

Characterization of Serpentine Reject from HPAL Technology for Refractory and Construction Use

Caracterización del rechazo serpentinitico de la tecnología HPAL para uso refractario y de construcción

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Abstract

The serpentinite material rejected in the ore preparation plant of the nickel company Moa-Nickel S.A. constitutes a solid mining-metallurgical environmental liability with potential for revalorization. The purpose of this research was to characterize the physical, chemical, mineralogical, and thermal properties of the serpentinite reject in order to assess its potential as a raw material for construction and refractory applications. Dry sieving (ASTM C136), atomic absorption spectrometry, X-ray diffraction, and thermal analysis tested samples obtained with the cones and rings method. The results showed that approximately 24 % of the reject, corresponding to the $-3.33\text{ mm} + 1.4\text{ mm}$ fraction, can be used as aggregate for concrete and pavements, while about 20 % of the fine fractions smaller than 1.4 mm is of interest for refractory applications such as molding sands and refractory paints. Magnesium oxide (33.20 %) and silicon dioxide (29.16 %) predominated, with an MgO/SiO₂ ratio of 1.14 that imparts a basic character to the material. The main phases identified were serpentine (lizardite and chrysotile), forsterite-rich olivine, and kaolinitic clays. Thermal analysis revealed a mass loss of 4 % associated with the removal of hygroscopic water (up to 260 °C) and a 12 % loss corresponding to the dehydroxylation and recrystallization of serpentine and olivine minerals (390 °C–825 °C), supporting its use as a refractory material.

Keywords: serpentinite reject, environmental liabilities, refractory materials, construction materials

Resumen

El material serpentinitico que se rechaza en la planta de preparación de minerales de la empresa Moa-Nickel S.A. con tecnología de lixiviación ácida a presión, constituye un pasivo ambiental minero-metalúrgico sólido con potencial de revalorización. El objetivo de esta investigación fue caracterizar física, química, mineralógica y térmicamente el rechazo serpentinitico para evaluar su posible utilización como material para la construcción y como refractarios. Mediante el método de conos y anillos se obtuvieron muestras representativas que fueron sometidas a análisis granulométrico por tamizado seco (ASTM C136), espectrometría de absorción atómica, difracción de rayos X y análisis térmico. Los resultados mostraron que aproximadamente un 24 % del rechazo, correspondiente a la fracción -3,33 mm +1,4 mm, puede emplearse como árido para hormigones y pavimentos, mientras que cerca de un 20 % de fracciones finas menores de 1,4 mm presenta interés para aplicaciones refractarias como arenas de moldeo y pinturas refractarias. Predominaron los óxidos de magnesio (33,20%) y silicio (29,16 %), con una relación MgO/SiO₂ de 1,14 que le confieren al material un carácter básico. Se identificaron las fases principales de serpentina (lizardita y crisotilo), olivino rico en forsterita y arcillas caoliníticas. Los análisis térmicos revelan una pérdida de masa del 4 % asociada a la eliminación del agua higroscópica (hasta 260 °C) y del 12 % correspondiente a la deshidroxilación y recristalización de los minerales de serpentina y olivino (390 °C - 825 °C), lo que fundamenta su utilización como material refractario.

Palabras clave: rechazo serpentinitico, pasivos ambientales, materiales refractarios, materiales para construcción

1. INTRODUCTION

The Mayarí-Moa-Baracoa mountain massif in northeastern Cuba forms part of the northern ophiolitic complex and hosts large reserves of chromium and ferronickel-bearing ores. Prolonged exploitation of these reserves has generated large volumes of solid waste that, when accumulated in surface deposits, become Mining-Metallurgical Environmental Liabilities (MMELs) (Lewis et al., 2006; Domínguez-Carretero et al., 2024).

In the Moa region, the nickel industry using high-pressure acid leaching (HPAL) technology - particularly the Pedro Sotro Alba-Moa Nickel S.A. company - reports an average rejection rate of 30% of the total ores supplied to the pulp preparation plant, equivalent to more than one million tonnes per year. This situation has prompted numerous researchers to explore alternatives for the use of the rejected serpentinite material (hereinafter, serpentinite reject), in line with the principles of the circular economy and

sustainable waste management (Longos et al., 2020; Pons-Herrera et al., 2021).

