75
Nd ₂ O ₃	0.04	0.19	0.00	0.00	0.06
ThO ₂	0.03	0.20	0.00	0.01	0.05
Al ₂ O ₃	0.07	0.43	0.00	0.01	0.11
Nb ₂ O ₅	0.00	0.00	0.00	0.00	0.00
Sm ₂ O ₃	0.03	0.13	0.00	0.00	0.04
UO ₂	0.11	0.43	0.00	0.08	0.12
SiO ₂	33.54	34.12	32.95	33.49	0.32
TiO ₂	0.03	0.09	0.00	0.03	0.03
MnO	0.01	0.09	0.00	0.00	0.02
CaO	0.08	0.40	0.00	0.04	0.10
Y ₂ O ₃	0.02	0.14	0.00	0.00	0.04
La ₂ O ₃	0.05	0.17	0.00	0.04	0.06
Gd ₂ O ₃	0.03	0.19	0.00	0.00	0.06
BaO	0.02	0.09	0.00	0.01	0.03
P ₂ O ₅	0.20	0.30	0.08	0.19	0.06
Na ₂ O	0.13	0.42	0.00	0.10	0.12
Pr ₂ O ₃	0.14	0.90	0.00	0.11	0.18
Cl	0.03	0.14	0.00	0.00	0.04
MgO	0.03	0.17	0.00	0.00	0.05
ZrO ₂	0.05	0.17	0.00	0.02	0.06
Nd ₂ O ₃	1.11	1.51	0.79	1.13	0.22
ThO ₂	0.00	0.00	0.00	0.00	0.00
Al ₂ O ₃	0.00	0.00	0.00	0.00	0.00
Nb ₂ O ₅	100.00				

Element concentrations are in wt. %. The values were normalised at 100 %.

et al., 2013). It outcrops as a discontinuous band oriented NNE–SSW and extends approximately 10 km along its longest axis (Noel and Roubichou, 1986; Nsifa et al., 2013). The Eboundja syenite belongs to a group of undersaturated syenitic plutons found along the southern Atlantic coast of Cameroon, including the Rocher du Loup and Mont des Éléphants massifs (Fig. 1b; Lerouge et al., 2006; Nsifa et al., 2013; Delor et al., 2021). These plutons intruded into the contact zone between Neoproterozoic metasedimentary rocks (quartzites and micaschists) and Archean to Paleoproterozoic orthogneisses of the Nyong Bayomen Domain (Fig. 1c; Owona et al., 2021; Delor et al., 2021; Toteu et al., 2022; Vic et al., 2025).

Situated at the northwestern margin of the Congo Craton (Ntem Complex), this domain comprises parts of the Archean Congo craton that have repeatedly undergone high-grade metamorphism, structural deformation, and north-eastward transposition during both the Eburnean (~2400–1750 Ma; Feybesse et al., 1998; Barbosa and Barbosa, 2017; Owona et al., 2021) and Pan-African (~660–540 Ma; Toteu et al., 2006; Li et al., 2017; Caxito et al., 2020; Vic et al., 2025) orogenies. These older basement rocks are associated with the Neoproterozoic supracrustal volcano-sedimentary cover of the Ndikinimeki Group, which has been intensely deformed, metamorphosed, and thrust toward the south-southeast during the Pan-African orogeny (Tchakounté et al., 2017; Delor et al., 2021; Vic et al., 2025). The emplacement of nepheline syenites in southwestern Cameroon is contemporaneous with sinistral movement along the Kribi-Campo shear zone (also known as the South-West Cameroon Shear Zone; Lerouge et al., 2006; Nsifa et al., 2013). The ongoing transcurrent deformation along this shear zone contributed to the development of fabrics, like foliation and boudinage, in the nepheline syenites (Nsifa et al., 2013).

3. Methods

Progressive weathering of the Eboundja nepheline syenite has

resulted in the formation of a thick weathering crust. The studied regolith is about 10.5 m thick. This profile was observed and described through a manually excavated pit. According to their physical properties, 3 horizons can be distinguished on the profile (Fig. 2). These are from bottom to top:

- Saprolite or C horizon (3.7–10.6 m): red (10R 5/6) to reddish brown (2.5 YR 4/6); sandy loam; friable;
- B horizon (1.2–3.7 m): yellowish red (5 YR 5/8) to reddish yellow (5 YR 6/8); loamy clayey to clayey; diffuse irregular boundary; and
- A horizon (0–1.2 m): yellowish red (5 YR 5/8); clayey; granular structure; diffuse irregular boundary.

The A and B horizons are grouped into the ‘pedolith’, which is the upper part of the regolith that has been subjected to soil-forming processes resulting in the loss of the fabric of the parent rock (Taylor and Eggerton, 2001). The boundary between saprolite and pedolith corresponds to the disappearance of the lithic fabric. Weathered nepheline block of various degree of weathering are present along the profile. Their size varies from few centimetres to about 50 cm. Smaller blocks are found in the A horizon. One slightly weathered bedrock sample (EB2R) and five undisturbed weathered samples (EB2C1, EB2C5, EB2C7, EB21, and EB20) were collected from various horizons of the regolith profile. Samples EB2C1, EB2C5, and EB2C7 were taken from the C horizon; EB21 from the B horizon; and EB20 from the A horizon. For microscopic analyses, polished thin sections were prepared. Weathered samples were first impregnated with resin before thin sectioning. The thin sections were then examined and described using an optical microscopy, scanning electron microscopy (SEM) and electron probe micro analysis (EPMA).

Scanning electron microscopy (SEM) analyses were carried out at Earth Science Department (DST) of the University of Turin (Italy), using a JEOL JSM IT300LV (High Vacuum - Low vacuum 10/650 P- 0.3–30 kV) scanning electron microscope, equipped with an Oxford INCA Energy 200 energy dispersive spectrometer (EDS) and an INCA X-act SDD thin window detector. The analyses were performed at a 20 kV voltage, a 5 nA beam current and a 10 mm working distance (WD), and the EDS spectra were acquired using a time of 30 s and a dead time of 30 %, that corresponds to ~1,000,000 cps. Calibration was performed every 30 min against a Co standard. Element maps were scanned using the same working voltage, beam current and WD at 1 h/frame at a 2048-pixel resolution. Data acquired were processed using the Aztec 6.0 software (Oxford Instrument).

Selected areas were further examined and imaged with a TESCAN VEGA microscope at the DST of the University of Turin, which is equipped with an Explora 30 mm² EDS (Oxford Instrument) and a cathodoluminescence (CL) panchromatic black and white detector covering the wavelength of 300–800 nm. The working conditions for EDS analyses were 15 kV voltage, a 15 mm WD, and a 300 pA beam current, whereas CL analyses were carried out with an electron beam at an energy of 15 kV and a current of 1 nA at a 15 mm WD.

Pyrochlore, zircon, ilmenite and Ti-oxides were further analysed by EPMA to understand the Nb mobilization at mineral scale. The analyses were carried out at the Earth Science Department “Ardito Desio”, University of Milan. The analyses were run using a JEOL 8200 Super Probe equipped with five wavelength-dispersive spectrometers. The analytical conditions were an accelerating voltage of 15 kV, a beam current of 5 nA, a beam diameter of 1–2 µm, and a dwell time of 20 s for each element. Element maps were rastered using a 15 kV acceleration voltage, a probe current of 5 nA and a dwell time of 20 ms for each element. The following standards were used for calibration: omphacite (Na); grossular (Al, Si, Ca); fayalite (Fe); rodonite (Mn); olivine (Mg); ilmenite (Ti); fluorapatite (P); scapolite (Cl); Celestine (Sr); Zircon (Zr, Hf); ThO₂ (Th); UO₂ (U); Nb (Nb); Ta (Ta); Sanbornite (Ba); REE Phosphates (for corresponding REE).

