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Chemical Characterization OF Clays Located in Palora, Morona Santiago Province of Ecuador

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Abstract: The chemical characterization of clays in Palora, Ecuador, aims to analyze their structure and elemental composition for industrial potential. The study focuses on the Mera and Tena geological formations. Two samples, designated LAPM-003 from Mera and LAPM-011 from Tena, were analyzed using X-ray fluorescence (XRF), X-ray diffraction (XRD), and scanning electron microscopy (SEM). The XRF results indicated 58% SiO_2 and 19.09% Al_2O_3 in the Mera sample, and 45.19% SiO_2 and 23.15% Al_2O_3 in the Tena sample. SEM analysis revealed moderately crystallized hexagonal structures in the Mera sample and medium crystallization in the Tena sample, indicating the presence of beidellite and other smectites. XRD analysis further confirmed the predominant minerals in the Mera sample as quartz and illite, with the Tena sample showing higher kaolinite content. The high kaolinite content and low plasticity of the Mera sample renders it well-suited for ceramic and porcelain production. Conversely, the Tena sample's smectite properties, with high cation exchange capacity and adsorptive properties, make it promising for environmental and water treatment applications. Industrial importance of Palora clays is shown for local production to promote economic development and environmental sustainability, with applications in ceramics and environmental remediation. Further research is suggested to fully exploit the potential.

Keyboards: Chemical Characterization, Ecuador, LAPM-003 and LAPM-011.

INTRODUCTION

In Ecuador, during the late 1980s and 1990s, an inventory of non-metallic minerals was conducted, highlighting the occurrence of kaolinitic clay deposits of good plasticity within the Morona Santiago province [1]. These clays contain quartz, kaolinite, and muscovite as the main minerals, which are of growing importance to ceramic companies located in the Andean region. Other outcrops of common clay, with potential applications for brick and tile manufacturing, have also been identified [2]. Over the last 40 years, Morona Santiago has been the leading producer of raw materials for the ceramic industry, contributing 60% of the material employed by companies such as EDESA, Franz Viegener, RIALTO, Italpisos, Graiman, and Cerámica Andina [3].

Clays are the primary source of raw material for the manufacture of ceramics, found in various types of rock formations at different stages of formation. Therefore, the physical, chemical, and mineralogical characteristics vary widely, even within the same mineral deposit, highlighting the need to characterize the raw material [4]. The main properties influencing the categorization of clays are chemical, physical, and mineralogical composition, as they affect the quality of the ceramic. For instance, clays with high kaolinite content produce ceramics with refractory characteristics, light coloration, and good mechanical resistance, while high illite and montmorillonite content confer high plasticity to ceramic pastes, which can be detrimental to ceramic production in high proportions [5].

This research addresses the characterization of clay minerals located in Ecuador, specifically in the Morona Santiago Province, continuing the research line of the Palora - San Juan Bosco trend to strengthen the ceramic industry in the province by utilizing the region's mineral resources. According to Jaramillo's research (2018), a deposit of clay minerals is located in the Nuevo Amundalo sector within the jurisdiction of Palora canton [6]. The geological configuration of this sector includes formations related to clay deposits such as the Arajuno Formation with clays, sandstones, and lignite; the Tena Formation with shales and red beds; and the Mera Formation, mainly composed of Quaternary piedmont fans, tuffaceous sandstones, and clays [7].

The Palora parish of Palora canton shows the presence of clay material with high plasticity, light coloration, and excellent resistance to high temperatures, tentatively suitable to produce high-quality bricks, tiles, sanitary porcelain, and refractory materials [8]. Despite the identification of natural resources, there is a lack of information on the mineralogical characteristics and chemical composition within the categorization of the raw material. This information is essential for better understanding the resource, ensuring better negotiation and commercialization. Therefore, this research presents the chemical composition of the material and its structure through the following tests: Scanning Electron Microscopy (SEM), X-ray fluorescence (XRF), and X-ray diffraction (XRD).

MATERIALS AND METHODS

The research was planned in the following phases: fieldwork and laboratory work.