Within the field of construction materials, Kattel (2017) carried out a technical assessment of serpentinite reject for its possible industrial use; Herrera-Casas (2018) proposed its preliminary use as a construction material, in concrete, pavements, railway ballast, and fluxes. Earlier, López-Peña (2006) prepared mortars using mixtures of reject material from different Moa technologies and obtained satisfactory results; Montero-Gil (2007) used serpentinite reject as aggregate in the production of concretes with compressive strengths of up to 20 MPa.

Regarding refractory applications, Pons-Herrera et al. (2019, 2021) investigated the prospects for industrial use of the environmental liability represented by serpentinite reject and the sustainable use of solid MMELs generated by the nickel industry in Moa, demonstrating the possibility of forming forsterite from serpentines, a product of major industrial interest for the manufacture of basic refractories.

The objective of this study was to physically, chemically, mineralogically, and thermally characterize the serpentinite reject from the HPAL technology used by Pedro Sotto Alba-Moa Nickel S.A., in order to evaluate its potential as a refractory and construction material. The study was based on the hypothesis that the coarse fractions of the serpentinite reject from HPAL technology are suitable for construction, whereas the fine fractions exhibit appropriate characteristics for refractory applications because of their chemical composition rich in magnesium and silicon oxides, their mineralogy dominated by serpentine and olivine, and their favorable thermal behavior.

2. MATERIALS AND METHODS

2.1. Geological and Technological Context of the Serpentinite Reject from HPAL Technology

The samples studied came from deposits generated by the accumulation of material rejected at the ore preparation plant of Pedro Sotto Alba-Moa Nickel S.A. (Figure 1), which uses HPAL technology to process Ni-Co lateritic ores. In this processing route, the ore is previously crushed, homogenized, and granulometrically conditioned before pressure acid leaching, thereby generating, in areas adjacent to the plant, an accumulation of reject material dominated by ultramafic minerals and composed mainly of serpentine and olivine phases.



Figure 1. Serpentine reject deposit at the pulp preparation plant of the HPAL nickel company.

2.2 Sampling and Sample Preparation

To ensure representativeness, composite sampling was carried out by the cone-and-ring method within the body of the reject deposit, in accordance with ASTM D75/D75M. Initially, 9 kg of serpentine reject were collected (Figure 2), homogenized, subjected to size reduction, and successively quartered until three 1 kg replicates were obtained for chemical, mineralogical, and thermal analyses.



Figure 2. Samples of the serpentine reject from HPAL technology.

The samples were air-dried at room temperature (25 ± 2 °C) for 48 h until constant mass, reaching an initial equilibrium moisture content of 3.5% (determined gravimetrically). The clods were manually disaggregated without crushing individual particles and were stored in hermetically sealed containers to prevent contamination and moisture absorption.

2.3 Particle-Size Characterization

Particle-size analyses were performed by dry sieving in accordance with ASTM C136/C136M, using a set of sieves with standardized openings between 10 mm and 0.10 mm. Sieving was carried out manually using a

combination of lateral, vertical, and shaking movements for ten minutes per replicate until stable separation of the fractions was achieved (mass variation of less than 0.5% during an additional minute).

The mass retained on each sieve was determined with a precision of 0.01 g, and the corresponding weight percentages were calculated. The following intervals were defined as fractions of technological interest: +10; -10+8.9; -8.9+6; -6+4.7; -4.7+3.33; -3.33+1.4; -1.4+0.8; -0.8+0.35; -0.35+0.20; -0.20+0.10; and -0.10 mm, covering sizes from coarse particles to fine fractions.

2.4 Chemical Characterization

The chemical composition of the main oxides was determined by atomic absorption spectrometry (AAS) at the Nickel Research and Development Center (CEDINIQ), using a single-beam UNICAM SOLAR 929 instrument.

