Isolation Mg(OH)$_2$ from Dolomite with Extraction Process used HNO$_3$

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Abstract. Dolomite MgCa(CO$_3$)$_2$ has not been used to the fullest in Indonesia, where the natural resources of dolomites are quite abundant and available in very large quantities. Mg(OH)$_2$ isolated of dolomite originated from the East Java region with the extraction process using nitric acid. Using variable time and temperature, to acquire the element Mg(OH)$_2$. To get the greatest Mg(OH)$_2$, the precipitation process from using NaOH, and isolating Mg(OH)$_2$ from the Elements of Ca(OH)$_2$ and other impurities with Na EDTA solutions. From research obtained the best condition Mg(OH)$_2$ from dolomite with NaOH additions with a variable stirring time for 5 hours and a temperature of 45 °C is at 175.60 mg/L.

Keywords: dolomite, extraction process, nitric acid, isolation

1. Introduction

Dolomite MgCa(CO$_3$)$_2$ is one of the natural resources that have not been used to the fullest in Indonesia, dolomite mutilated limited fertilizer manufacture for the plant, and bricks for building materials. Dolomite Natural resources in Indonesia are quite abundant and available in very large quantities, ranging from the provinces of Aceh to Papua, namely the provinces of North Sumatra, West Sumatra, Central Java, East Java (Madura Island) and Papua. Dolomite is a mineral that is widely used in the industry, namely as a flux substance in metallurgy (glass and ceramic industry), as a filler material in several industries (paper, rubber, and plastic) produced, absorbent exhaust gases, and filters for water treatment. In the chemical industry, dolomite is a source of magnesium that reacts with oxide, hydroxide, calcium bases, and carbonates, thereby forming a compound of magnesium oxide, magnesium hydroxide, and magnesium carbonate [1]. Dolomite rocks haven't economical value, since dolomite purely theoretically has 45.6% MgCO$_3$ or 21.9% MgO and 54.3% CaCO$_3$ or 30.4% CaO [2].

Dolomite rocks mutilated only as building materials and agricultural fertilizers (dolomite fertilizer) with a very low selling value of IDR 500 every kg. Though if we will use as a magnesium carbonate product that we will apply in the pharmaceutical industry and paper industry, it can increase the economic value is high enough [3]. Dolomite is one of the natural rocks based on mineral carbonates, such as limestone, calcite (CaCO$_3$), and magnesite (MgCO$_3$). Dolomite has a chemical formula CaMg(CO$_3$)$_2$, white, it is also greyish, bluish and light yellow color, has a type between 2.8 until 2.9 g/ml and is soft (hardness only 3.5 until 4 Mohr scale) and easily absorbs water [4], [5].
Magnesium extracted and calcium increases, with the increased concentration of acids and the temperature of the dissolving process. We will achieve at a temperature of 30 °C with a concentration HCl 4 N [6] for 5 hours with calcium and magnesium extract amounting to 60.31% and 25.79%. The dissolving process will be done against 20 grams of dolomite samples with an acid concentration HCl 1 N until 4 N at a temperature of 30 °C until 90 °C [7], dissolving using KOH 4 N and solvent HNO₃ rate of Ca and Mg in Dolomite Rock by 29.69% and 13.85% [8].

In this study, Mg(OH)₂ isolated of dolomite came from the East Java region with the extraction process of HNO₃ using a variable time and temperature, since dolomite has a larger Mg element than the Ca element, so to acquire an Mg(OH)₂ greater determined by Mg elements solubility in HNO₃ addition solvent in the extraction process to get Mg(OH)₂ it is necessary to do precipitation with NaOH solution, and isolate Mg(OH)₂ of the Elements Ca(OH)₂ as well as other substances with a Ca-EDTA solution.