Fieldwork

The research was conducted in the Tena and Mera geological formations in the Republic of Ecuador, Morona Santiago Province, Palora canton, Palora parish, Nuevo Amundalo sector, where 15 samples of clay material were collected, each weighing approximately 6 kilograms (Figure 1).

The sample selection modality was based on the morphological characteristics of the terrain and the accessibility of each sample point, choosing random sampling for the prospective character of the clay mineral, made possible by the geological knowledge of the formations comprising the study area.

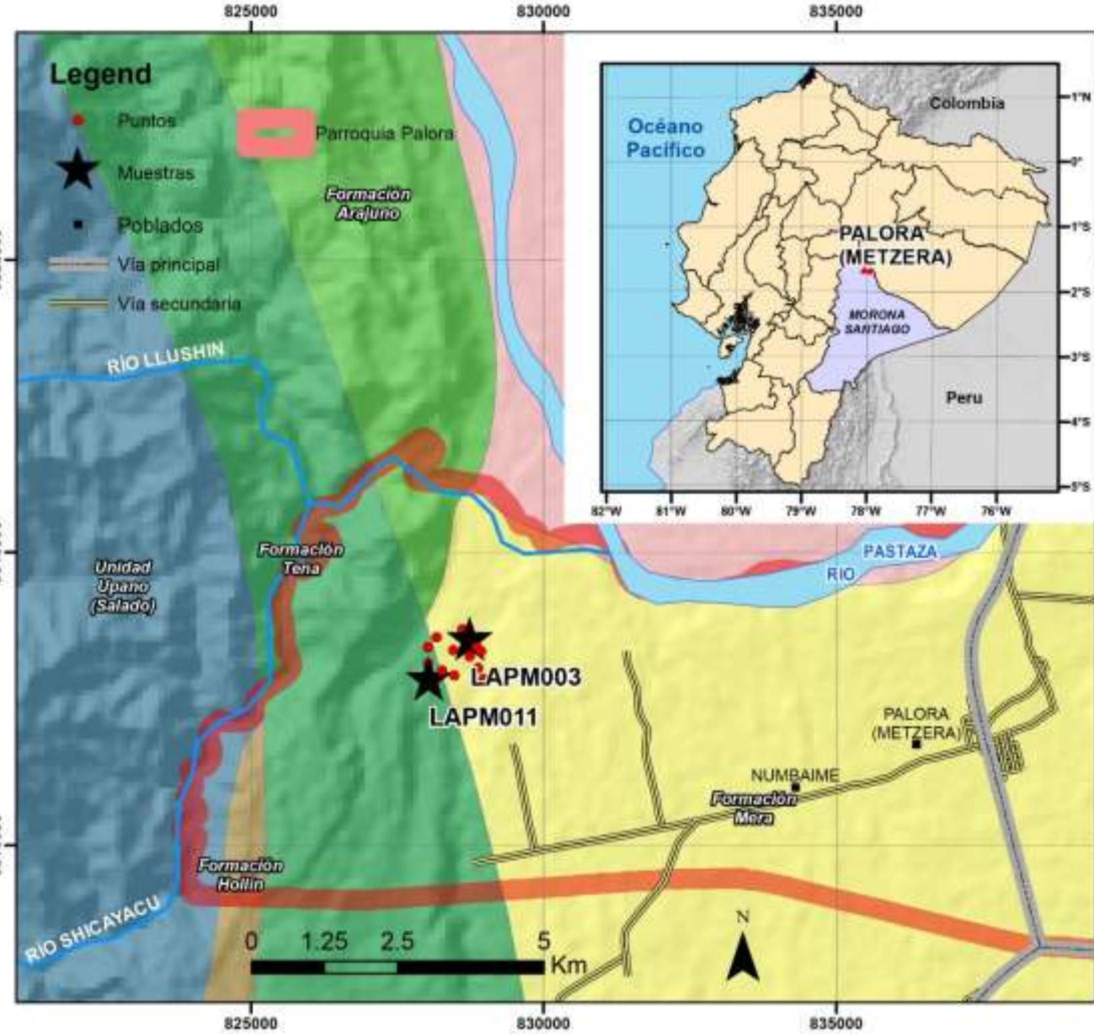

Figure 1. Location map of the study area. Geology and samples are shown.

