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Magnesium Extraction from Serpentine for Carbon Capture and Storage

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Abstract

This study aims to enhance availability of feedstock for mineral carbonation (MC) and evaluate viability of lizardite found in Pakistan as lizardite has a high reactivity compared to other ultramafic rocks. Leaching experiments were conducted to extract magnesium from heat-activated lizardite. Sulfuric acid (2% solution), hydrochloric acid (2% solution), and nitric acid (2% solution) were used in leaching experiments. The results showed that 71.8%, 65.2%, and 59% magnesium were extracted using H_2SO_4, HCl, and HNO_3 acids, respectively. Mg extraction was highest in H_2SO_4 dissolution as compared to HCl and HNO_3 dissolution. The leaching process was conducted at 25°C with 6 grams of solids using 2% acid solution for 5 hours. Similarly, silicon and iron were also extracted from heat activated lizardite. The findings suggest that lizardite can be a promising raw material for MC, though further studies on reaction kinetics and scaling potential are necessary.

Keywords: Acid Dissolution; Lizardite; Magnesium Extraction

1. Introduction:

Fossil fuels, which consists of 80-85% of current global energy use, are a significant energy resource. Fossil fuels provide several benefits such as plentiful availability, affordability, high energy concentration, convenience in use and storage and established infrastructure. This crucial energy source is encountering a difficulty because of the significant quantities of CO_2 being emitted into the environment during burning [1]. CO_2 emissions have a detrimental impact on the environment and human health. This results in glaciers ice melting and leading to severe weather occurrences [2, 3]. The atmospheric CO_2 content has increased to 422 parts per million (ppm) from 280 ppm during the preindustrial era [4]. Anthropogenic CO_2 causes global warming. Two degrees Celsius of warming would cause the release soil carbon [5]. There are three main approaches to reducing CO_2 emissions: employing energy-efficient technologies to reduce the amount of fossil fuels utilized; using renewable energy sources (such as solar, wind, biomass, or nuclear energy) that are not derived from fossil fuels; and utilizing carbon capture and storage (CCS) technology [6, 7]. CCS was first proposed in 1977 [8]. CCS is a chemical technique that captures and stores CO_2 [9]. There are four basic CO_2 sequestration strategies including ocean, geological, biological and mineral carbonation [10, 11, 12, 13 ,14, 15 ,16, 17]. MC process converts CO_2 into calcite, dolomite, magnesite, and siderite which are stable and this CO_2 cannot return back to atmosphere. Magnesium-based silicates ($xMgO\cdot ySiO_2\cdot zH_2O$) are frequently utilized in making cementitious materials because to their widespread availability [18,19].Magnesium silicates may be classified into many subgroups. Serpentine may be a viable raw material for MC as it has 40% MgO which is very reactive and available [20]. Direct carbonation and indirect carbonation are the two techniques for MC [21,22,23, 24]. The simplest technique for mineral sequestration is called direct carbonation, and it involves carbonating a suitable feedstock—for example, mineral sources high in calcium (Ca) or magnesium (Mg)—in a single step [25 ,26]. The two-step MC process is known as the indirect carbonation method. It indicates that in the first stage, the reactive component (Ca or Mg) is extracted from the minerals as an oxide or hydroxide, and in the second step, it reacts with CO_2 to produce stable carbonates [27, 28,29].

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One of the important factors for creating effective MC technology is to maximize Mg extraction from feedstock [30,31]. Magnesium is not found naturally due to its high reactivity and is abundantly presence in magnesite, periclase, asbestos, meerschaum, serpentine, talc, and epsomite [32]. Several studies have been conducted to extract magnesium from serpentine or other magnesium-rich rocks using acid leaching. Previously Tier et.al has achieved 70% Mg extraction using 74–125 μm serpentine with 2 M HNO_3 at 70 °C[33] . Sana et.al has attained 75% Mg extraction at 140 °C [34]. Acid-leaching is an easy way to extract magnesium, however high temperature dissolution is energy intensive and focus of the current study is to enable extraction process at atmospheric conditions. Amorphous silicate (SiO_2) is a solid residue that is left behind after the serpentine is leached [35]. The purpose of this study is to check the feasibility of lizardite found in Pakistan for mineral carbonation and make process operation at atmospheric conditions. There is no published study on magnesium extraction of lizardite, which is found in Pakistan. This research discusses acid dissolution of heat-activated lizardite at room temperature using nitric acid to investigate whether lizardite as a stock for MC to mitigate greenhouse gas emissions in Pakistan.

