Comparison of Dolomite Crystal Structure, Calcinations Dolomite and Magnesium Hydroxide in Partial Calcinations and Slaking Process

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Abstract. Dolomite is a mineral that consists of calcium carbonate and magnesium carbonate with various mole ratio depend on the formation of mineral source. Recently, Utilization of dolomite only used as raw material for fertilizer and building materials, so that the enhancement of its added value becomes low. If the components in dolomite can be separated, magnesium carbonate and calcium carbonate will be produced then provide high added value. To separate these two components in dolomite is through partial calcinations followed by slaking process. The purpose of this paper is to prove that the partial calcinations can be used as a fundamental process for calcium and magnesium separation process in dolomite. SEM-EDX and XRD analysis proved that partial calcinations at 675°C for 6 hours is able to produce magnesium oxide (MgO) and calcium oxide (CaO). Then sea water was added to calcinations product so magnesium hydroxide and calcium carbonate that easily separated by sea water. The weakness of partial calcinations process at 675°C and processing time 2 hours is the dolomite has not perfectly reacted yet. XRD analysis showed that MgCO₃·CaCO₃ compounds still exist, so there is a possibility that magnesium was not fetched after the separation process.

1. Introduction
Dolomite is a mineral that consists of calcium carbonate and magnesium carbonate with various mole ratio depend on the formation of mineral source [1]. Gresik is one place that produce dolomite with a very good quality and has a potentially to be processed to become advanced materials [2-3]. One of advanced material product that developed from dolomite is magnesium carbonate with an ultrafine grain size and high purity [4]. It obtained from dolomite processing which started from dolomite calcinations, slaking process, carbonation, separation process between calcium and magnesium and the final step is magnesium carbonate formation [5].

Separation process between calcium and magnesium is an important role in making magnesium carbonate from dolomite. This process uses carbon dioxide from the result of calcinations process so it makes this process environment friendly. Separation process between magnesium and calcium from dolomite with carbonation process will be successful if in calcinations process, the calcium carbonate does not change into calcium oxide (CaO) [6]. In this research, calcinations process at low temperature
will be done. This purpose of this research is to transform magnesium carbonate into magnesium oxide without transform calcium carbonate into Calcium Oxide [7].

2. Experimental

2.1. Raw Materials
Raw material that used in this study was dolomite powder from PT. Polowijo Gosari. Based on XRF analysis, other oxides that contained in the raw material are CaO, MgO, Na₂O, Fe₂O₃, K₂O, SiO₂, and Al₂O₃. XRF analysis can be seen in Table 1.

| No | Oxides          | % wt |
|----|-----------------|------|
| 1  | Calcium (CaO)   | 61.83|
| 2  | Magnesium (MgO)| 25.75|
| 3  | Sodium (Na₂O)   | 7.93 |
| 4  | Silica (SiO₂)   | 1.19 |
| 5  | Aluminium (Al₂O₃)| 0.84|
| 6  | Potassium (K₂O)| 0.40 |
| 7  | Iron (Fe₂O₃)    | 0.37 |

Table 1 show that CaO content in dolomite is higher than MgO content. Calculations by Stoichiometry on XRF analysis showed that dolomite consisted of 61.83 % wt CaO and MgO 25.73% (wt) and it mole ratio is 1: 0.33. It indicates that calcium oxide is released from magnesium oxide.

2.2. Experimental Procedure
This experiment was done to determine the differences between the dolomite structure, the results of dolomite calcinations and the results of the hydration process. Dolomite was crushed until it has -60 mesh and calcinations the dolomite at 625°C, 650°C and 675°C for 1 hour to 6 hours. Then sea water was added to the calcinations in slaking process in order to transform magnesium oxide (MgO) into magnesium hydroxide (MgOH). Magnesium hydroxide can be easily separated with calcium carbonate after carbonation process because in this process magnesium hydroxide will change to become soluble magnesium bicarbonate. The optimum results were analysed by XRD, XRF and SEM to know that calcium oxide was not produced. Flow diagram of this experiment can be seen in Figure 1.
3. Results and Discussion

3.1. Calcinations Process

Calculation of the weight reduction in the dolomite calcinations at 625°C, 650°C and 675°C for 1 to 6 hours can be seen in Table 2.