Laboratory Work

Based on the physical properties of the material described in [8], the plasticity index was considered the main criterion for conducting chemical quantification tests, selecting two samples: LAMP-003 from the Mera Formation and LAMP-011 from the Tena Formation. These samples underwent X-ray fluorescence (XRF) tests using the S8 Tiger equipment with the Spectra Plus program, X-ray diffraction (XRD) by means of the BRUKER D8 Advance equipment with the HighScore Plus program and scanning electron microscopy (SEM) employing the JSM-IT100LA model with the JOEL EDS System software.

Results

Two representative samples were selected, one from each geological formation in the study area, LAPM-003 (Mera Formation) and LAPM-011 (Tena Formation). These samples underwent XRF, XRD, and SEM tests to obtain chemical composition and structure.

3.1. Analysis and Interpretation by Scanning Electron Microscopy (SEM)

For the LAPM-003 sample (Mera Formation), Figure 2 shows the micrographs obtained by SEM with magnifications of 200X and 1100X. These micrographs reveal moderately crystallized clay mineral structures similar to hexagonal crystalline structures of kaolinite-type clays 1:2-3 with flattened particles characteristic of clay mineral layers.

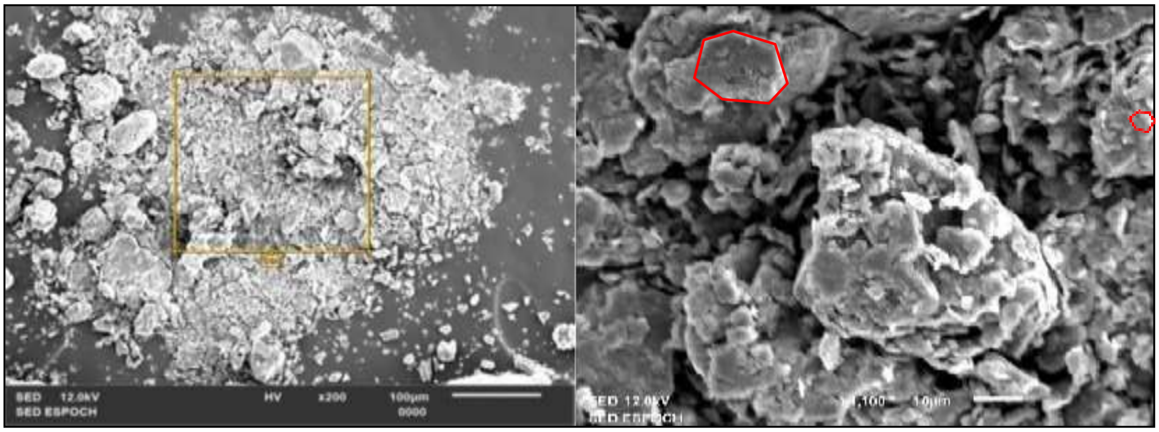

Figure 2. Micrographs obtained for sample LAPM-003 (Mera Formation) by SEM with a magnification of 200X (left) and 1100X (right). Modified from [8].

For the LAPM-011 sample (Tena Formation), Figure 3 shows the micrographs obtained by SEM with magnifications of 400X and 1500X. These images identify clay mineral structures with a medium degree of crystallization, like hexagonal crystalline structures of certain types of clays, as well as orthorhombic structures characteristic of the beidellite variety belonging to the smectite or montmorillonite group.