2. Material, methods, and experimental set-Up:

2.1 Lizardite:

A rock sample of Lizardite (almost five kg) was obtained from the Baluchistan area in Pakistan (Figure 1). Initially, Lizardite was crushed into small pieces by using hammer and Jaw crusher and then these small pieces were fed in a Blake jaw crusher to obtain a 1-2 cm diameter. Material was put in a ball mill for 2 hours to yield sub 75 µm fraction. Sieve analysis is performed using a sieve shaker (mesh sizes respectively used 0.5 mm, 0.350 mm, 0.150 mm, and 0.75 mm.

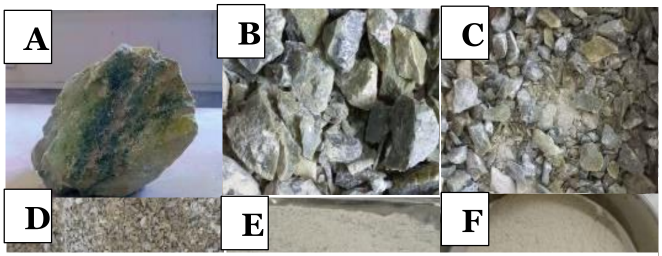

Figure 1: Picture of lizardite before heat activation , (A): Lizardite in rock form, (B): small pieces of lizardite after hammering , (C): After Jaw crusher , (D): After Blake jaw crusher ,(E): After ball mill ,(F): After sieve analysis

2.2 Heat activated Lizardite:

Lizardite (approximately 70 g) is subjected to heat activation in muffle furnace at 630 ºC for a duration of 4 hours. At 630 °C, most of the lizardite is converted into amorphous phase while at higher temperatures enstatite formation starts. Lizardite was converted into more reactive amorphous phase through heat activation. Heat activation is a technique that releases water vapor from the sample by destabilizing hydroxyl groups associated within the mineral matrix. This results in the de hydroxylation of serpentine minerals .It has been observed that 635 °C is the ideal temperature for lizardite to decompose, and enstatite formation starts above this temperature [36].

2.3. Acid dissolution experiments:

Acidic dissolution experiments were carried out in a lab using a 1000 ml beaker. Mixing was performed at room temperature by using a magnetic stirrer. The stirring speed was 800 rpm. The leaching parameters that were studied and their corresponding value ranges are shown in Table 1. Leaching experiments were conducted using nitric acid and examined the impact of time on the leaching. Six gram heat-activated Lizardite sample and 200 ml of a diluted solution of nitric acid were mixed in a beaker corresponding acid-to-solid ratio of 33:1 and agitated using a magnetic stirrer. After a specific amount of time of leaching, the agitation was stopped and the separation of solid and liquid was carried out using filter paper. Leaching residue was subsequently rinsed with distilled water, dried at 100°C for 24 h, and then examined by XRF equipment to determine elemental composition. Mg extraction yield was calculated according to XRF analysis results using equation 1.

$$Yield, \% = \frac{(F*f - R*r)}{F*f} * 100 \qquad (1)$$

Where, F is the quantity of feed (starting) material (g), f is the Mg content of feed (starting) material (wt.%), R is the quantity of solid leaching leftover (g), and r is the Mg percentage of solid leaching residue (wt.%).

Table 1: Conditions of leaching experiments

AcidConcentration (wt%)	Leaching Time (hours)	Sample Quantity (g)	T(ºC)
2	1	6	25
2	2	6	25
2	3	6	25
2	5	6	25

2.4 Material characterization:

Particle size distributions were analyzed using BT-9300ST laser particle size analyzer. XRD analyses were carried out using a diffractometer on Lizardite dried powder with Cu radiation before and after heat activation to determine mineralogical composition of samples. Elemental composition of samples were performed using X-ray fluorescence spectroscopy.