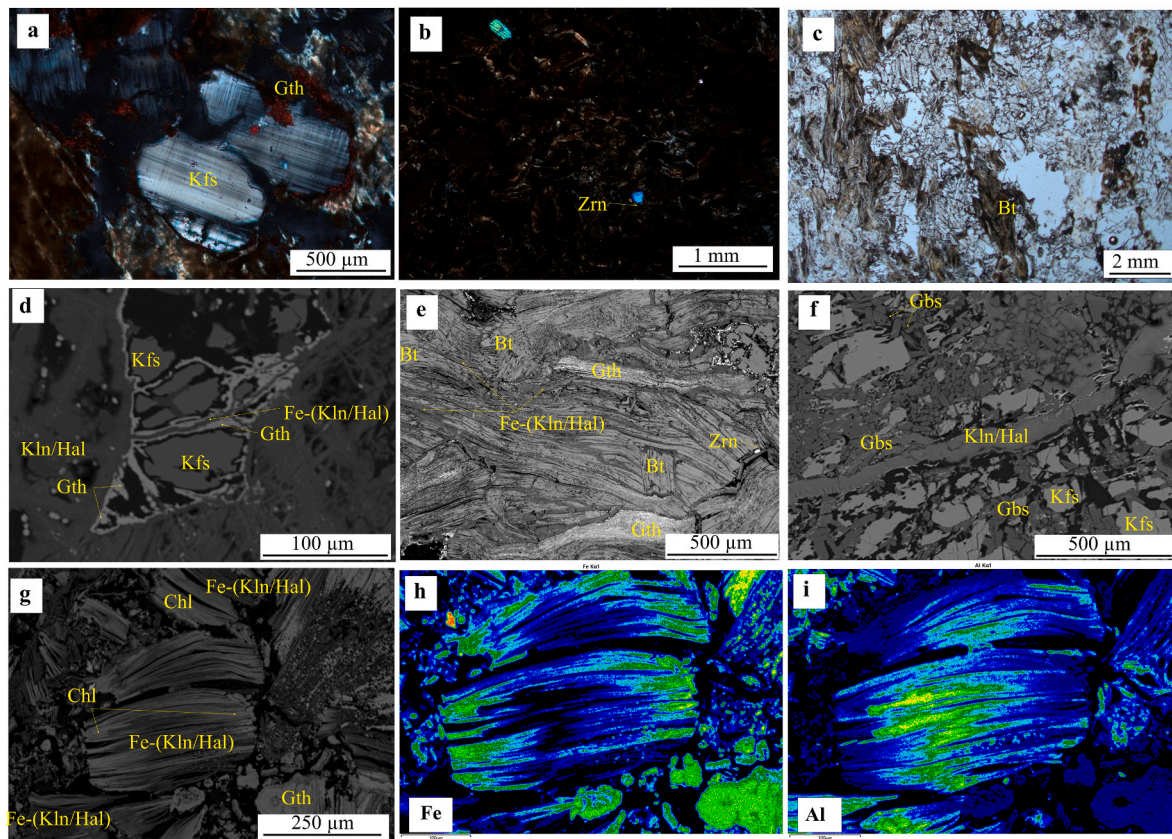


Fig. 4. (a)–(c) Photomicrograms and (d)–(g) SEM-BSE images of the saprolite, and (h)–(i) SEM-EDS element maps of (g): (a) K-feldspar relics surrounded by Fe-(hydr)oxides in clay matrix; (b) Zircon relic in clay + Fe-(hydr)oxides matrix; (c) Biotite relic in clay + Fe-(hydr)oxides matrix; (d) K-feldspar with goethite coating; (e) Weathered K-feldspar with kaolinite/halloysite and gibbsite; and (g–i) Chlorite weathering to Fe-kaolinite. (a) and (b) are taken under cross polarizing light and (c) under plane polarizing light. Abbreviations: Bt: biotite; Chl: chlorite; Gbs: gibbsite; Gth: goethite; Kfs: K-feldspar; Kln/Hal: kaolinite/halloysite; Ms: muscovite; Zrn: zircon.

4. Results

4.1. Petrography and mineral chemistry of bedrock and regolith

- **Bedrock.** EB2R is a slightly weathered nepheline syenite. The sample exhibits a heterogranoblastic texture, with grain sizes ranging from 0.1 to 8 mm. The primary minerals include microcline, nepheline, plagioclase, and amphibole, with minor amounts of pyroxene. Zircon, magnetite with ilmenite exsolution (Fig. 3c), allanite, pyrochlore and biotite are the accessory phases (Fig. 3, Table 1). Euhedral to subhedral microcline have size varying from 0.2 to 3 mm, smaller microcline grains are found as inclusion in amphibole grains (Fig. 3b). Amphibole occurs as euhedral to subhedral grains of which the size varies from 0.3 to 1.5 mm (Fig. 3a and b). Allanite (0.1–0.3 mm) occurs as zoned, sub-to euhedral grains and often as inclusions in amphibole and plagioclase. Zircon grains are euhedral, and their sizes range from 0.1 to 0.4 mm (Fig. 3f). Inclusions of zircon are found in amphibole. EPMA results on zircon grains shows a minor content of Hf (0.79–1.51 wt % HfO₂), U (b.d.l. – 0.43 wt % UO₂) and Fe (b.d.l. – 0.9 wt % FeO) (Table 2). Pyrochlore grains of size of ~0.1 mm often occur as inclusions in amphibole and K-feldspar. This sample is largely free of weathering and supergene minerals, with minor occurrence of kaolinite along grain boundaries and along cracks in nepheline, K-feldspar and plagioclase grains (Fig. 3d and e).
- **Saprolite.** Saprolite specimens still generally show the relict texture of the syenite bedrock but also a partial decomposition of the primary minerals. Relict K-feldspar (microcline) and zircon are visible in

EB2C1 and EB2C7. In addition, plagioclase is barely observed in sample EB2C1; and weathered biotite is observed in sample EB2C7. However, a mesh network of kaolinite developed by partial weathering of feldspars, particularly along the grain boundary (Fig. 4d and f), indicating the process of weathering towards interior of the mineral grains from the outer micro-fractures. From SEM-BSE imaging, a thin coat of goethite is also observed rimming the weathered K-feldspar grains (Fig. 4d). Occurrence of biotite is restricted to sample EB2C7. They appear as clusters of aggregates with a preferential orientation. Their chemical compositions are non-stoichiometric, likely reflecting annite weathering to chlorite (Table S3). Most biotite grains are surrounded by a coating of Fe-rich kaolinite/halloysite, and have goethite precipitates along cleavages (Fig. 4c and e). Relict zircon grains with size varying from 75 to 250 μm are also observed (Fig. 4b), and sometimes as inclusions in the relict biotite grains.

The matrix is made up of kaolinite, hematite, goethite, Fe-Ti-oxides, gibbsite, and chlorite (Fig. 4, Table 3). EDS analyses highlighted Fe-rich kaolinite/halloysite (here after Fe-kaolinite), especially in samples EB2C5, EB2C7, where the Fe content varies from 3.5 to 25 wt % (Table 3). Minor amounts of Ti are frequently detected (Table 2). Gibbsite is observed only in the sample EB2C1. They are in micrometric baguettes shape and present in cluster spatially associated with kaolinite/halloysite and K-feldspar (Fig. 4f), with minor amounts of Fe (varying from 0.17 to 0.34 wt %; Table 2). Chlorite are detected in sample EB2C5 (Fig. 4g–Table 3), with progressive weathering from the

Table 3
Quantitative analyses (EPMA) on Ti- and Fe-Ti-oxides in weathered samples.

