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INFLUENCE OF GRANULOMETRY IN THE LEACHING PROCESS ENCIT 2020

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Abstract. *The increase in energy demand by this company is responsible for the release of (GHG) into the atmosphere. In order to mitigate these emissions, proposals have been disseminated to continue the use of traditional fuels, without interfering in the worldwide warming. Among these proposals, Carbon Capture and Storage (CCS) technologies play an essential role in mitigating CO₂ emissions in the atmosphere. These consist of the separation of CO₂ from anthropogenic activities. Mineral carbonation is classified as one of the CCS technologies, in which it is a technology based on the natural process of CO₂ sequestration, known as the disintegration of the rocks, generating thermodynamic and environmentally stable products. This study aimed to evaluate the influence of granulometry on the leaching process of indirect mineral carbonation of a Brazilian serpentinite, in order to compare the extraction efficiency of the elements of interest (Mg and Fe). Based on the analysis of the experimental results, it was observed that although the SB sample was mechanically activated, there was no significant increase in the efficiency of Mg extraction indicating that the SM sample is the most viable to be used.*

Keywords: *Granulometry, Mineral Carbonation, Leaching, Serpentinite.*

1. INTRODUCTION

The presence of carbon dioxide (CO₂) in the atmosphere, as well as other gases that cause greenhouse effect (GHG), has increased significantly in recent centuries, mainly since the Industrial Revolution. The increased demand for energy in developed and underdeveloped countries raises concerns about CO₂ from anthropological action into the atmosphere (ARCE et al., 2017).

Given this scenario, alternative proposals have been studied by researchers in order to mitigate CO₂ emissions into the atmosphere. The term CCS, Carbon Capture and Storage, refers to capture and storage technologies recognized by the Intergovernmental Panel on Climate Change (IPCC) (IPCC, 2005). These play an important role in climate change caused by CO₂ emissions into the atmosphere, consisting of the separation of CO₂ mainly from energy generating systems. (IPCC, 2005; ARCE et al., 2017).

One of the CCS technologies that has gained importance is the one called mineral carbonation (MC) through mineral sequestration (WANG et al., 2018). This is based on the natural CO₂ mitigation process, known as rock breakdown, which is considered to have an important role in the historical reduction of CO₂ concentrations in the atmosphere. One of the main advantages of the extraction is that it allows the production of almost pure minerals, since other impurities available in the natural mineral core can be removed after the extraction of the reactive metal (IBRAHIM et al., 2019).

Mineral carbonation is classified by direct injection of CO₂ into the rock (in situ) or indirect (ex situ) in both cases, it can occur by gas-solid reactions or in an aqueous phase. The indirect ex situ process can take place in two or more stages. The first stage consists of a leaching for the extraction of metals of interest for use in MC, usually Ca, Mg and Fe, from a silicate mineral, through the application of different solutions, which can be acidic or basic. In the second stage, carbonation is done by injecting CO₂ into the solution obtained in the dissolution, thus generating insoluble and stable carbonates. (SANNA; GAUBERT; MAROTO-VALER, 2016; SONG et al., 2019; GALINA; ARCE; ÁVILA, 2019). Researchers carefully choose the selection of process routes, process conditions and pre-treatment options in order to overcome the slow kinetics of the process reaction (IPCC, 2005; LI; HITCH, 2018).

The advantages of indirect aqueous carbonation over direct aqueous carbonation are the production of pure carbonates with high added value since there is the possibility of optimization separately for each step, with acid dissolution being the limiting step of the process due to the slow kinetics of the reaction and the large consumption of products (HITCH, 2020; IBRAHIM et al., 2019).

One of the main advantages of the extraction is that it allows the production of almost pure minerals, since other impurities available in the natural mineral core can be removed after the extraction of the reactive metal (IBRAHIM et al., 2019). A key factor in the mineral carbonation process is the particle size. Once the particle size is reduced, the surface area and the reactivity of the metals increase, consequently increasing the reaction rate. Although the mechanical activation of the raw material can make the mineral carbonation process more efficient, the energy costs involved in the pre-treatment have been a challenge (LI; HITCH, 2018; WOODALL et al., 2019).

As the carbonation step depends on the efficiency of the extraction of the elements of interest, this study aims to evaluate the influence of granulometry on the leaching process in order to compare the extraction efficiency of the elements of interest (Mg and Fe), using a national serpentinite.

2. MATERIALS AND METHODS

In Figure 1, it illustrates the simplified scheme of the methodology for the acid dissolution process of the serpentine sample, which details in detail in the sections below.