3. Results and discussion:

Particle sizes in the feed lizardite sample ranged from 1 to 80 micrometers (shows below in Table 2). The term "d_{50}" indicates that 50% of the particle volume is less than the size of 10.8 µm. Similarly, "d_{90}" signifies that 90% of the particle volume is smaller than the size of 48.28 µm, while "d_{99}" indicates that 99% of the particle volume is smaller than the size of 75 µm (shows below in table 2 and figure 2). The x-axis represents particle sizes in micrometers (µm), while the y-axis represents cumulative volume percentages of particles (Figure 2).

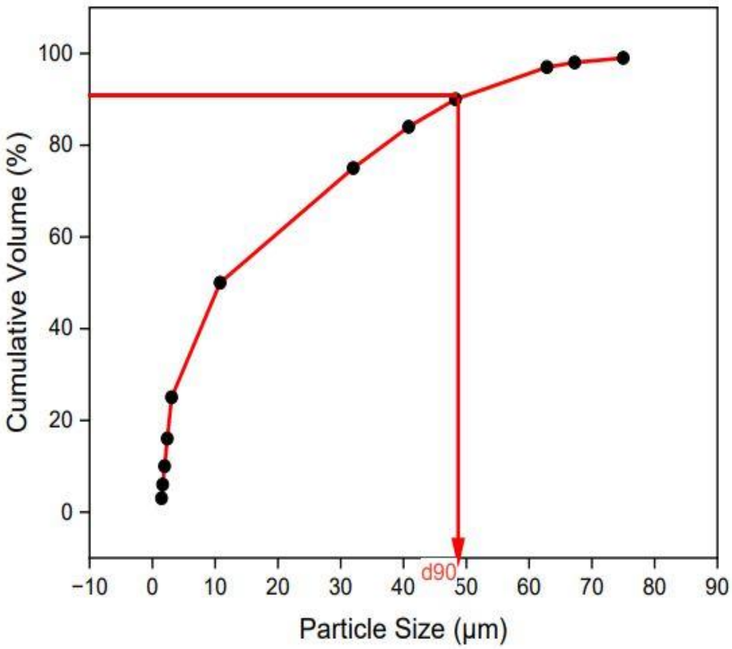

Figure 2: Particle Size Analysis(µm)

Table 2: Particle size Analysis of Sample

Particle diameters	**d_{3} µm**	**d_{6} µm**	**d_{10} µm**	**d_{16} µm**	**d_{25} µm**	**d_{50} µm**	**d_{75} µm**	**d_{84} µm**	**d_{90} µm**	**d_{97} µm**	**d_{98} µm**	**d_{99} µm**
	1.472	1.658	1.955	2.373	3.061	10.8	32	40.82	48.28	62.84	67.26	75

The X-ray diffraction patterns of ground Lizardite (Sample 1) and heat-activated Lizardite (Sample 2) are shown in Figure 3.

The XRD analysis of feed and heat-activated Lizardite revealed that the samples included mostly Lizardite [$Mg_3Si_2O_5(OH)_4$], with small quantities of talc [$Mg_3Si_4O_{10}(OH)_2$] and clinochlore [$Mg_5Al(AlSi_3O_{10}OH)_8$]. XRD analysis of Lizardite subjected to heat-activation demonstrates that Lizardite completely transformed into a more reactive amorphous form.

Elemental Composition of raw Lizardite identified by XRF analysis is reported in Table 3. The findings indicated that the sample include high proportion of Si and Mg (74.21 wt.%, in total) demonstrating the existence of serpentine minerals. The sample includes also minor quantity of iron approximately 8.81wt.%.