Table 2. Sample weight lost during calcinations (% wt)

| Time (Hour) | 625°C | 650°C | 675°C |
|-------------|-------|-------|-------|
| 1           | 10.79 | 13.99 | 14.82 |
| 2           | 14.62 | 17.74 | 21.29 |
| 3           | 19.69 | 22.79 | 28.86 |
| 4           | 26.07 | 29.17 | 31.07 |
| 5           | 28.95 | 31.48 | 31.62 |
| 6           | 31.58 | 31.89 | 32.09 |

Table 1 show that the content of MgO is 25.75%, it can be estimated that 28.0923 %wt will be lost during partial calcinations process. Based on that weight reduction, the maximum results in theoretical occur at 625°C for 5 and 6 hours. Table 2 shows the weight lost during calcinations process is caused by temperature and when the temperature at 675°C for 3 to 6 hours the partial calcinations process perfectly occurred. The highest temperature caused 28.86 %wt, 31.07 %wt, 31.62 %wt and 32.09 %wt of sample weight lost. In this study, samples at 675°C for 4 hours were taken subsequently. Results of experiments conducted by Eni Febriana show that the mass loss at temperatures 700°C and calcinations time for 6 hours is only 27.68 wt% dolomite [7].

3.2. XRD Analysis

XRD analysis of dolomite, calcinations dolomite and slaking process using sea water can be seen in Figure 2. It shows that the calcinations process is not running perfectly. There is still a peak of MgCO₃, CaCO₃ from calcinations process so partial calcinations process is not perfect. Consequently of partial calcinations process was not occur so that if CO₂ was streamed into hydration products, Mg and Ca cannot be easily separated. Peak of CaO from calcinations process and peak of calcium hydroxide (Ca(OH)₂) from slaking process were not appear in XRD analysis. Results of experiments conducted by Eni Febriana show that peak dolomite was formed at temperatures of 700°C and was not formed at temperatures of 800°C [7].

![Figure 2. XRD analysis on the dolomite, calcinations dolomite and slaking process](image)
3.3 SEM-EDX Analysis

EDX analysis of mineral dolomite shows that the amount of Ca and Mg are not always in same ratio so in dolomite not every calcium and magnesium bonded to form MgCO$_3$, CaCO$_3$ but there were loose in the form CaCO$_3$. Figure 3 shows that the oxygen distribution tends to spread more evenly than the carbon distribution in dolomite. This suggests that oxygen which bonded with carbon is also bound up with Ca and Mg, than it can be concluded that very difficult to make Ca and Mg in metal from dolomite.

![Figure 3](https://example.com/figure3.jpg)

**Figure 3.** The results of SEM - EDX mapping for the element of magnesium, carbon, calcium and oxygen in the dolomite, (a – d) before calcinations and (f – h) after calcinations.
By comparing the right image with the left in Figure 3, there is a reduction in the concentration of carbon and oxygen are also quite a lot. This indicates that the result of the calcinations process is the release of carbon dioxide (CO$_2$). Based on Figure 3, the dispersion of carbon and oxygen concentration in calcinations dolomite was slightly decrease compare to dolomite ore. It tells that partial calcinations process has occurs. The carbon dioxide was released and MgCO$_3$ was transformed into MgO, but CaCO$_3$ still attached with carbon.

SEM analysis on Figure 4 shows that the dolomite after calcinations has a porous surface. It caused by CO$_2$ released from dolomite grain and it proves that the dolomite calcinations reaction occurs not only on the surface of the dolomite but also in all parts of dolomite. From this result can be concluded that the grain size from calcinations process will not give an effect on the reaction kinetics. In the calcinations process it is required a large grain size so circulation of CO$_2$ gas will easily happened.

![Figure 4](image-url)  
(a) (b)  
Figure 4. The appearance of granular dolomite, which (a) is dolomite before calcinations process and (b) after calcinations dolomite.

4. Conclusion
From the experimental results on calcinations process, hydration and drying on a dolomite with partial calcinations can be concluded that calcinations process at 675°C for 4 hours is still partial calcinations process. Partial calcinations process makes separation process between magnesium and calcium in dolomite easy to do. However, calcinations at 675°C for 4 hours causing an imperfect calcinations reaction and characterized by the presence of peak MgCO$_3$CaCO$_3$ and it can reduce magnesium carbonate producing. Calcinations reactions of dolomite occur not only on the surface of the grains but also inside of the grain.

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