2. Extraction Process

The physically dense-liquid extraction process of the dissolved components into the banishment, where the dissolved components are returned to the original form without any chemical changes, the solid-liquid extraction process can be performed if the material is soluble in the extraction solvent, the continuous process is required when the solids are only slightly soluble in the solvent but are often also used in solids that have high solvent effectiveness, HNO₃ 4N solvent concentrations with pH 11-12 the particle size of dolomite 200 mesh with a mass of 20 grams [9]. The extraction process it takes a filtration process, where filtration is a process of separation of solid-liquid materials, based on its type consisting of gravity and vacuum filtration, for the gravity filtration used glass funnel and filter paper, based on the natural gravitational style, for example, gravity filtration consists of filter cones, fluted filters, pipette filtration, heat gravity filtration, and decantation. Vacuum filtration by pouring a mixture that will be separated through a filtered paper in the Buchner funnel or the Hirsch funnel and its solids will be left in a filter paper while the liquid will be attracted to the flask due to vacuum pressure, for example, Buchner Funnels and Hirsch Funnels [10].

Dolomite rocks were scaled down to 325 mesh, after the size of the 325 mesh dolomite powder, sampled 10 grams of dolomite powder for analysis of raw materials in the form of magnesium levels, dolomite powder that has been analyzed, taken 10 grams reconstituted with HNO₃ 4N is inserted in the beaker glass 500 ml with stirring 400 rotaries per minute, in the extraction time variables: 2, 3, 4, 5, and 6 (hours) and variable temperature: 30, 35, 40, 45 and 50 (°C). Then deposited with NaOH 4N in each time and temperature variable, after the deposition process is inserted EDTA, deposits are formed separated by filtered and washed with H₂O, deposits formed are Mg(OH)₂ and filtrate form CaEDTA. Deposits containing Mg are reconstituted with the addition of 30 ml HNO₃ 4N. The addition of HNO₃ 4N was discontinued when the total dissolved deposits, the formed solution was transferred into the 100 ml measuring flask and added H₂O to a volume of 100 ml, taken 5 ml to be analyzed with the Atomic Absorption Spectrophotometer (AAS) [11].

The factors affecting the extraction process are the concentration of reagent, temperature, time, and stirring. The effect of reacting concentrations on the extraction process, the more H + ions react so the more magnesium is formed, the faster the reaction progresses, if the concentration is reduced, then H + ions will be slightly and the reaction rate will also decrease. Increased magnesium extract is increasingly higher, as acid addition can increase the rate of reaction speed and the diffusion rate of H + ions. Effect of temperature in the extraction process, the number of particles that react more and move faster at low temperatures [12], [13]. The high temperature of kinetic energy particles will be greater. The results of the impact of temperature experiments on the percent of magnesium and calcium extracted, i.e. percent of extractions increased by rising temperatures. The length of the extraction time affects the extract volume obtained. The longer the extraction time the longer the contact time between the solvent and raw materials, so the more dissolved substances are contained [14]. With stirring, it can increase the speed of mass transfer of the particle surface into the solution
and prevent precipitation. Stirring speed is done to determine the stability of calcium and magnesium extracted [15].

The mechanism of dissolving dolomite with HNO3 4N as follows [16]:
\[
\text{CaMg(CO}_3\text{)}_2\text{(s)} + 4\text{HNO}_3\text{(aq)} \rightarrow \text{Ca(NO}_3\text{)}_2\text{(aq)} + \text{Mg(NO}_3\text{)}_2\text{(aq)} + 2\text{H}_2\text{O(l)} + 2\text{CO}_2
\]

After the dissolving process will cause deposits, processed by adding NaOH 4N, and filtrate separated from liquids, so that the reaction occurs as follows:
\[
\text{Ca(NO}_3\text{)}_2\text{(aq)} + \text{Mg(NO}_3\text{)}_2\text{(aq)} + 4\text{NaOH} \rightarrow \text{Ca(OH)}_2\text{(s)} + \text{Mg(OH)}_2\text{(s)} + 4\text{NaNO}_3\text{(aq)}
\]