Sample preparation for AAS consisted of: (1) acid digestion of 0.5 g of sample ground to -0.074 mm with an HCl (37%) and HNO₃ (65%) mixture at a 3:1 ratio; (2) heating at 120 °C for two hours on a hot plate; (3) dilution to 100 mL with deionized water; and (4) filtration through a 0.45 µm membrane. Si, Al, Mg, Ni, Co, Mn, Cr, Fe, Cu, and Zn were quantified and expressed as oxides, following the UPL-PT-A-26 technical test procedures (detection limit: 0.001%). Ferrous iron (FeO) was determined by volumetric titration with potassium dichromate (UPL-PT-V-03 method).

Loss on ignition (LOI) was evaluated gravimetrically according to procedure UPL-PT-G-01: calcination of 1 g of sample in a porcelain crucible at 1000 °C for one hour in an air atmosphere.

2.5 Mineralogical Characterization

Mineralogical analysis was carried out at the Mineralogy Laboratory of the Research Center for the Mining-Metallurgical Industry (CIPIMM), using a Bruker AXS D8 Focus X-ray diffractometer under the following measurement conditions: Cu-Kα radiation ($\lambda = 1.5406 \text{ \AA}$), voltage of 40 kV, current of 40 mA, 2θ range from 5° to 70°, step size of 0.02°, counting time of 0.5 s per step, and single-crystal silicon sample holders.

The phases were identified by comparison with the ICDD-PDF4+ database (2022) using DIFFRAC.EVA v6.0 software. The semiquantitative estimate of the main phases present, with an estimated uncertainty of $\pm 5\%$, was performed by the relative-intensity method based on characteristic peaks, with special attention to serpentines (lizardite and chrysotile), forsterite-rich olivine, and kaolinitic clays.

2.6 Thermal Characterization

Thermal analyses were performed using a NETZSCH STA 449 F3 instrument, recording mass variation (TG). The experimental conditions were as follows: standard Al_2O_3 crucibles, SiC furnace, argon (Ar) atmosphere at a flow rate of 20 mL/min as protective gas, heating rate of 10 °C/min, temperature range from 27 to 1000 °C, sample mass of 25.30 mg, and Al_2O_3 reference mass of 60.20 mg.

3. RESULTS

3.1 Particle-Size Characteristics

The particle-size distribution of the reject serpentinite from HPAL technology (Figure 3) shows a distribution dominated by coarse fractions. Particles larger than 1.4 mm and up to 10 mm represent 56% of the total samples analyzed, whereas fractions smaller than 3.33 mm represent 44%.

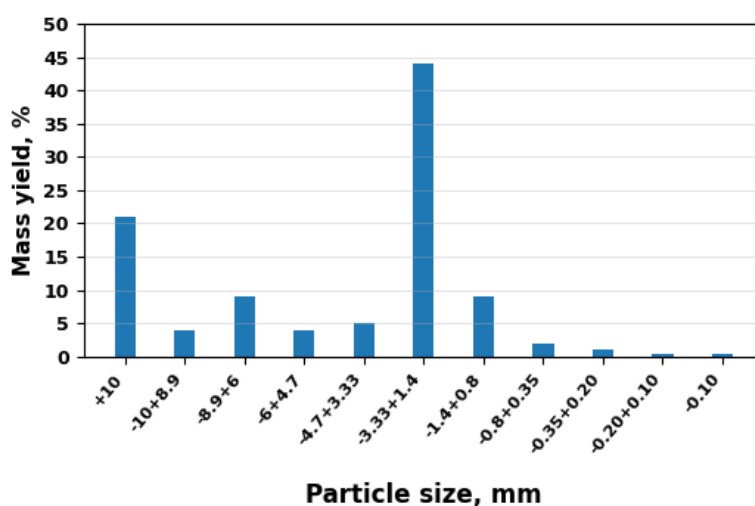


Figure 3. Particle-size distribution of the serpentinite reject (HPAL technology).

Particles larger than 10 mm, representing approximately 21% of the samples, occur as clods and rocks formed by irregular particles, which significantly affects their particle-size distribution. The intermediate fractions (-10+3.33 mm) represent an additional 11% of the material and may be classified for use as coarse aggregate.

Based on the combination of particle-size characteristics and previous experience in using serpentinite wastes as aggregates, it was determined that approximately 24% of the reject, corresponding to the -3.33+1.4 mm fraction, can be used as aggregate for concrete, pavements, and ballast. Nearly 20% of the fine fractions smaller than 1.4 mm is of interest as a base for molding sands and refractory coatings, indicating the possibility of

redirecting part of the material toward production chains in the construction and foundry industries.