	EB2C5-A1-1	EB2C5-A1-2	EB2C5-A1-3	EB2C5-A1-4	EB2C5-A1-5	EB2C5-A4-1	EB2C5-A4-2	EB2C5-A4-3	EB2C5-A4-4	EB2C5-A4-5	EB2C5-A2-1	EB2C5-A2-2	EB2C5-A2-3	EB2C5-A2-4	EB2C5-A2-5
	Altered Ilmenite	Altered Ilmenite	Altered Ilmenite	Altered Ilmenite	Altered Ilmenite	Ilmenite	Ilmenite	Rutile	Altered Ilmenite	Altered Ilmenite	Ilmenite	Altered Ilmenite	Altered Ilmenite	Altered Ilmenite	Altered Ilmenite
	Aanalisi 1	Aanalisi 2	Aanalisi 3	Aanalisi 4	Aanalisi 5	Aanalisi 6	Aanalisi 7	Aanalisi 8	Aanalisi 9	Aanalisi 10	Aanalisi 11	Aanalisi 12	Aanalisi 13	Aanalisi 14	Aanalisi 15
Na ₂ O	0.14	0.04	0.04	0.03	0.06	0.07	0.11	0.12	0.02	0.03	0.06	0.01	0.04	0.00	0.04
SrO	0.00	0.11	0.00	0.00	0.00	0.00	0.18	0.09	0.00	0.09	0.00	0.00	0.15	0.11	0.09
Pr ₂ O ₃	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Cl	0.01	0.03	0.00	0.01	0.02	0.00	0.02	0.01	0.01	0.02	0.02	0.00	0.00	0.00	0.02
MgO	0.01	0.01	0.00	0.04	0.01	0.06	0.04	0.07	0.02	0.02	0.04	0.05	0.02	0.08	0.01
ZrO ₂	0.12	0.31	0.19	0.31	0.31	0.00	0.04	0.19	0.25	0.12	0.00	0.19	0.10	0.26	0.22
Nd ₂ O ₃	0.00	0.00	0.03	0.01	0.08	0.00	0.12	0.02	0.00	0.01	0.07	0.10	0.06	0.08	0.00
ThO ₂	0.04	0.03	0.00	0.01	0.03	0.05	0.00	0.05	0.00	0.00	0.00	0.00	0.05	0.03	0.03
Al ₂ O ₃	3.71	3.73	3.14	3.47	2.34	2.39	0.00	0.29	3.09	3.04	0.14	3.62	3.77	3.38	3.53
Nb ₂ O ₅	1.62	1.40	1.04	0.79	0.75	0.41	0.69	1.22	1.01	0.63	0.57	0.62	1.10	0.78	0.95
Sm ₂ O ₃	0.02	0.00	0.00	0.04	0.09	0.00	0.00	0.00	0.09	0.00	0.12	0.10	0.00	0.05	0.00
UO ₂	0.07	0.04	0.02	0.05	0.14	0.07	0.03	0.05	0.02	0.08	0.00	0.07	0.16	0.07	0.00
SiO ₂	0.89	0.81	1.00	1.03	1.13	2.71	0.01	0.63	1.00	1.54	0.10	0.79	1.48	0.84	1.18
TiO ₂	75.16	78.07	79.33	73.98	80.11	48.53	52.32	95.35	77.93	77.74	50.52	78.49	76.30	76.65	77.41
MnO	0.00	0.02	0.00	0.01	0.00	3.08	2.72	0.04	0.00	0.05	6.52	0.00	0.03	0.00	0.02
CaO	0.06	0.28	0.11	0.28	0.32	0.03	0.01	0.10	0.05	0.19	0.04	0.25	0.20	0.18	0.56
Y ₂ O ₃	0.00	0.00	0.01	0.00	0.07	0.00	0.00	0.03	0.04	0.03	0.00	0.03	0.00	0.00	0.00
La ₂ O ₃	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Gd ₂ O ₃	0.00	0.00	0.04	0.13	0.01	0.00	0.12	0.00	0.02	0.00	0.09	0.06	0.03	0.00	0.00
BaO	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
P ₂ O ₅	1.88	1.41	1.39	1.56	0.84	0.06	0.00	0.01	1.45	0.98	0.00	1.41	1.59	1.41	1.20
Ce ₂ O ₃	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
FeO	10.00	9.16	8.11	12.47	9.11	40.85	43.58	0.75	9.55	8.91	39.85	9.37	10.41	10.83	9.05
Dy ₂ O ₃	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Er ₂ O ₃	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Yb ₂ O ₃	0.00	0.02	0.00	0.00	0.00	0.00	0.00	0.00	0.02	0.02	0.15	0.00	0.00	0.00	0.00
HfO ₂	0.00	0.01	0.00	0.17	0.06	0.13	0.24	0.19	0.26	0.11	0.00	0.00	0.03	0.21	0.00
Ta ₂ O ₅	0.07	0.17	0.00	0.18	0.05	0.06	0.20	0.00	0.28	0.00	0.00	0.00	0.01	0.28	0.13
F	0.00	0.00	0.03	0.03	0.00	0.00	0.09	0.00	0.00	0.00	0.03	0.00	0.00	0.00	0.00
Total:	93.80	95.64	94.48	94.60	95.53	98.51	100.50	99.22	95.11	93.61	98.32	95.17	95.53	95.24	94.44
<i>apfu (2 Oxygens)</i>															
Na	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Sr	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Pr	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Mg	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Zr	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Nd	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Th	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Al	0.06	0.06	0.05	0.06	0.04	0.05	0.00	0.00	0.05	0.05	0.00	0.06	0.06	0.06	0.06
Nb	0.01	0.01	0.01	0.01	0.00	0.00	0.01	0.01	0.01	0.00	0.00	0.00	0.01	0.01	0.01
Sm	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
U	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Si	0.01	0.01	0.01	0.02	0.02	0.05	0.00	0.01	0.01	0.02	0.00	0.01	0.02	0.01	0.02
Ti	0.83	0.85	0.86	0.82	0.87	0.61	0.66	0.97	0.85	0.86	0.65	0.85	0.83	0.84	0.85
Mn	0.00	0.00	0.00	0.00	0.00	0.04	0.04	0.00	0.00	0.00	0.09	0.00	0.00	0.00	0.00
Ca	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.01
Y	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
La	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00

(continued on next page)

Table 3 (continued)

	EB2C5-A1-1	EB2C5-A1-2	EB2C5-A1-3	EB2C5-A1-4	EB2C5-A1-5	EB2C5-A1-6	EB2C5-A1-7	EB2C5-A1-8	EB2C5-A1-9	EB2C5-A1-10	EB2C5-A1-11	EB2C5-A2-12	EB2C5-A2-13	EB2C5-A2-14	EB2C5-A2-15
	Altered Ilmenite	Altered Ilmenite	Altered Ilmenite	Altered Ilmenite	Altered Ilmenite	Altered Ilmenite	Altered Ilmenite	Rutile	Altered Ilmenite	Altered Ilmenite	Altered Ilmenite	Altered Ilmenite	Altered Ilmenite	Altered Ilmenite	Altered Ilmenite
	Analisi 1	Analisi 2	Analisi 3	Analisi 4	Analisi 5	Analisi 6	Analisi 7	Analisi 8	Analisi 9	Analisi 10	Analisi 11	Analisi 12	Analisi 13	Analisi 14	Analisi 15
Gd	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Ba	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
P	0.02	0.02	0.02	0.02	0.01	0.00	0.00	0.00	0.00	0.00	0.00	0.02	0.02	0.02	0.01
Ce	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Fe	0.12	0.11	0.10	0.15	0.11	0.57	0.61	0.01	0.12	0.11	0.57	0.11	0.13	0.13	0.11
Dy	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Er	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Yb	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Hf	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Ta	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Fe ₂ +	0.12	0.11	0.10	0.15	0.11	0.57	0.61	0.01	0.12	0.11	0.57	0.11	0.13	0.13	0.11
Fe ₃ +	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
*Fe ₃ +/Fe ₂ +	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Fe/Ti	0.15	0.13	0.11	0.19	0.13	0.94	0.93	0.01	0.14	0.13	0.88	0.13	0.15	0.16	0.13