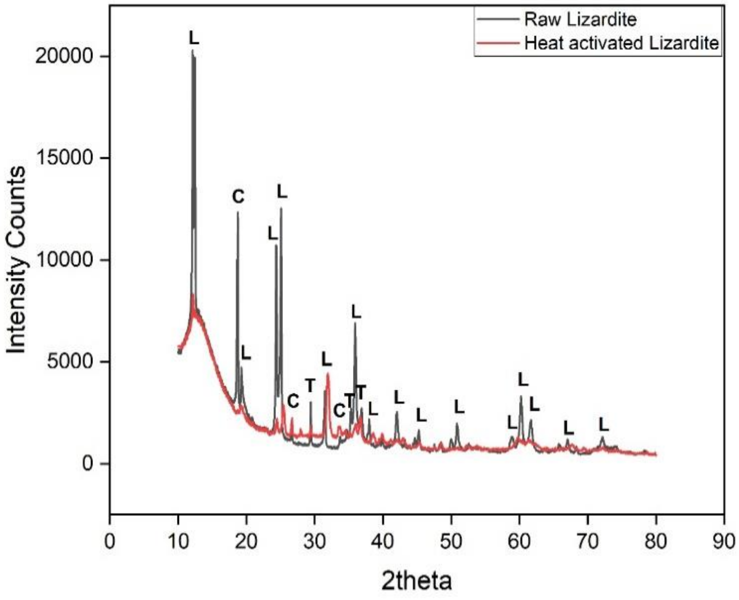

Figure 3: XRD pattern of Raw and heat-activated of Lizardite. L = Liz C = Clinochlore, T = Talc

Table 3: Elemental Composition of Sample from XRF analysis

Comonent	**Mg**	**S Si**	**Fe**	**Ni**	**Al**	**Mn**	**Ca**	**Ti**	**Na**	**K**
Content (wt.%)	34.95	39.26	8.81	0.24	2.42	0.10	0.10	0.10	0.19	0.01

Heat-activated (630 °C, 4 h) sub-75 µm feedstock was used for Mg-leachability experiment. High magnesium is extracted from heat-activated Lizardite compared to raw Lizardite [37]. The trend of Mg extraction in H_2SO_4, HCl and HNO_3 dissolution increased with time. Magnesium extraction was 62.56%, 64.35%,67.4%, and 71.83% after 1 hour,2hours, 3 hours, and 5 hours of H_2SO_4 dissolution at atmospheric conditions. Mg extraction efficiency (71.83% in H_2SO_4) aligns with previous studies on heat-activated serpentine, where values ranged from 65% to 78%[22]. Previously Tier et.al has achieved 70% Mg extraction using 74–125 µm serpentine with 2 M HNO_3 at 70 °C [33]. Sana et.al has attained 75% Mg extraction at 140 °C [34]. Acid-leaching is an easy way to extract magnesium, however high temperature dissolution is energy intensive and focus of the current study is to enable extraction process at atmospheric conditions. After 1 hour, 2 hour, 3 hours, and 5 hours of HCl dissolution, magnesium extraction was 54.47%, 56.8%,59.12%, and 65.19%, respectively. Mg extraction during 1 hour, 2 hour ,3 hours, and 5 hours of HNO3 dissolution was 46%,49%, 54%, and 59%, respectively. The acids concentration, solid amount, and leaching temperature were all maintained constant at 2%, 6 gram heat activated Lizardite, and 25 oC, respectively, while the influence of leaching period was investigated. This finding indicates that Lizardite could be used as a feedstock for MC if Lizardite is properly heat activated.

The silicon extracted from heat-activated Lizardite is high compared to raw Lizardite, similar to the extraction of magnesium [37]. After one hour, 2 hours,three hours, and five hours of HNO3 dissolution, silicon extraction was 49%,50.59%, 52%, and 55%, respectively. Similar to HNO3 dissolution, silicon extraction for H2SO4 and HCl dissolution were 59.4%, 62.8, 67.68%,72.53, and 54.45%, 62.58%, 70.31%, 74.99%, respectively. Silicon extraction was highest in HCl dissolution as compared to H_2SO_4 and HNO_3 dissolution.

Iron extracted from heat-activated Lizardite was 88%, 90%,92%, and 95%, with corresponding leaching times of 1 hour,2 hours, 3 hours, and 5 hours for HNO_3. Similar for H_2SO_4 and HCl dissolution iron extracted were 94.131%, 95.7%,96.98%, 98.25%, and 97.711%, 98.25%,98.98%, and 99.38%, respectively. High iron efficiencies are due to Fe_2O_3 solubility. Figures 4,5,6 show magnesium, silicon and iron that were extracted during a 5-hour dissolution, respectively.