3.2 Chemical Characteristics

The content of chemical elements expressed as oxides (Table 1) in the serpentinite reject shows a significant predominance of magnesium oxide (33.20%) and silicon dioxide (29.16%), typical of serpentinite minerals. This confirms the ultramafic nature of the material and its affinity with magnesium silicates, a positive indicator for refractory applications.

Table 1. Chemical composition (%) of the serpentinite reject from HPAL technology

NiO	CoO	MnO	MgO	Cr ₂ O ₃	SiO ₂	Al ₂ O ₃
0.57	0.02	0.16	33.20	0.62	29.16	0.86
FeO	Fe ₂ O ₃	LOI	Others / Undetermined	Total		
2.02	3.34	8.21	20.55	98.72		

The total sum of analyzed oxides, plus loss on ignition and the undetermined fraction, reaches 98.72%, an acceptable value for this type of material. The undetermined values (20.55%) include volatile components not individually quantified, traces of minor oxides (CaO, Na₂O, K₂O, TiO₂, P₂O₅) present at concentrations below the method detection limit, carbonates, sulfates, and possible systematic errors inherent to the conversion of elements into oxides. The remaining elements (1.28%) correspond to traces of other unquantified components.

It should be noted that iron oxides predominate at approximately 5.36% (FeO + Fe₂O₃), and they can react with alumina and silica to form stable compounds that do not adversely affect the properties of the material.

In addition, manganese, magnesium, chromium, and aluminum oxides are present and are relevant for refractory materials because they confer mechanical strength and thermal stability. The basicity ratio (MgO/SiO₂) of 1.14 is higher than the acidity ratio (SiO₂/MgO) of 0.88, providing relevant information for the use of serpentinite reject as a refractory material.

3.3 Mineralogical Characteristics

The serpentinite reject is composed mainly of particles larger than 0.84 mm, which contain important chemical elements. The main mineralogical phases include lizardite and chrysotile, phases whose structures incorporate mainly

aluminum, silicon, nickel, cobalt, and magnesium, as well as olivine (forsterite) and kaolinitic clays (Figure 4).

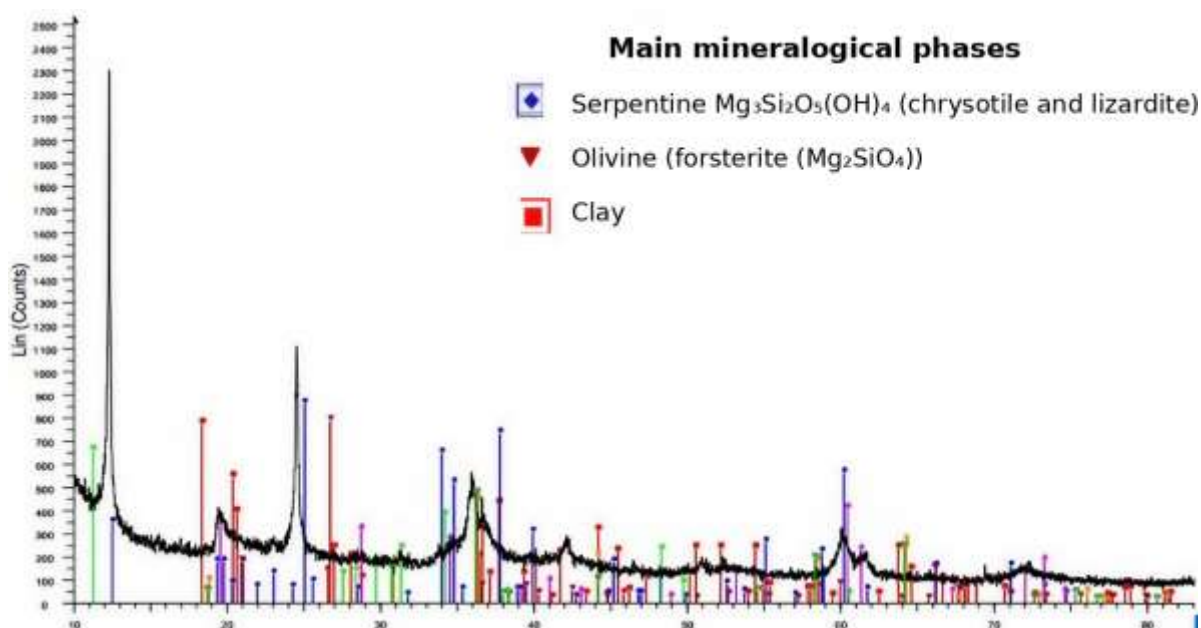


Figure 4. X-ray diffractogram of the serpentinite reject from HPAL technology.