Element concentrations are in wt. %. * Calculated on the basis of stoichiometric formula.

core to the rim of the mineral grains, and associated by a decreasing trend in Fe content but an increasing trend in Si and Al contents. Weathered chlorite is often spatially associated with Fe-kaolinite (Fig. 4g, h and i; Table S3). Titanium-bearing minerals include ilmenite (Fe/Ti apfu ratio of ~0.01), Ti-oxides (Fe/Ti apfu ratio of ~1) and altered ilmenite (pseudorutile and leucoxene with a Fe/Ti apfu ratio of ~0.12) (Table 3). They are observed in all saprolite samples but mostly in EB2C7 and EB2C5. Altered ilmenites (pseudorutile and leucoxene) and Ti-oxide are the weathering products of ilmenite. Leucoxene, in a skeletal form with a granular outline (Fig. 6c), represents the main Ti-bearing phases in the saprolite. They are sub-rounded, highly fractured and in association with ilmenite, pseudorutile, and hematite. These grains, in the sizes of 50–250 μm , are often observed in the matrix of clay minerals. Identified Fe-(oxy)hydroxides minerals include goethite and hematite. Hematite is scarcer and only observed in sample EB2C5. They are subhedral crystals with sizes of less than 50 μm . In optical microscope, goethite form clusters of micrometric red rounded patches. They have high contents of Al and Si (generally of ~2–9 wt % both for Al_2O_3 and SiO_2), and lower P and Ti contents (Table 3).

-Pedolith. In the pedolith, only zircon and ilmenite are detected as the relict primary minerals (Fig. 5, Table 4). Zircon grains appear fractured and smaller in size (usually less than 40 μm). The pedolith is primarily composed of fine-grained Fe-(oxy)hydroxides and intergrowths of clay minerals (Fig. 5a and f). The kaolinite/halloysite phases are notably enriched in Fe, with Fe concentrations ranging from 5 to 25 wt %, and contain minor amounts of Ti (Fig. 5b–e, f, and g; Table 4). Hematite is observed exclusively in sample EB20, with grain sizes ranging from 40 to 125 μm (Fig. 5c). Fe-Ti-oxides (ilmenite, pseudorutile and leucoxene) are only identified in sample EB21 where leucoxene is the main Fe-Ti-oxides (Fig. 5f).

4.2. Characterization of the niobium carriers

The main Nb-bearing mineral in the rock sample (EB2R) is pyrochlore (Nb_2O_5 content of ~55–77 wt %; Table 5). The mineral grains show internal zonation (Fig. 6a and b), as expressed by differences in chemical composition between the core and the rim of the mineral grain (Table 5). The core has higher average CaO (~19 wt %), Na_2O (~6 wt %), SrO (~0.25 wt %), F (~5 wt %) contents and lower average Nb_2O_5 (~57 wt %), TiO_2 (~8 wt %) contents than the rim (CaO of ~4 wt %; Na_2O of ~1 wt %; SrO of ~0.06 wt %; F below detection limit; Nb_2O_5 of ~74 wt %; TiO_2 of ~11 wt %), though an outlier of 4.67 wt % SrO occurs. The Ce_2O_3 content is low very (<0.5 wt % on average in both the core and the rim). Three variants of pyrochlore can be classified: Ca-Na-F-rich pyrochlore, Ca-Na-F-poor pyrochlore, and Sr-rich pyrochlore (Fig. 7); Ca-Na-F-rich pyrochlore may correspond to the fresh pyrochlore, while Ca-Na-F-poor pyrochlore and Sr-rich pyrochlore represent products from alteration (Palmieri et al., 2022; Bollaert et al., 2023b). Binary plots show positive correlations between F, Na_2O and CaO; negative correlation between CaO and Nb_2O_5 ; and no correlation between CaO, SrO and Ce_2O_3 (Fig. 7). The pyrochlore grains often show micro-fracturation along grain boundaries and no pyrochlore was observed in the weathered samples.

Titanium-bearing oxides (ilmenite, pseudorutile and leucoxene) are instead identified as the main Nb carriers in the regolith (Fig. 6c). Nb_2O_5 contents in ilmenite, altered ilmenite and Ti-oxides are ~1 wt %, ~1 to ~2 wt %, and ~1 wt %, respectively (Table 3). Altered ilmenite grains are additionally characterised by low totals compare to ilmenite and Ti-oxides. The binary plots highlight a positive correlation between Nb_2O_5 and TiO_2 contents for ilmenite (Fig. 8). However, the abovementioned positive Nb_2O_5 - TiO_2 trend is not applicable to altered ilmenite, while its Na_2O content show a more causatively positive correlation with its Nb_2O_5 content. Minor contents of SiO_2 and Al_2O_3 are ubiquitous in the Ti-bearing oxide minerals with a range of b.d.l. to ~3 wt % for SiO_2 and of b.d.l. to ~4 wt % for Al_2O_3 . Lowest Al_2O_3 content occurs in ilmenite whereas the highest in altered ilmenite (see Fig. 8).

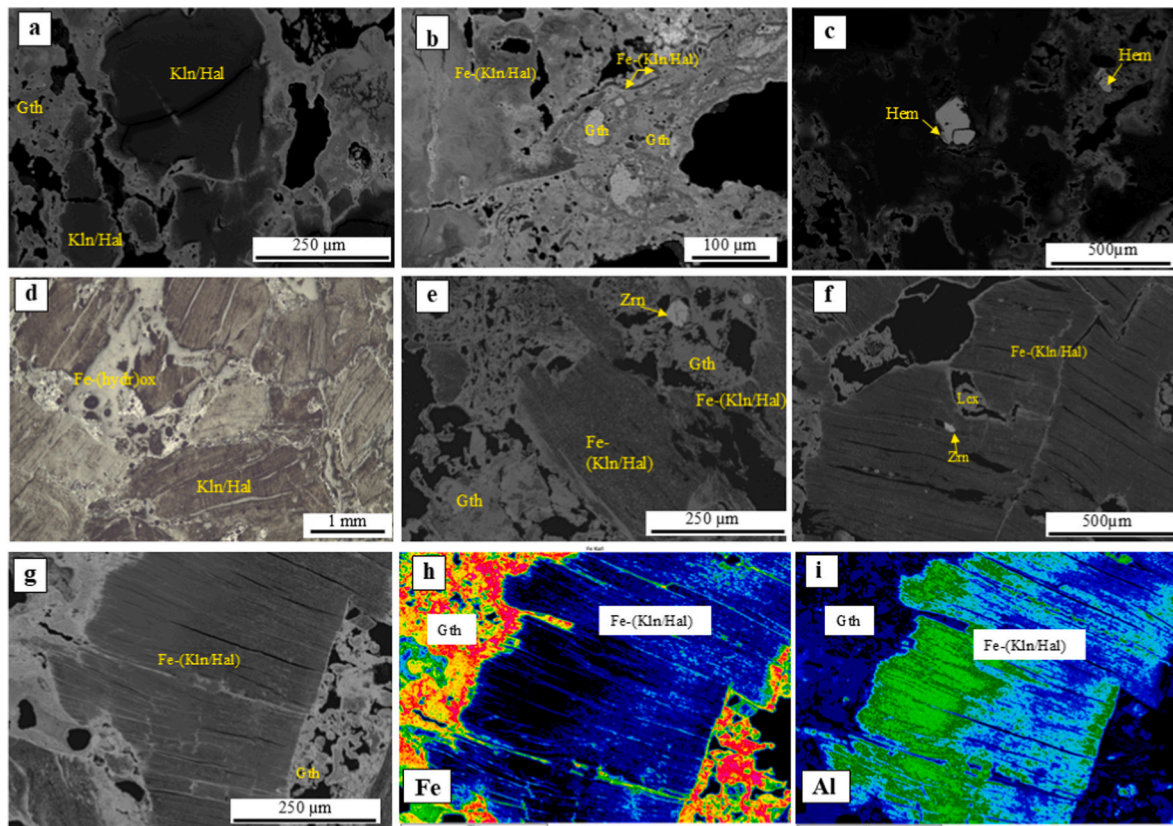


Fig. 5. (a)–(g) SEM-BSE images of the pedolith and (h)–(i) SEM-EDS element maps of (g): (a–b) Fe-(hydr)oxides crusts + kaolinite/halloysite (EB20); (c) Hematite in weathered material (EB20); (d) Fe-(hydr)oxides crusts + kaolinite/halloysite (EB21); (e) Relic zircon in clay + Fe-(hydr)oxides matrix (EB21); (f) Leucoxene and zircon inclusion in Fe-kaolinite/halloysite (EB21); and (g–i) Fe-kaolinite/halloysite in a Fe-hydroxides matrix (EB21). Abbreviations: Lcx: leucoxene, other abbreviations are same as those in previous figures.