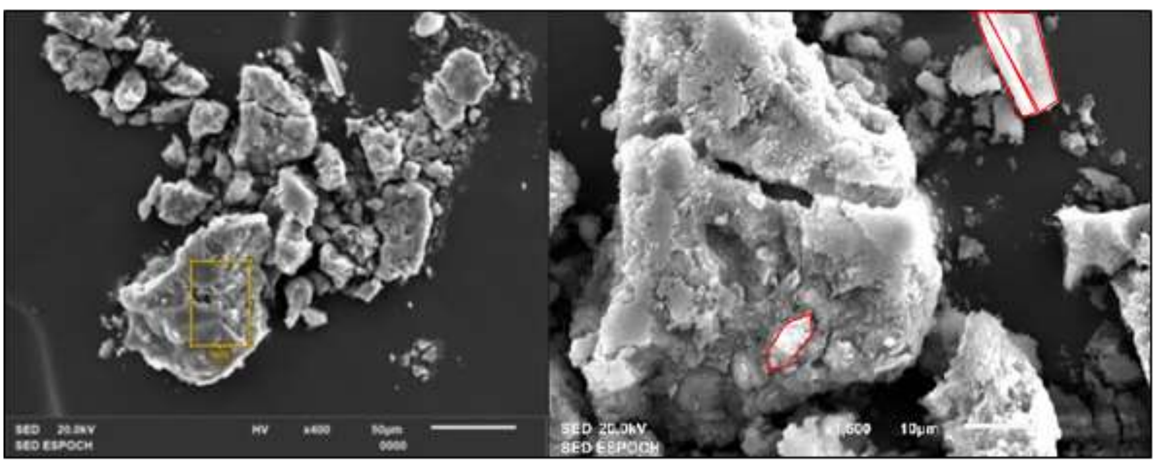

Figure 3. Micrographs obtained for sample LAPM-011 (Tena Formation) by SEM with a magnification of 400X (left) and 1500X (right). Modified from [8].

X-ray Fluorescence (XRF) Analysis

Table 1 presents the results of the chemical analysis of sample LAPM-003 (Mera Formation) by XRF. The following element percentages were identified: SiO_2 58%, Al_2O_3 19.09%, Fe_2O_3 2.9%, K_2O 2.1%, TiO_2 1.03%, and MgO 0.88%. Other compounds were negligible by the employed technique. According to the analyzed characteristics, the clays of the LAPM-003 sample (Mera Formation) would belong to the kaolin group. This interpretation is based on the low plasticity of the clay, the 1:3 ratio between Al_2O_3 and SiO_2, and its morphology according to [9] research.

Additionally, the clay has a white to slightly reddish-white color. The mineral composition is not entirely crystallized, as it is not in a pure state, and its minerals are associated with clay-sized granulometries with sand impurities (74%), which hinders crystallization. Then, it is interpreted as Anauxite type due to the presence of hexagonal crystals belonging to the kaolin group composed of kaolinite and interlayered silica with low Fe content.

Table 1. Chemical composition of Sample LAPM-003 obtained by XRF.

Element	**LAPM-003** *content* (%)	**Oxide**	**LAPM-003** *content* (%)
Si	2711	SiO_2	58
Al	101	Al_2O_3	19.09
Fe	203	Fe_2O_3	2.9
K	174	K_2O	2.1
Ti	62	TiO_2	1.03
Mg	53	MgO	0.88
Na	17	Na_2O	0.23
Ca	4	CaO	0.06
Zr	3	ZrO_2	0.04
P	2	P_2O_5	0.04
Cr	1	Cr_2O_3	0.02
Mn	1	MnO	0.01
V	~	V_2O_5	~
Pb	~	PbO	~
Cl	2	~	~
S	1	~	~

Table 2 presents the results of the chemical analysis of sample LAPM-011 (Tena Formation) by XRF. The following element percentages were observed: SiO_2 45.19%, Al_2O_3 23.15%, Fe_2O_3 3.17%, TiO_2 1.98%, and MgO 0.21%. Other elements identified by the technique did not exceed the detection threshold and are not considered significant for this analysis.

According to the tested characteristics and in contrast with Singer's research, the clays of the LAPM-011 sample (Tena Formation) may belong to the kaolin group and present minerals from the smectite group. This analysis is based on the components provided in Table 2, where the chemical composition of the minerals aligns with the proportions of beidellites with the Al_2O_3 to SiO_2 ratio. It presents a pale reddish color in its fine portion [8] along with the identification of hexagonal and orthorhombic crystals. It is interpreted that the clay contains minerals from both the kaolin and smectite groups.

Table 2. Chemical composition of Sample LAPM-011 obtained by XRF.