The semiquantitative phase estimate based on the relative intensities of characteristic peaks indicates approximate contents of 34% olivine and 33% clays, along with significant proportions of serpentines whose structures contain Mg, Si, Al, Ni, and Co, consistent with the lateritic origin of the deposit. The coexistence of serpentines and olivine in comparable proportions, with a strong presence of forsterite, makes the mineralogical composition of these rejects similar to that of olivine deposits traditionally used as raw materials for basic refractories.

3.4 Thermal Characteristics

The thermal characteristics of the serpentinite reject are typical of olivine minerals from northeastern Cuba. The thermogravimetric analysis (TG) performed showed the following main thermal stages: up to 260 °C, a 4% mass loss associated with the removal of hygroscopic water; and, in the interval from 390 °C to 825 °C, a 12% loss corresponding to the removal of structural water (dehydroxylation of serpentines) and to recrystallization and restructuring of olivine and serpentine minerals.

Although these processes appear overlapped in the thermogravimetric curve (Figure 5) due to the heating rate of 10 °C/min, the combination of thermal signals (TG-DTG) and mineralogical information makes it possible to infer the typical stages of serpentine decomposition described for obtaining forsterite from serpentinite wastes.

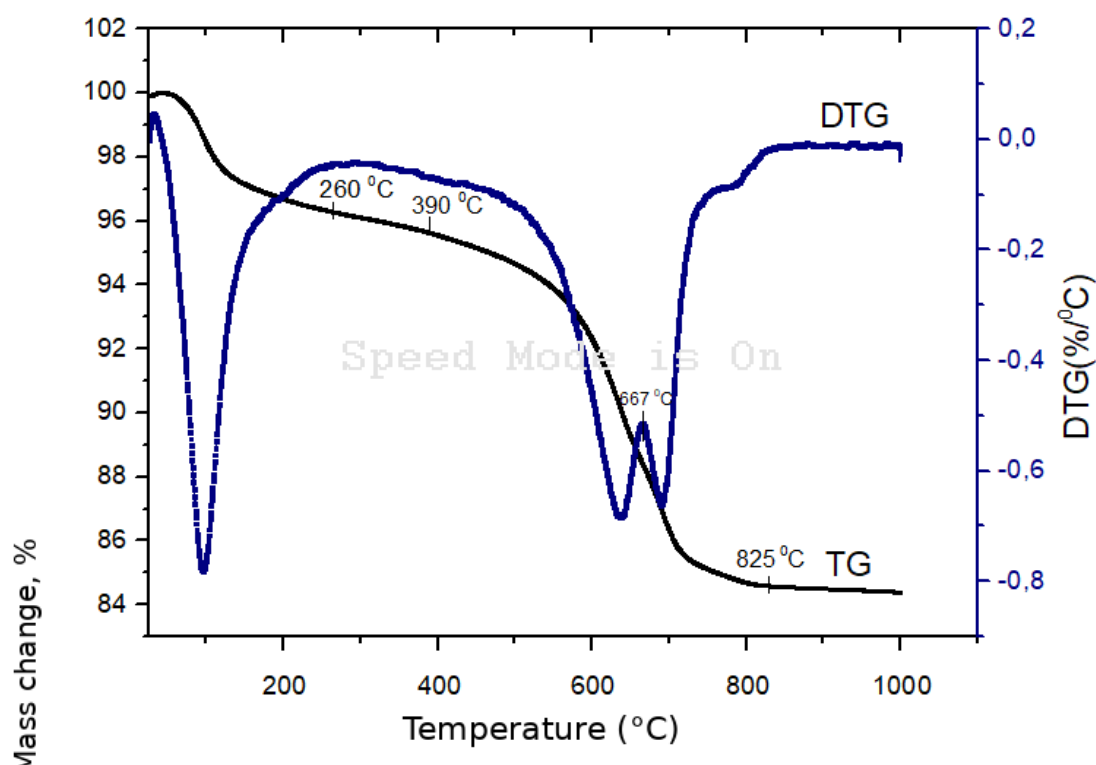


Figure 5. Thermogram of the serpentinite reject from HPAL technology.