5. Discussion

5.1. Behaviours of primary minerals during weathering

Textural, petrographical, and geochemical features of the Eboundja regoliths show that micro-fracturing, dissolution, and chemical replacement play a key role in the mineral-scale weathering processes. Dissolution enhanced by micro-fracturing is the main mechanism for weathering of K-feldspar and nepheline. Similar processes were reported in studies of igneous rocks weathering under tropical conditions (Braun, 1991; Navarre-Sitchler et al., 2013). Other studies also highlighted the importance of micro-fracturing in enhancing weathering process through creating pathways for acidic fluids to percolate and increasing surface areas for dissolution (Graham et al., 2010; Hampl et al., 2023; Navarre-Sitchler et al., 2013). In this study, kaolinite/halloysite are the weathering products following dissolution of K-feldspar in the micro-fractures. However, previous works show that precipitation of secondary minerals (e.g., clay minerals) in pore spaces could also retard weathering by hindering percolation of the soil solutions (Braun, 1991; Hampl et al., 2023). This inhibition process may be balanced by crystallization of goethite along primary minerals grains (e.g., goethite coating on K-feldspar), as stress due to continuous growth of Fe coating on mineral surface may initiate micro-fracturing (Anovitz et al., 2021; Hampl et al., 2023). Furthermore, Fe-(oxy)hydroxides precipitation leads to acidification, favorable to silicate dissolution (Bilong, 1988; Braun, 1991; Hampl et al., 2023). On the other hand, illite, as a common weathering product of K-feldspar in syenites (e.g., Wang et al., 2024), is not observed in this study. It is likely that the lower pH and higher water-to-regolith ratio under a humid climate at Eboundja would rapidly remove K during K-feldspar weathering and disfavor the

neof ormation of illite but kaolinite.

Chlorite has undergone weathering through clay transformation reactions, leading to Fe-rich kaolinite (Fig. 9) as the resultant weathering product (Xu and Veblen, 1996; Jin et al., 2010). Previous studies in temperate and sub-tropical climates have demonstrated that chlorite weathering generally occurs through a series of clay transformation reactions: Chlorite – vermiculite - hydroxy interlayered vermiculite - kaolinite/halloysite + Fe-(oxy)hydroxides (Wilson, 2004; Jin et al., 2010; Li and Zhou, 2023). However, the more intense weathering and more acidic conditions that prevailed under tropical climates would accelerate the decomposition of the intermediate phases to kaolinite, which prevails in the studied saprolite and pedolith samples (Ojanuga, 1973; Ahn and Peacor, 1987; Dong et al., 1998; Tan et al., 2017; Cheng et al., 2022). Similarly, weathering of Fe-rich biotite, i.e., annite, and amphibole forms kaolinite/halloysite and Fe-(oxy)hydroxides (Fig. 9) as final products of intense weathering without the presence of any intermediate phases (Ojanuga, 1973; Bisdom et al., 1982). Weathering of chlorite to Fe-kaolinite/halloysite is indicative of an oxidative condition where ferric iron was incorporated into the octahedral sheet of kaolinite (Nguyen-Thanh et al., 2017). During annite weathering, Fe-(oxy)hydroxides result from the oxidation of Fe^{2+} to Fe^{3+} while kaolinite/halloysite may result from dehydroxylation (lost of hydroxyl groups OH^-) and cation loss processes (Bisdom et al., 1982). The transformation of biotite to kaolinite/halloysite can also be achieved through either reaction of one layer of altered biotite to form two layers of kaolinite (Ahn and Peacor, 1987; Dong et al., 1998) or transition of one layer of altered biotite to one layer of halloysite, which subsequently reacted to form one layer of kaolinite (Dong et al., 1998). The release of $\text{Ti}(\text{OH})_4$ from annite weathering may contribute to Ti-oxide formation (Fig. 9) through the precipitation of $\text{Ti}(\text{OH})_4$ to form an amorphous

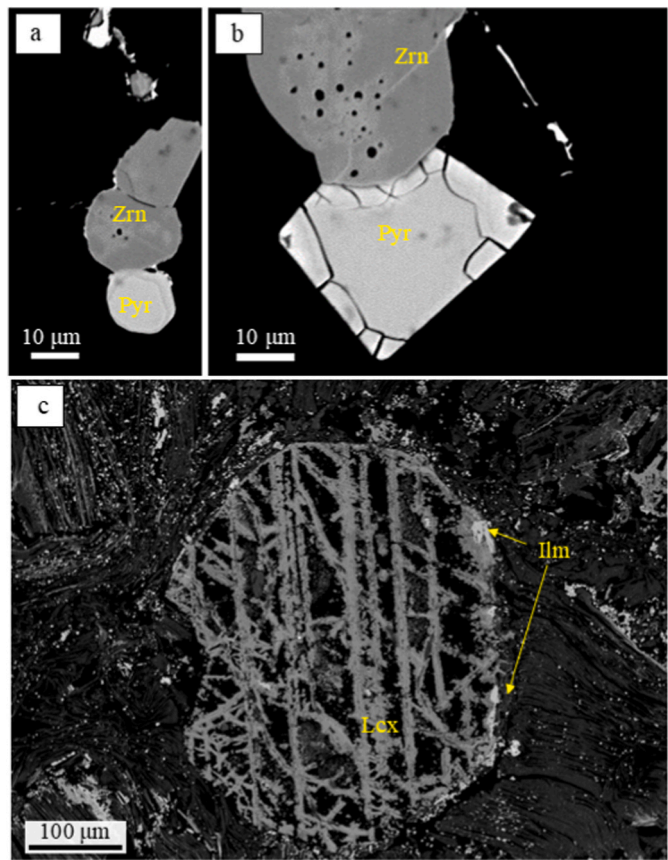


Fig. 6. SEM-BSE images of (a) zoned and (b) fractured pyrochlore with zircon in parent rock (EB2R); and (c) Occurrence of ilmenite and leucoxene in the saprolite (sample EB2C5).

hydrous oxide gel, and its subsequent dehydration to form a granular aggregate of small anatase crystals (Craig and Loughan, 1964; Bisdorn et al., 1982).

The presence of zircon in all samples can be explained by its relative resistance to chemical weathering (Finch and Hanchar, 2003; Liu et al., 2022). Results instead show features of micro-fracturing and size reduction. This evidences physical weathering as main weathering process for resistant phases (Wilson, 2004).