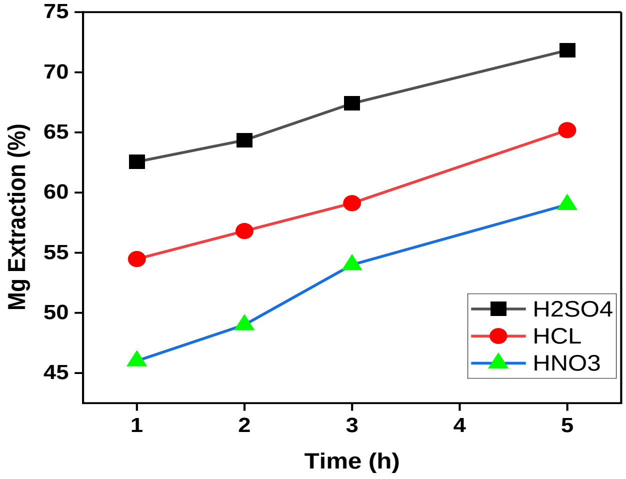

Figure 4: Mg extracted from sub-75 µm heat-activated (630 0C,4h) Lizardite

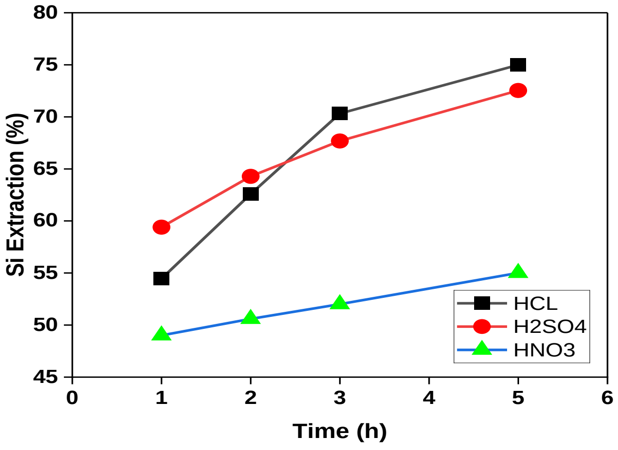

Figure 5: Si extracted from sub-75 µm heat-activated (630 ^{0}C,4h) Lizardite

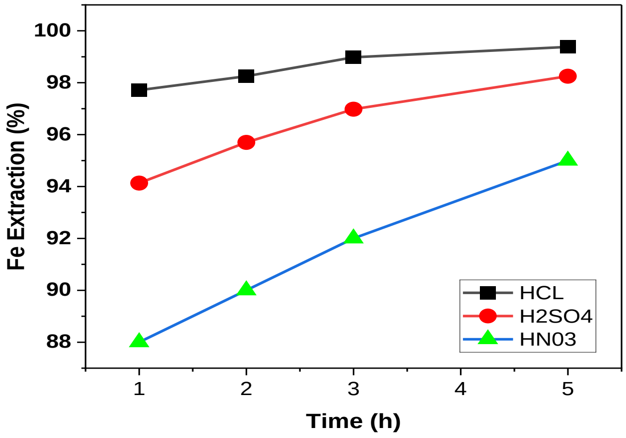

Figure 6: Fe extracted from sub-75 µm heat-activated (630 ^{0}C,4h) Lizardite

4. Conclusions:

Heat-activated Lizardite can be used as a stock for MC process. XRF revealed that Mg and silicon are present in high proportion in the Lizardite sample. The dissolution of Mg from lizardite in H_2SO_4, HCl and HNO_3 acid using a glass beaker and magnetic stirrer was investigated successfully. Mg extraction using H_2SO_4, HCl, and HNO_3 acid were determined to be 71.83, 65.19, and 59% during 5 hours of dissolution. Mg extraction was highest in H_2SO_4 dissolution as compared to HCl and HNO_3 dissolution. The trend of Mg extraction in H_2SO_4, HCl and HNO_3 dissolution increased with time.

5. Challenges of MC:

Energy intensive pretreatment is main challenge of MC as heat-activation for long durations requires significant amount of energy. Recently Lewis et.al (2025) has proposed high flux radiation thermal treatment for 480 s which requires considerable small amount of energy for heat-activation and corresponding Mg extractions are higher compared to traditional heat-activation methods paving the way for large scale carbon sequestration.

Conflict of 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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