It is important to note that the total mass loss measured by TG in an argon atmosphere (16%) differs from the LOI value determined in air (8.21%). This discrepancy is explained by the fact that, during LOI in air, Fe^{2+} is simultaneously oxidized to Fe^{3+} , a process that involves mass gain through oxygen incorporation and therefore partially offsets the mass loss caused by water volatilization. In TG under argon (an inert atmosphere), this oxidation does not occur, and only mass loss due to dehydration is recorded.

4. DISCUSSION

The particle-size results of this study demonstrate that the serpentinite reject from HPAL technology has a size distribution that allows differentiated use. The 56% of particles between 1.4 mm and 10 mm constitutes a suitable fraction for applications as coarse aggregate in concrete and pavements, provided that it is subjected to selective crushing to remove clods larger than 10 mm (21% of the total).

The -3.33+1.4 mm fraction (24%) has optimal characteristics for use as construction aggregate, with sizes that meet technical specifications for medium-strength concrete (up to 25 MPa). These results are consistent with those reported by Montero-Gil (2007), who obtained 20 MPa concretes using similar rejects from the Moa Occidental deposit, and with Herrera-Casas

(2018), who proposed the use of rejects from the Moa Oriental deposit for construction applications.

From the chemical standpoint, the predominance of MgO (33.20%) and SiO₂ (29.16%), with an MgO/SiO₂ ratio of 1.14, places the material under study within the field of basic magnesium silicates suitable for refractory manufacture. These values are comparable to those reported by Pons-Herrera et al. (2000) for serpentized dunites from the Moa-Baracoa region (MgO: 35%-40%; SiO₂: 28%-32%) and by Gámez-Matos (2011) for serpentinite wastes from the Pedro Sotto Alba mine (MgO: 32.5%; SiO₂: 30.1%).

The FeO + Fe₂O₃ content (5.36%) is suitable for refractory applications because it acts as a moderate flux that facilitates sintering without compromising the refractoriness of the final product. The low contents of NiO (0.57%) and CoO (0.02%) represent an environmental and economic advantage, since they do not constitute significant losses in HPAL technology.

Nevertheless, the discrepancy observed between LOI (8.21%) and mass loss by TG (16%) warrants detailed discussion. In an argon atmosphere, TG records only mass loss by dehydration (hygroscopic water + structural water), reaching 16%. In contrast, LOI in air simultaneously involves the oxidation of ferrous to ferric iron, with mass gain through oxygen incorporation that partially offsets volatilization losses. This phenomenon, consistently reported for ferromagnesian minerals (Pons-Herrera et al., 2019; Ojeda-Pardo et al., 2023), explains the observed difference.

The mineralogical identification of serpentines (lizardite and chrysotile), forsterite-rich olivine, and kaolinitic clays is consistent with the ultramafic origin of the deposit and its history of supergene alteration (Zhou et al., 2023). The presence of forsterite (Mg₂SiO₄) as the main component of olivine is particularly relevant, since this mineral has a melting point of 1 890 °C, making it suitable for refractories with high thermal resistance (Pons-Herrera et al., 2019). Although the qualitative and semiquantitative information obtained makes it possible to establish the material's potential as a refractory raw material, the semiquantitative percentages obtained by the relative peak-intensity method (olivine ~34%, clays ~33%, serpentines ~30%) should be interpreted as approximate estimates with an uncertainty of ±5%.