5.2. Characteristics of secondary minerals and indication to the weathering environment

The main secondary minerals of the studied samples include kaolinite, goethite, hematite, and gibbsite. This mineral assemblage is characteristic for ferralsols, typical in humid tropical regions (Deckers et al., 2024). Similar secondary mineral assemblage has been reported in regoliths derived from the weathering of granite (Nguetnkam et al., 2007; Fu et al., 2019; Li et al., 2020; da Silva et al., 2020; Liu et al., 2022), gabbro (Ngo Bidjeck Bondje et al., 2019), undersaturated alkaline rocks (Wall et al., 1996; Etame et al., 2009a; 2009b; Bollaert et al., 2023b; Wang et al., 2024), and syenite (Braun, 1991; Ntomb et al., 2024) in tropical and subtropical climatic regions. These comparisons suggest that the formation of kaolinite, goethite, hematite, and gibbsite is not unique to the studied nepheline syenite but reflects a broader pattern of mineral transformation under prolonged leaching and hydrolysis, and intense chemical weathering in warm, humid climates.

Kaolinite is ubiquitous in the profile and appears to result mostly from the dissolution of primary minerals and neoformation. The occurrence of kaolinite also indicates a moderate to good drainage condition (Nguetnkam et al., 2007; Deckers et al., 2024). As previously highlighted, kaolinite may be Fe-enriched, with up to 20 wt % of FeO_T.

Table 4
Semi-quantitative EDS Analyses of observed minerals in pedolith samples.

	Zircon		Kaolinite/halloysite		(Fe)-Kaolinite/halloysite		Goethite		Hematite		Ilmenite		Altered ilmenite	
	EB21	EB20	EB21	EB20	EB21	EB20	EB21	EB20	EB21	EB20	EB21	EB20	EB21	EB20
Al ₂ O ₃	-	-	-	-	34.12	38.98	35.26	10.98	0.66	0.00	5.44	3.02	-	-
SiO ₂	31.85	39.81	36.32	37.16	34.76	34.95	9.52	1.93	0.47	0.34	0.92	0.66	-	-
K ₂ O	-	41.03	36.92	-	0.25	-	2.12	-	-	-	-	-	-	-
FeO _T	0.21	-	21.06	21.37	9.76	14.37	79.35	84.61	84.55	95.29	36.46	13.10	-	-
CdO	-	2.03	-	0.27	-	-	-	-	-	-	-	-	-	-
TiO ₂	-	0.40	-	-	-	-	-	-	-	-	-	-	-	-
CaO	-	-	0.60	-	0.45	-	0.87	1.07	-	-	42.09	69.19	-	-
Nb ₂ O ₅	-	-	-	-	-	-	0.00	0.17	-	-	0.13	0.28	-	-
SO ₃	-	-	-	-	0.62	0.20	-	0.30	-	-	1.30	1.90	-	-
ZrO ₂	65.64	-	-	-	-	-	-	-	-	-	-	-	-	-
HfO ₂	0.68	-	-	-	-	-	-	-	-	-	-	-	-	-
Ac	-	-	-	-	-	-	-	-	-	-	-	-	-	-
UO ₂	-	-	-	-	-	-	-	-	-	-	-	-	-	-
P ₂ O ₅	-	-	-	-	-	-	1.90	0.64	-	-	1.08	0.84	-	-
Total	98.38	83.27	94.90	97.99	79.98	84.58	94.26	99.69	85.68	95.63	87.41	89.00	-	-

Element concentrations are in wt. %.

Table 5
Quantitative EPMA analyses of pyrochlore.

	EB2R											
	n. 11 Pyrochlore core					n. 2 Pyrochlore border					n. 1 Pyrochlore border	
	Average	Max	Min	median	SD	Average	Max	Min	Median	SD	Analysis	
Na ₂ O	6.42	6.75	6.23	6.39	0.16	0.91	1.55	0.14	0.91	0.91	0.14	
SrO	0.25	0.41	0.09	0.25	0.11	0.06	0.06	0.06	0.06	0.00	4.67	
Pr ₂ O ₃	0.00	0.03	0.00	0.00	0.01	0.04	0.07	0.00	0.04	0.05	0.00	
Cl	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	
MgO	0.01	0.03	0.00	0.00	0.01	0.00	0.00	0.00	0.00	0.00	0.00	
ZrO ₂	0.02	0.11	0.00	0.00	0.04	0.00	0.00	0.00	0.00	0.00	0.00	
Nd ₂ O ₃	0.12	0.34	0.00	0.10	0.12	0.13	0.24	0.19	0.13	0.17	0.19	
ThO ₂	0.76	0.91	0.56	0.76	0.13	1.08	1.26	1.08	1.08	0.26	1.08	
Al ₂ O ₃	0.01	0.02	0.00	0.01	0.01	0.04	0.04	0.03	0.04	0.01	1.42	
Nb ₂ O ₅	56.79	57.88	55.37	56.99	0.91	75.14	77.64	70.07	75.14	3.54	70.07	
Sm ₂ O ₃	0.05	0.17	0.00	0.04	0.06	0.07	0.12	0.00	0.07	0.07	0.00	
UO ₂	2.09	3.65	1.11	1.76	1.04	4.27	4.36	4.18	4.27	0.13	4.54	
SiO ₂	0.00	0.00	0.00	0.00	0.00	0.03	0.07	0.00	0.03	0.05	1.85	
TiO ₂	7.64	8.21	7.02	7.57	0.43	10.91	11.13	10.75	10.91	0.31	10.75	
MnO	0.01	0.05	0.00	0.01	0.02	0.03	0.05	0.00	0.03	0.04	0.31	
CaO	19.04	20.08	18.39	19.03	0.50	4.50	6.36	2.35	4.50	2.64	2.35	
Y ₂ O ₃	0.23	0.44	0.06	0.21	0.12	0.22	0.24	0.00	0.22	0.02	0.00	
La ₂ O ₃	0.05	0.19	0.00	0.04	0.07	0.08	0.10	0.07	0.08	0.02	0.07	
Gd ₂ O ₃	0.04	0.12	0.00	0.01	0.05	0.07	0.14	0.08	0.07	0.10	0.08	
BaO	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	
P ₂ O ₅	0.09	0.15	0.04	0.09	0.03	0.02	0.02	0.02	0.02	0.00	0.06	
Ce ₂ O ₃	0.20	0.38	0.00	0.20	0.13	0.29	0.38	0.38	0.29	0.13	0.43	
FeO	0.23	0.67	0.00	0.20	0.22	0.14	0.26	0.02	0.14	0.17	0.21	
Dy ₂ O ₃	0.04	0.10	0.00	0.03	0.04	0.01	0.01	0.00	0.01	0.01	0.00	
Er ₂ O ₃	0.03	0.18	0.00	0.00	0.06	0.01	0.01	0.01	0.01	0.00	0.06	
Yb ₂ O ₃	0.09	0.28	0.00	0.07	0.09	0.17	0.34	0.00	0.17	0.24	0.00	
HfO ₂	0.04	0.41	0.00	0.00	0.14	0.00	0.00	0.00	0.00	0.00	0.00	
Ta ₂ O ₅	1.09	1.56	0.78	1.10	0.25	1.44	1.56	1.32	1.44	0.17	1.65	
F	4.66	4.94	4.18	4.71	0.25	0.36	0.71	0.00	0.36	0.50	0.06	
Total:	100					100					100	

Element concentrations are in wt. %. The values were normalised at 100 %.

The presence of Fe-rich kaolinite could be explained either by the incorporation of Fe into the crystal structure of kaolinite or by the presence of fine inclusions of Fe-oxides in the clay mineral (Jigel et al., 2018). Although the present study does not clearly verify the structural incorporation of Fe in kaolinite, experimental synthesis has shown that up to 30 wt % of FeO_l can be accommodated in the crystalline structure of kaolinites, mainly in their octahedral layers (Iriarte et al., 2005), while decreases in the Al:Fe ratio of the experimental media are proportional to increases in the substitution rate of Fe³⁺ for Al³⁺ (Rozenon et al., 1982; Iriarte et al., 2005). Also, Fe-rich kaolinite (up to 12.8 % Fe₂O₃; Herbillon et al., 1981; Wada and Mizota, 1982; Ryan et al., 2016) was also identified in lateritic soils in previous studies suggesting a common occurrence of Fe-rich kaolinite during tropical weathering (e. g., Herbillon et al., 1981; Mendelovici et al., 1979; Tardy and Nahon, 1985; Hart et al., 2002; Ryan et al., 2016).