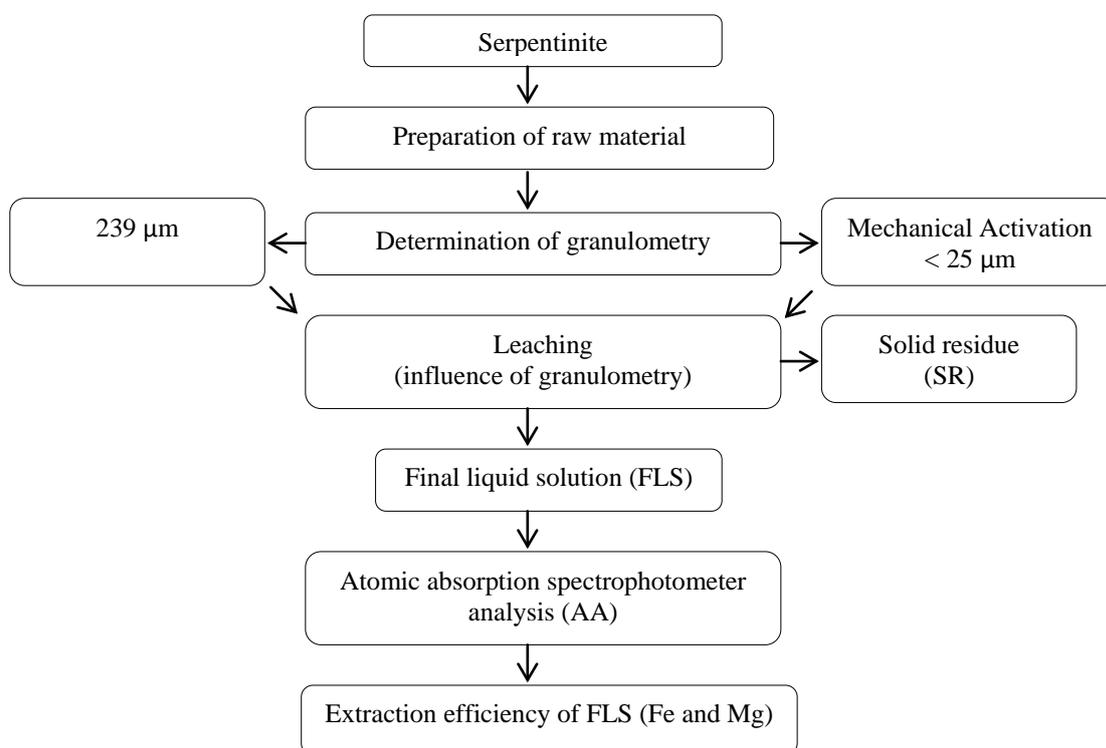


Figure 1. Simplified flowchart of the methodology used in the research
Source: The author

This study was carried out in the carbon capture combustion laboratory (LC3) and in the Materials Characterization Laboratory laboratory of the Materials Engineering Department, both belonging to FEG-UNESP.

2.1 Raw material

In this study, a national serpentinite supplied by the company Pedras Congonhas, located in Belo Horizonte, was used. The main elements that make up the raw material are silicon (SiO_2), magnesium (MgO) and iron (Fe_2O_3) oxides.

2.1.1 Preparation of raw materials

The raw material received had an open grain size band. In this study, two samples with different average particle sizes were used, named as:

- SM: A particle size analysis was performed and an average particle size of $239 \mu\text{m}$ was obtained.
- SB: The raw material was milled in a Jar mill and sieved with alcohol using a sieve shaker for the selection of particle sizes below $25 \mu\text{m}$.

2.2 Leaching

Table 1. Reaction conditions for the tests

<i>Conditions</i>	<i>Time (min.)</i>	<i>Temperature (°C)</i>	<i>Concentration HCL (Mol/L)</i>	<i>Granulometry (µm)</i>
Influence of granulometry	120	80	2,5	239 e < 25

Source: The author

In order to evaluate the influence of granulometry on the acid leaching process, the tests were performed using the same conditions, that is, 120 minutes of reaction at a temperature of 80 ° C using 100 ml of HCL with a concentration of 2.5 Mol / L , varying only the granulometry, as shown in Tab. 1. A reactor was used in which hydrochloric acid (HCL) was introduced and heated under stirring through a magnetic stirrer. After stabilizing the temperature of the experiment, 5.4 g of serpentinite sample was inserted into the reactor, keeping it under heating. After the reaction time, the system was turned off and the solution was filtered in a vacuum system, in order to separate the solid residue (SR) from the final liquid solution (FLS). The determination of the magnesium (Mg) and iron (Fe) concentrations contained in the final liquid solution were analyzed in the atomic absorption spectrophotometer (AA).

2.2.1 Atomic absorption spectrophotometer (AA)

A Shimadzu AA-7000 atomic absorption spectrophotometer available at the Materials Characterization Laboratory of the Materials Engineering Department (FEG / UNESP) was used to determine the Mg and Fe concentrations obtained in the final liquid solution (FLS). To obtain the equipment calibration curve, standard solutions of Iron and Magnesium were used, diluted in the proportions of 0.5; 5; 10; 15 and 20 ppm.