Mg(OH)\text{2 and Ca(OH)2 in the form of solids and not separated from each other, the solids in the form of deposits are separated with NaEDTA with the following reactions}
\[
\text{Ca(OH)}_2\text{(s)} + \text{Mg(OH)}_2\text{(s)} + \text{NaEDTA(aq)} \rightarrow \text{CaEDTA(aq)} + \text{Mg(OH)}_2\text{(s)} + 2\text{NaOH(aq)}
\]

3. Results and Discussion

Based on Table 1 it can be seen that at the time extraction of 2 hours with a variable temperature 40 °C obtained a maximum Mg(OH)\text{2 rate of 123.693 mg/L. At the beginning of the temperature of 30 °C has a higher rate, this is due to the greater the concentration of reagent, the more H\text{+ ions react so the more Mg(OH)2 is formed, thus, the faster the reaction progresses, the increased Mg(OH)2 is extracted increasingly higher, increasing the rate of reaction speed and the rate of diffusion of H\text{+ ions. The five variables the extraction time indicates the extraction temperature of 45 °C indicates the maximum Mg(OH)2 concentration [9].}

| No. | Extraction time (hours) | Extraction temperature (°C) | Concentration Mg(OH)\text{2 (mg/L)} |
|-----|-------------------------|-----------------------------|--------------------------------------|
| 1   | 30                      | 164.03                      |
| 2   | 35                      | 63.12                       |
| 3   | 2                      | 64.91                       |
| 4   | 45                      | 123.69                      |
| 5   | 50                      | 106.07                      |
| 6   | 30                      | 81.04                       |
| 7   | 35                      | 35.75                       |
| 8   | 3                      | 120.20                      |
| 9   | 45                      | 164.81                      |
| 10  | 50                      | 156.99                      |
| 11  | 30                      | 198.11                      |
| 12  | 35                      | 68.68                       |
| 13  | 4                      | 71.33                       |
| 14  | 45                      | 129.86                      |
| 15  | 50                      | 80.75                       |
| 16  | 30                      | 175.41                      |
| 17  | 35                      | 85.46                       |
| 18  | 5                      | 64.91                       |
| 19  | 45                      | 175.60                      |
| 20  | 50                      | 91.36                       |
| 21  | 30                      | 225.84                      |
No. | Extraction time (hours) | Extraction temperature (°C) | Concentration Mg(OH)_2 (mg/L) |
---|------------------------|-----------------------------|-------------------------------|
22  | 35                     | 58.00                       |
23  | 6                      | 103.06                      |
24  | 45                     | 111.01                      |
25  | 50                     | 81.26                       |

**Table:** Relationship between temperature and concentration of Mg(OH)_2.

**Figure 2.** Graph of relationship between temperature with concentration Mg(OH)_2.

In Figure 2 temperature 45 °C, the number of particles reacts more and moves faster than at room temperature, this is due to the temperature of 45 °C the kinetic energy of the particles will be greater. Results of the study of the influence of temperature against the concentration of magnesium and calcium extracted, at a temperature of 50 °C the concentration began to decline, caused due to the level of saturation of the solution. The length of extraction time affects the volume of extracts obtained, 5 hours of extraction time indicating contact time between the solvent and dolomite more maximum after a 6-hour extraction time showed a decrease in the concentration of Calcium and Magnesium. With stirring can increase the speed of mass transfer of the particle surface into the solution and prevent precipitation. Stirring speed is done to determine the stability of Calcium and Magnesium extracted [15].

**4. Conclusion**

The best condition of concentration Mg(OH)_2 from dolomite with the HNO_3 extraction process, as well as precipitation process with the addition of NaOH, isolation Ca(OH)_2 with NaEDTA, with a time variable for 3 hours and a temperature of 30 °C obtained concentration Mg(OH)_2 as much as 175.60 mg/L.

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