Fe-(oxy)hydroxides, particularly goethite, are found in all the weathered materials studied. The predominance of goethite over hematite along the entire profile can be related to high water activity during regolith formation under wet tropical climate (Tardy and Nahon, 1985; Bollaert et al., 2023b), and under such situation, goethite is the most thermodynamically stable Fe-(oxy)hydroxide phase (Santoro et al., 2021).

5.3. Behaviour of Nb-bearing mineral during weathering

Niobium is known as a geochemical tracer because of its low solubility. The main minerals identified as Nb carriers in the bedrock and regolith are pyrochlore and Fe-Ti-oxides respectively. Pyrochlore weathering has been studied in various geological contexts, primarily in relation to carbonatites and their regolith cover, which can host valuable Nb ore deposits. There is broad consensus that pyrochlore alteration occurs under low-temperature conditions and through supergene

weathering processes (Lottermoser and England, 1988; Lumpkin and Ewing, 1995; Wall et al., 1996; Palmieri et al., 2022; Bollaert et al., 2023b).

The weathering of pyrochlore (A_xB_yO₅) proceeds by the continuous leaching of elements from the A-site, which may include Na⁺, Ca²⁺, Mn²⁺, Sr²⁺, Ba²⁺, Pb²⁺ and/or REE³⁺, leading to a decrease in A site occupancy. On the other hand, weathering of calcipyrochlore, the most common type in fresh carbonatite-alkaline rocks, by replacement of Ca-Na by Ba, Sr, K and Ce in the A-site could form bariopyrochlore, strontipyrochlore, kaliopyrochlore and/or ceriopyrochlore during weathering (Wall et al., 1996). In both processes, the pyrochlore B-site, occupied by six fold-coordinated cations such as Nb⁵⁺, Ta⁵⁺, Ti⁴⁺, Fe³⁺, Al³⁺, Si⁴⁺ (Atencio, 2021; Bollaert et al., 2023b), is relatively less affected, causing an enrichment of Nb in the pyrochlore (Lumpkin and Ewing, 1995; Palmieri et al., 2022; Bollaert et al., 2023b). For a weathering pathway with continuous leaching of the A-site cations, a metastable Nb₂O₅ skeleton would be left behind and eventually dissolved (Williams-Jones and Vasyukova, 2023). Niobium liberated during this process goes into solution then will be fixed on either Fe-hydroxides or Ti-oxides (Wall et al., 1996; Atencio, 2021; Bollaert et al., 2023a; Williams-Jones and Vasyukova, 2023). Both leaching and replacement of the A-site cations are recognised in this study. The former process is expressed by a decrease in the Na, Ca and F contents and an increase in Nb content from Ca-Na-F-rich pyrochlore in the core to Ca-Na-F-poor pyrochlore in the rim (Figs. 7 and 9). This process may have been facilitated by the micro-fractures along the rim. Meanwhile the latter process corresponds to a major decrease in the Na, Ca and F contents; with an increase in Sr (in A site), Al, and Si (in B site) contents leading to the formation of Sr rich-pyrochlore (Fig. 9). Similar processes are described by several authors (Lumpkin and Ewing, 1995; Palmieri et al., 2022) who linked it to hydrothermal alteration (Bollaert et al., 2023b) or weathering processes of primary pyrochlore (Issa Filho et al.,

Pyrochlore

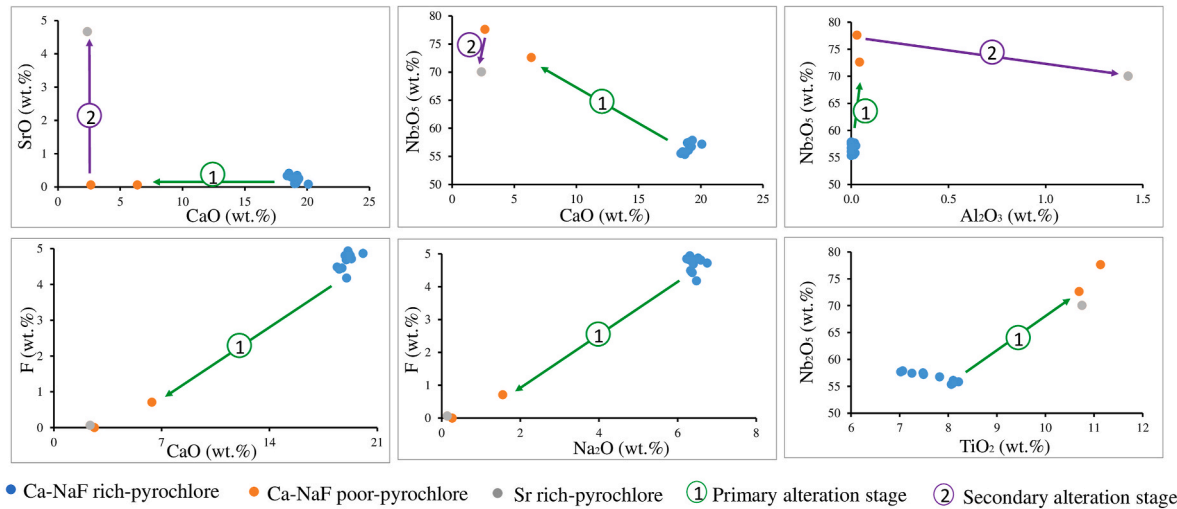


Fig. 7. Bivariant correlation diagrams between the major and minor elements of different generations pyrochlore in the parent rock. The green and purple arrows represent the primary and secondary alteration pathways of pyrochlores respectively. (For interpretation of the references to colour in this figure legend, the reader is referred to the Web version of this article.)

Ti- and Fe-Ti-Oxides

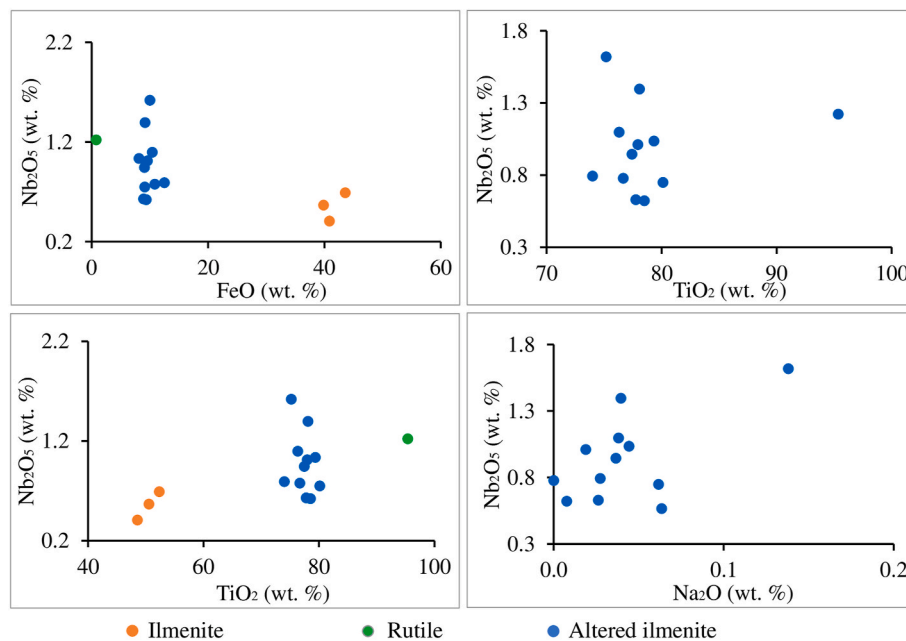


Fig. 8. Bivariant correlation diagrams between Na₂O, TiO₂, FeO and Nb₂O₅ contents of the Ti- and Fe-Ti-oxides in the weathered samples.