2.3 Extraction efficiency of final liquid solution

After determining the Mg and Fe concentrations obtained in the final liquid solution, the extraction efficiency of each element was determined by Eq. [1]

$$E_i \% = \left[\frac{C_{is}}{C_{iserp}} \right] \cdot 100 \quad [1]$$

Ei% being the fraction of Mg and Fe extracted from the material, Cis the concentration of metals in the dissolution solution and Ciserp the concentration of Mg and Fe in the serpentinite sample, determined in the X-ray Fluorescence (FRX) tests.

3. RESULTS AND DISCUSSION

Table 2 shows an expected concentration of Mg and Fe in 5.4 g of sample (Ciserp) provided in the FRX tests.

Table 2. Expected concentration of Mg and Fe from both samples

<i>Sample</i>	<i>Concentration (mg/L)</i>	
	<i>Mg</i>	<i>Fe</i>
SM	22,59	4,92
SB	23,96	4,43

Source: The author

After carrying out the experiments, the concentration of Mg and Fe found in the final liquid solution (Cis) was determined by the atomic absorption spectrophotometer, as shown in Tab. 3.

Table 3. Concentration of Mg and Fe in the final liquid solution of both samples

Sample	Concentration (mg/L)	
	Mg	Fe
SM	11,64	3,42
SB	12,64	3,94

Source: The author

After the leaching step, the SB sample has a higher concentration in the two elements, as shown in Tab. 3.

Since eq. [1], considering Tab. 1 as Ciserp and table 2 as Cis of the equation, the extraction efficiency (Ei%) was determined.

In this study the efficiency of extraction of Mg and Fe obtained in both samples was compared. It was found that in the SM sample the extraction efficiency (Ei%) of the Fe was around 70% and the Mg of 52%, and in the SB sample the extraction efficiency of the Fe was around 89% and the Mg of 53%, as shown in Fig. 2.

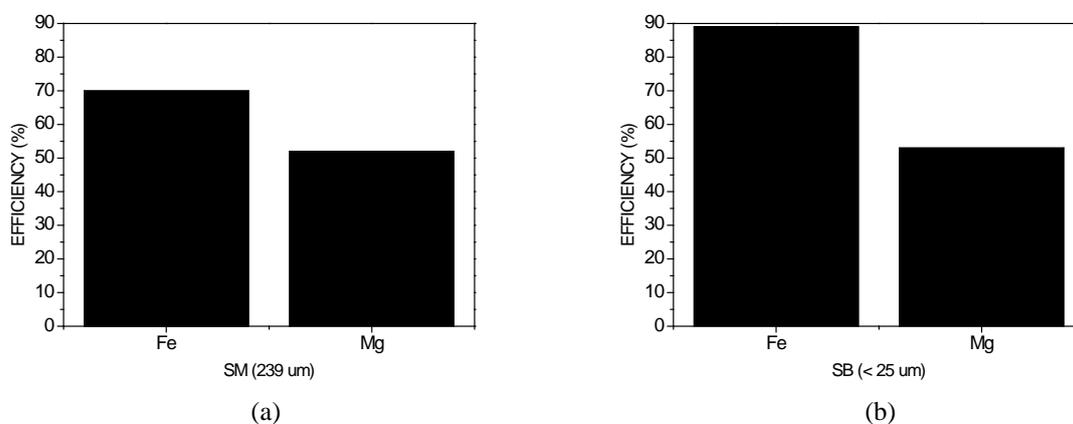


Figure 2. Extraction efficiency of both particle sizes: (a) SM - 239 µm (b) SB - < 25 µm

Source: The author

Looking at Fig. 2, the results of the efficiency of the acid dissolution process obtained in both particle sizes, showed that the extraction efficiency of Fe is superior to that of Mg. The Fe may have been extracted first because the sample had a higher concentration of silicon oxide, thus making it difficult to extract Mg through the pores in the silica matrix (LACINSKA et al., 2016; TEIR et al., 2007a).

Comparing the two samples, it is noted that the extraction of Mg did not suffer a significant variation when the granulometry of the raw material was modified. However, the extraction efficiency of Fe showed a difference of 19%. Thus, it is concluded that mechanical activation did not have a significant effect for the extraction of Mg, which may be due to its mineralogical complexity and its crystalline structure according to the article (ARCE et al., 2017). Therefore, it is concluded that the SM sample is the most viable to be used in the research, as it does not need to go through the process of particle size reduction, as was done for the SB sample and, consequently, an increase in the process cost (LI; HITCH, 2018).

4. CONCLUSIONS

According to the experiments, the effect of granulometry in the acid dissolution step was evaluated, considering two samples of serpentinites, denominated by SM (obtained in an open particle size range with an average diameter of 239 µm) and SB (narrow particle size below 25 µm). It was found that, although the SB sample was mechanically activated, there was no significant increase in the efficiency of Mg extraction indicating that the SM sample is the most viable to be used.

5. ACKNOWLEDGEMENTS

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