The observed thermal behavior can be interpreted through the typical decomposition mechanisms of serpentines. The first stage (up to 260 °C, 4% mass loss) corresponds to the removal of hygroscopic and surface-adsorbed water. The second stage (390 °C -825 °C, 12% mass loss) involves more

complex processes: (1) dehydroxylation of serpentine (lizardite and chrysotile) according to the reaction $\text{Mg}_3\text{Si}_2\text{O}_5(\text{OH})_4 \rightarrow \text{Mg}_2\text{SiO}_4 + \text{MgSiO}_3 + 2\text{H}_2\text{O}\uparrow$; (2) recrystallization of forsterite (Mg_2SiO_4) from serpentine decomposition; and (3) transformation of enstatite (MgSiO_3) into stable crystalline phases. These mechanisms are consistent with those reported by Aleixandre-Ferrandis and González-Peña (1972) for the formation of forsterite refractories from serpentines, as well as with those published by Kalaitzidou et al. (2022) for the valorization of serpentinite rejects from Moa.

The onset temperature of dehydroxylation (390 °C) is slightly lower than that reported for pure crystalline serpentines (450 °C -500 °C), which may be attributed to the presence of impurities and to the partially altered nature of the material. From a technological standpoint, the temperature ranges described allow the appropriate temperatures for transforming serpentines into forsterite to be identified without requiring prior thermal treatment. For molding sands, temperatures between 600 °C and 700 °C would provide a porous structure with good water and gas absorption capacity. For refractory coatings, treatments at 800 °C - 900 °C would generate a more densified matrix with higher mechanical strength, in agreement with reports for Cayo Guam dunites (Loyola-Brefe et al., 2020).

Comparison of the results of the present study with previous works by Cuban researchers reveals consistency in the general trends, but specific differences attributable to deposit variability and HPAL process conditions. Whereas Montero-Gil (2007) and Gámez-Matos (2011) worked with rejects as products, the present study focuses on rejects from HPAL technology, which show differences in particle-size distribution due to the mechanical preparation stages. This technological differentiation represents an original contribution of the present work, since the international literature on the valorization of HPAL rejects remains scarce and is dominated by studies on lateritic debris from pyrometallurgical processes.

It is important to acknowledge the limitations of the present study. First, sampling was punctual in both time and space; therefore, the seasonal variability of the deposit was not characterized. Second, mineralogical quantification was semiquantitative and would require refinement using more precise methods such as Rietveld analysis. Third, the study did not include technological tests on manufactured products (concretes, mortars, refractory coatings); consequently, the proposed applications require further experimental validation.

5. CONCLUSIONS

- Twenty-four percent of the serpentinite reject generated by HPAL technology in Moa (the -3.33+1.4 mm fraction) can be used as construction material, and 20 % (fractions <1.4 mm) can be used as refractory material, opening possibilities for differentiated use as aggregates and refractory raw materials.
- The predominance in the reject of magnesium oxide (33.20 %) and silicon dioxide (29.16 %), together with an MgO/SiO₂ ratio of 1.14, confirms the ultramafic character of the material and its affinity with magnesium silicates suitable for forsterite generation and basic refractories. The discrepancy between LOI (8.21 %) and mass loss by TG (16 %) is explained by the oxidation of ferrous to ferric iron during calcination in air.
- The potential of the material as a refractory raw material is mineralogically confirmed by the presence of the main phases of serpentines (lizardite and chrysotile), forsterite-rich olivine, and kaolinitic clays, and is justified by its thermal behavior, involving dehydroxylation and recrystallization of olivine minerals between 390 °C and 825 °C.
- The findings of the study suggest that proper management of these environmental liabilities can contribute significantly to reducing imports of silica sands and refractory coatings in Cuba. However, the following limitations should be considered: (1) sampling was punctual and did not characterize the temporal variability of the deposit; (2) mineralogical quantification was semiquantitative, with an estimated uncertainty of ±5%; (3) no technological tests were performed on products manufactured using the reject; and (4) the potential presence of chrysotile fibers in the fine fractions was not evaluated.

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Additional Information

Conflict of Interest

The authors declare that there are no conflicts of interest regarding the publication of these results.

Author Contributions

AFB: Study design, data collection, experimental development, interpretation of results, writing and revision of the original manuscript, and approval of the final version. **JAPH:** critical review and corrections of the article, approval of the final version. **PYV** and **STV:** scientific advice and planning of the experimental component. **MCRP:** corrections of the article and planning of the experiments.

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