2013). Similar chemical variation is also observed in Araxá carbonatite, with barium-bearing calciopyrochlore and bariopyrochlore (with ~15 wt % BaO) as the main types in the regolith (Issa Filho et al., 2013; Li and Zhou, 2023; Williams-Jones and Vasyukova, 2023); in Lueshe carbonatite, with bariopyrochlore (~15 wt % BaO), strontio-pyrochlore (~6 wt % SrO), ceriopyrochlore, and kaliopyrochlore (~5 wt % K₂O) present in the laterite but absent in the bedrock, and vice versa for the Ca-Nb-F-rich calciopyrochlore (Wall et al., 1996). On the contrary, in Seis Lagos carbonatite, the Ba-Ce-enriched pyrochlore (~10 wt % BaO; ~5 wt % Ce₂O₃) are instead observed as the main Nb carrier in the parent rock while Pb-Ce pyrochlore (~20 wt % PbO; ~12 wt % Ce₂O₃) are observed in the regolith. In this study, the lack of Ba in the syenite may restrict the formation of any Ba-rich pyrochlore in the parent rock or the regolith, while Sr can be source from weathering of feldspars.

Likewise, weathering of feldspars can provide sufficient Si and Al for their incorporation into the weathered pyrochlore, which is rarely observed in the silica-undersaturated carbonatitic systems as exemplified above.

However, the alteration of pyrochlore chemistry is only observed in the slightly weathered bedrock samples. In weathered horizons, no pyrochlore were identified. This may be due to (1) a complete decomposition of pyrochlore in the weathered horizons; and/or (2) the low initial abundance of pyrochlore in the parent nepheline syenite, in comparison to other Nb-rich laterites developed on weathered carbonatites. On the other hand, Nb is variably hosted in ilmenite, altered ilmenite (leucoxene/pseudorutile) and Ti-oxides in the regolith (Table 3). Although there is no direct evidence for the genetic relationship between pyrochlore and these minerals in our samples, likely

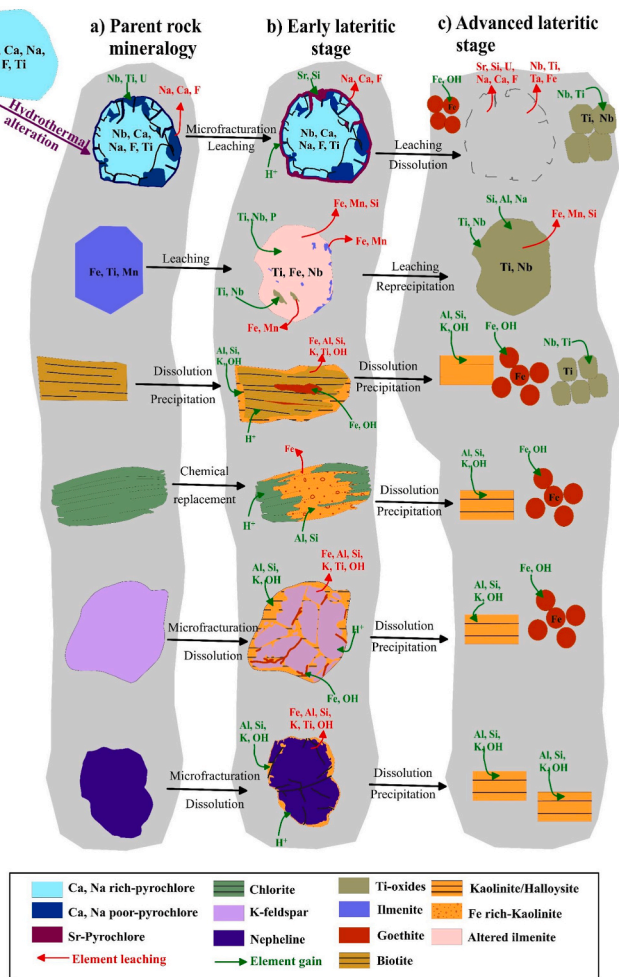


Fig. 9. Scheme of mineral weathering, neoformation and element mobilization during progressive weathering of the Eboundja nepheline syenite.

due to the complete weathering of pyrochlore (Fig. 9) as stated above, it is expected that, the Nb_2O_5 present in Ti- and Fe-Ti-oxides originates from dissolution of the pyrochlore grains (cf. Bollaert et al., 2023b). Furthermore, Ti-oxide minerals may result either from the dissolution of Ti-bearing minerals in the weathered rocks, like pyrochlore and annite following the processes described above (Fig. 9) (Bisdorn et al., 1982; Bollaert et al., 2023b); or directly as weathering products of other Fe-Ti-oxide minerals (Deysel, 2007; Kundsén et al., 2015). The latter could proceed by leaching of Fe, Mn and Mg from ilmenite grains, associated with the introduction of elements such as Al, Si, P, Nb, Ti, and other HFSE (Fig. 9). Progressive oxidation and expel of Fe promote the Nb content from ilmenite to altered ilmenite (leucoxene) and eventually to Ti-oxides (Fig. 9) (Meinhold, 2010). The increase in Na content with alteration of ilmenite is probably tied to precipitation of clay mineral internally in the altered grains, as previously observed (Deysel, 2007; Kundsén et al., 2015; Bollaert et al., 2023a). The low totals obtained for altered ilmenite may be due to the introduction of microporosity (Kundsén et al., 2015).

6. Conclusions

This study elucidates the complex interplay of mineralogical variability and weathering processes of a nepheline syenite in Southwest Cameroon. The observed diversity in the mineral assemblage reflects both varying degrees of weathering and the inheritance of the source rock. Micro-fracturing, dissolution, and chemical replacement were identified as critical factors at the mineral-scale weathering process. Notably,

dissolution enhanced by micro-fracturing emerged as the predominant weathering mechanism for K-feldspar and nepheline. The formation of secondary minerals such as kaolinite, goethite, and hematite underscores the effects of intense weathering, particularly under humid conditions. The presence of Fe-rich kaolinite suggests significant incorporation of Fe into its structure. Furthermore, the study highlights the nuanced behavior of Nb-bearing minerals, particularly pyrochlore, ilmenite and Ti-oxides, during weathering. Alteration of pyrochlore under low-temperature conditions and the formation of secondary Ti-oxides indicate a complex geochemical evolution. A complete dissolution of pyrochlore is likely and the released Nb is mobilized and potentially trapped in secondary Ti-oxides. Meanwhile, weathering of ilmenite causes progressive enrichment of Nb and Ti in the structure, contributing to part of the Nb mineralization in the regolith. Overall, these findings contribute valuable insights into the mineralogical and geochemical dynamics of tropical weathering, emphasizing the potential of Ti- and Fe-Ti-oxides as important niobium sources. Future research should focus on the long-term implications of these processes, particularly in the context of resource exploration and environmental management in tropical regions.

CRediT authorship contribution statement

Kenneth Kathy Kwanang: Writing – original draft, Methodology, Investigation, Formal analysis, Data curation, Conceptualization. **Louise Marie Ngo Bidjeck Bondje:** Writing – review & editing, Validation, Supervision, Conceptualization. **Licia Santoro:** Writing – review & editing, Writing – original draft, Supervision, Methodology, Formal analysis, Conceptualization. **Martin Yan Hei Li:** Writing – review & editing, Validation, Data curation, Conceptualization. **Paul-Désiré Ndjigui:** Writing – review & editing, Visualization, Validation, 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.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at <https://doi.org/10.1016/j.jafrearsci.2025.105901>.

Data availability

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

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