Element	**LAPM-011** *content* (%)	**Oxide**	**LAPM-011** *content* (%)
Si	2112	SiO_2	45.19
Al	1225	Al_2O_3	23.15
Fe	222	Fe_2O_3	3.17
K	26	K_2O	0.31
Ti	119	TiO_2	1.98

Mg	12	MgO	0.21
Na	6	Na_2O	0.08
Ca	5	CaO	0.07
Zr	9	ZrO_2	0.12
P	1	P_2O_5	0.03
Cr	1	Cr_2O_3	0.02
Mn	---	MnO	---
V	2	V_2O_5	0.04
Pb	2	PbO	0.02
Cl	---	---	---
S	2	---	---

X-ray Diffraction (XRD) Analysis

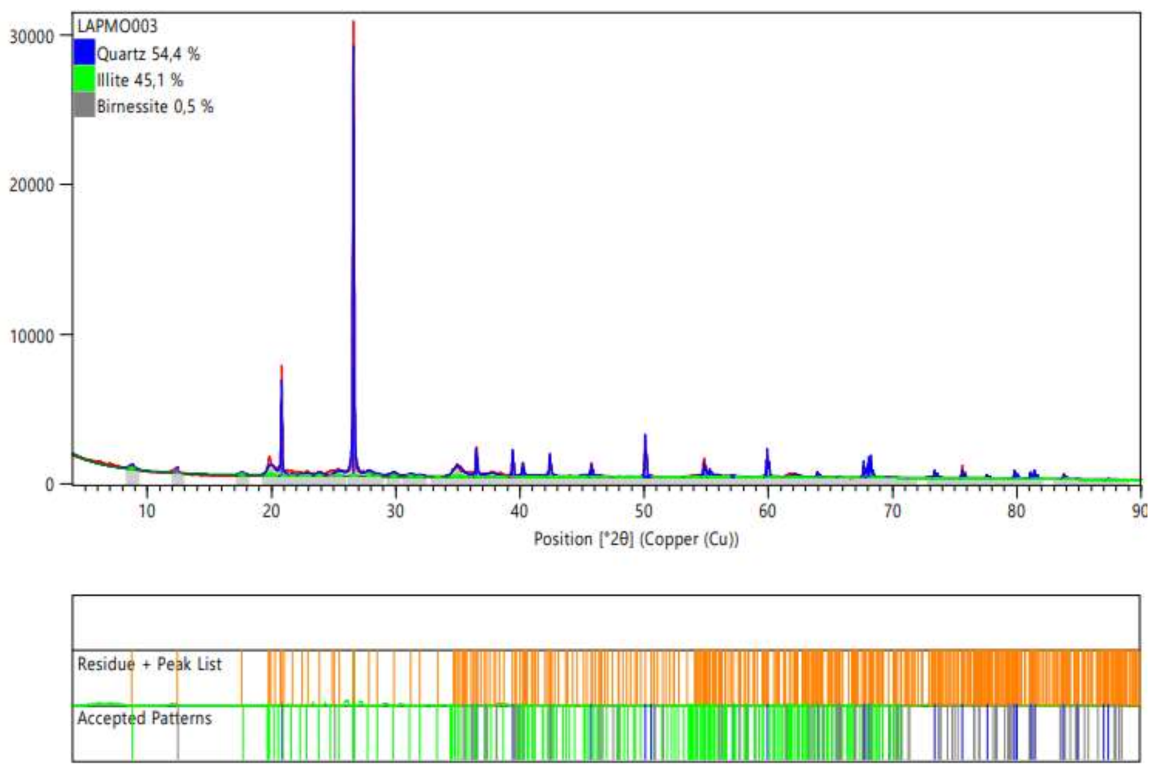

Figure 4. X-ray diffractogram of sample LAPM-003 (Mera Formation).

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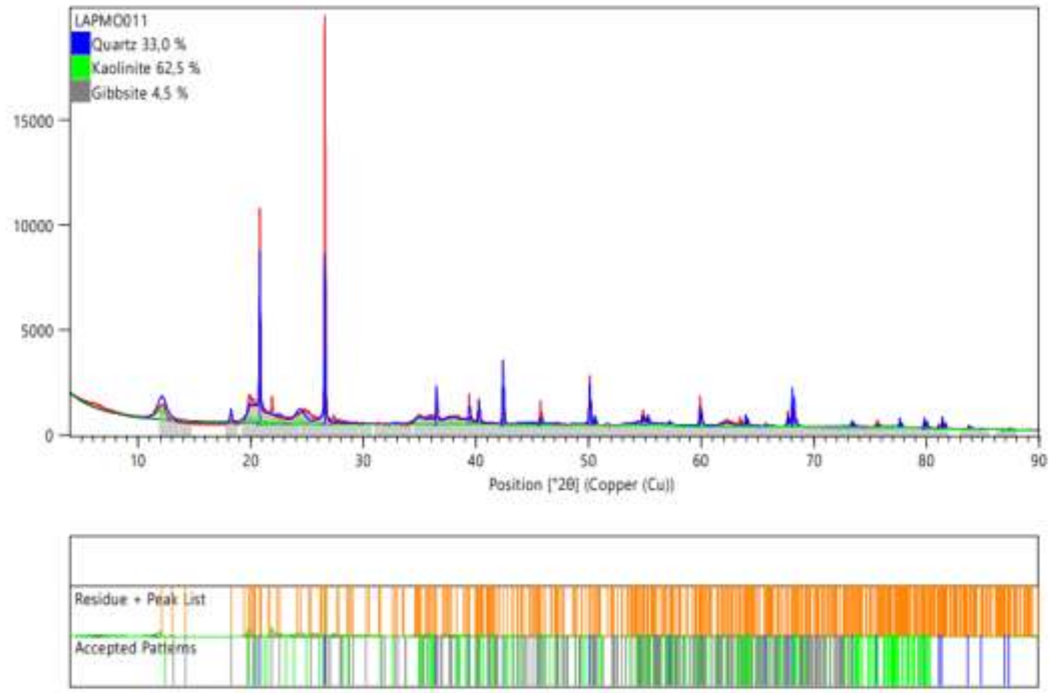

Figure 5. X-ray diffractogram of sample LAPM-011 (Tena Formation).

Figure 4 shows the diffractogram of the LAPM-003 (Mera Formation) clay sample, primarily composed of quartz (54.4%) and illite (45.1%) with a small amount of birnessite. This composition suggests high mechanical resistance and chemical stability due to the significant presence of quartz. Illite provides plastic properties and water retention, while birnessite, although in smaller quantities, can affect the sample's chemical properties.

Figure 5 shows the diffractogram of the LAPM-011 (Tena Formation) clay sample, primarily composed of kaolinite (62.5%) and quartz (33%) with a small amount of gibbsite. This composition suggests high water retention capacity and plastic properties due to the significant presence of kaolinite. Quartz contributes to mechanical resistance and chemical stability, while gibbsite can influence the chemical properties due to its moisture retention capacity.

DISCUSSION

The chemical and mineralogical characterization of clays from the Mera (LAPM-003) and Tena (LAPM-011) formations reveals significant properties that could influence their industrial applications. The SEM, XRF, and XRD techniques provided a detailed analysis of the morphology, composition, and crystalline structure of the samples, determining their potential use.

Chemical Composition and Mineralogical Structure

The XRF and XRD analyses indicate that the LAPM-003 sample (Mera Formation) has a composition rich in SiO_2 (58%) and Al_2O_3 (19.09%) with smaller proportions of other oxides such as Fe_2O_3, K_2O, and TiO_2. These characteristics are typical of kaolin clays, known for their low plasticity and high refractoriness, making them suitable for ceramics and porcelain production [10], [11].

The SEM micrograph shows moderately crystallized clay mineral structures with hexagonal morphologies characteristic of kaolinite [12]. The low presence of impurities and moderate crystallinity suggest that these clays can be used as raw materials in the production of high-quality ceramic materials, taking advantage of their whiteness and ability to form a dense, resistant matrix after sintering [13].

In contrast, the LAPM-011 sample (Tena Formation) reflects a different chemical composition with higher Al_2O_3 content (23.15%) and significant SiO_2 proportion (45.19%). This sample is characterized by the presence of orthorhombic and hexagonal structures identified by SEM, corresponding to beidellite and other smectite types [14]. Smectites, with their cation exchange capacity and high specific surface area, have potential applications in contaminant adsorption and catalytic processes [15].

The XRD analysis confirms the predominance of quartz and kaolinite in the LAPM-003 sample, suggesting its possible use as a degreaser in ceramic paste formulations, improving the plasticity and mechanical resistance of the final products [16]. On the other hand, the LAPM-011 sample, with its high kaolinite content and smaller amounts of quartz and gibbsite, presents characteristics advantageous for use in the paper industry and coating manufacturing due to its high whiteness and fineness [11].

Industrial Application Potential

The high purity and rheological properties of the LAPM-003 sample (Mera Formation) suggest its suitability for applications in the ceramic industry, specifically in the production of porcelains and refractory materials. Kaolinite clays are widely used in these sectors due to their ability to withstand high temperatures without deforming and their low thermal expansion [17]. Additionally, the global demand for kaolin in the paper industry, where it is used as a filler and coating, could benefit the local economy if commercial exploitation of these deposits is considered [18].

Regarding the LAPM-011 sample (Tena Formation), the adsorptive properties of smectites could be exploited in environmental and water treatment applications. Smectite clays are known for their ability to adsorb heavy metals and other contaminants, making them ideal for use in geological barriers and industrial effluent treatment systems [19], [20]. Implementing technologies based on local clays would not only promote environmental sustainability but also reduce costs associated with importing adsorbent materials.

The economic impact of using clays in Latin America is significant, reflecting the value of mineral deposits and the industrial opportunities they offer [18]. In Brazil and Mexico, the exploitation of kaolin and smectites has boosted exports and job creation, fostering economic growth [19]. The relevance of smectite clays in civil and environmental engineering applications is highlighted, emphasizing their value in sustainability contexts. In agriculture, the cation exchange properties of smectites improve soil fertility and agricultural productivity [21]. This research confirms that clays are a strategic resource for Latin America's economic development, promoting both industrial growth and technological innovation in key sectors [9], [22].

Thus, implementing these materials in industrial processes in Ecuador presents a significant opportunity for technological advancement and environmental sustainability in the country, as demonstrated in certain studies [2], [3], [23]–[25].

CONCLUSIONS

According to the analyses, the LAPM-003 sample (Mera Formation) presented hexagonal crystalline structures of kaolinite-type clays 1:2-3 with flattened particles characteristic of clay mineral layers. Additionally, the following major element percentages were identified by XRF: SiO_2 58%, Al_2O_3 19.09%, Fe_2O_3 2.9%, K_2O 2.1%, TiO_2 1.03%, and MgO 0.88%. Finally, the following minerals were identified by XRD: quartz (54.4%) and illite (45.1%) with a small amount of birnessite.

On the other hand, in the LAPM-011 sample (Tena Formation), clay mineral structures with a medium degree of crystallization, similar to hexagonal crystalline structures of certain types of clays and orthorhombic structures characteristic of the beidellite variety belonging to the smectite or montmorillonite group, were identified. According to its XRF chemical analysis, the following element percentages were observed: SiO_2 45.19%, Al_2O_3 23.15%, Fe_2O_3 3.17%, TiO_2 1.98%, and MgO 0.21%. Finally, the sample was primarily composed of kaolinite (62.5%) and quartz (33%) with a small amount of gibbsite, as identified by XRD.

The clays characterized in this study have a composition and properties making them suitable for various industrial applications. The LAPM-003 sample from the Mera Formation, with its high kaolinite content and low plasticity, is ideal for the ceramic and paper industries. In contrast, the LAPM-011 sample from the Tena Formation, rich in smectites, could have significant use in environmental and water treatment applications. Integrating these materials into local industries could drive economic development and contribute to environmental sustainability, positioning the region as a key provider of high-quality materials.

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Author contributions

SN, JC MF and ST designed the study. SN, JC MF and ST collected data. SN, JC MF and ST curated and analyzed the dataset. SN, JC MF and ST wrote the first version of the manuscript. SN, JC MF and ST supervised the project. All authors read, reviewed and approved the final version of the manuscript.

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Data availability

The data supporting the findings of this study are available from the corresponding author, upon reasonable request.

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