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Characteristic of the CaO-MgO Material Derived from Dolomite via Precipitation-Dehydration Method

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ABSTRACT. This research utilized dolomite rock ($CaMg(CO_2)_2$) to produce CaO-MgO material using precipitation-dehydration. The precipitation-dehydration method was carried out by dissolving dolomite in 10% hydrochloric acid (HCl) solvent and precipitating in 2 N sodium hydroxide (NaOH) solution at a temperature of 80 ºC. The resulting products were analyzed using Scanning Electron Microscope-Energy Dispersive X-Ray (SEM-EDX) and Brunauer-Emmett-Teller (BET). The acidity level of the solution was analyzed by using a pH meter. The analysis results using SEM-EDX show that the material has a characteristic chemical composition with a Ca/Mg ratio less than 1. The results of BET show that the average surface area, average pore volume, average pore size and average particle diameter are 71.1213 m^2/g, 0.1081 cc/g, 20.5165 nm, and 36 nm, respectively. The solution's acidity (pH level) in precipitation-dehydration is 8-8.5. It is identified that precipitation-dehydration can be used for CaO-MgO recovery and further application in catalyst materials.

1. INTRODUCTION

For the preparation of mixed oxides as solid base catalysts, dolomite, a typical rock-forming mineral composed of mixed calcium and magnesium carbonate ($CaMg(CO_3)_2$), is an inexpensive, non-toxic, and plentiful natural source [1]. After the carbonate groups have degraded, basic calcium oxide (CaO) and magnesium oxide (MgO) are produced, according to a study of temperature-controlled tests conducted on fresh dolomite [2].

CaO and MgO are required for the production of medications, slow-release fertilizer, fire-retardant materials, catalysts, etc. [3–6]. The most popular heterogeneous base catalyst is CaO, which is found in large quantities in nature, like limestone. In addition to limestone, other sustainable and efficient natural sources of calcium oxide include dolomite, goat bone, oyster, crab, egg, capiz, abalone, snail, and mussel shells [7]. Additionally, additional catalysts, like MgO, have been investigated for biodiesel synthesis in addition to CaO-based catalysts.

Dolomite extraction was used in this study to create the CaO-MgO mixture. In Indonesia, dolomite is abundantly accessible. Dolomite minerals are found in Madura, Papua, Central Java, East Java, and North and West Sumatra [8]. Dolomite were doped with sodium nitrate and calcined at 900 ˚C to produce CaO-MgO and Na-CaO-MagO mixed metal oxides for the catalyst of biodiesel production [9]. CaO-MgO from dolomite also generates for CO_2 sorbent by the calcination method at 950 ˚C [10]. Several applications of CaO-MgO have been reported. A key component of catalytic performance, CaO-MgO has been employed as a catalyst for the isomerization of glucose into fructose. It has been established that this kind of catalyst can be reused for a minimum of three cycles [5]. The transesterification of triglycerides with methanol to create biodiesel has also been reported to be possible using CaO-MgO as a solid base catalyst [1].

From the literature study, the calcination method has been carried out to extract CaO-MgO from dolomite. The calcination method still has several shortcomings, such as high energy requirements, the risk of forming unwanted compounds, and the potential for physical degradation of raw materials. Therefore, in this study the precipitation-dehydration method was used to overcome the shortcomings of the calcination method. By adding alkali (sodium carbonates, sodium hydroxides, lime, etc.), calcium and magnesium are precipitated out of brines and seawater.

Because Ca and Mg are in a fragile equilibrium, precipitation could result from even small changes in alkalinity and carbon dioxide tension [11]. In this work, we used existing dolomite rock as a cheap and environmentally favourable catalyst to perform precipitation-dehydration, which has not been reported before. The precipitation-dehydration method establishes the overall amount of CaO-MgO recovered from the dolomite mineral and should disclose the properties of the final product. The effect of dehydration temperature to the characteristic of CaO-MgO was systematically investigated. The properties of CaO-MgO were observed by Scanning Electron Microscope-Energy Dispersive X-Ray (SEMEDX), Brunauer-Emmett-Teller (BET) and acidity level using pH meter.

2. MATERIALS AND METHODS

The CaO-MgO in this research was prepared from natural dolomite using the precipitation-dehydration method, as seen in Figure 1.

2.1 The Precipitation-Dehydration Method

The precipitation-dehydration method involves reducing the size of dolomite to 100 mesh. 50 g of dolomite was dissolved with 460 mL of 10% hydrochloric acid (HCl), stirred for 60 minutes and filtered to separate the remaining dolomite. The filtrate is subjected to precipitation by slowly adding 2 N sodium hydroxide (NaOH) solution until it reaches an acidity of 10-10.5. The solution was filtered to get calcium-magnesium hydroxide ($CaMg(OH)_4$) solid and washed using distilled water to separate sodium chloride (NaCl). The washed solid is dried and dehydrated at 400, 550, 700 and 800ºC for 3 h with an air furnace. The dehydrated product was then analyzed using the SEMEDX, BET and acidity level.

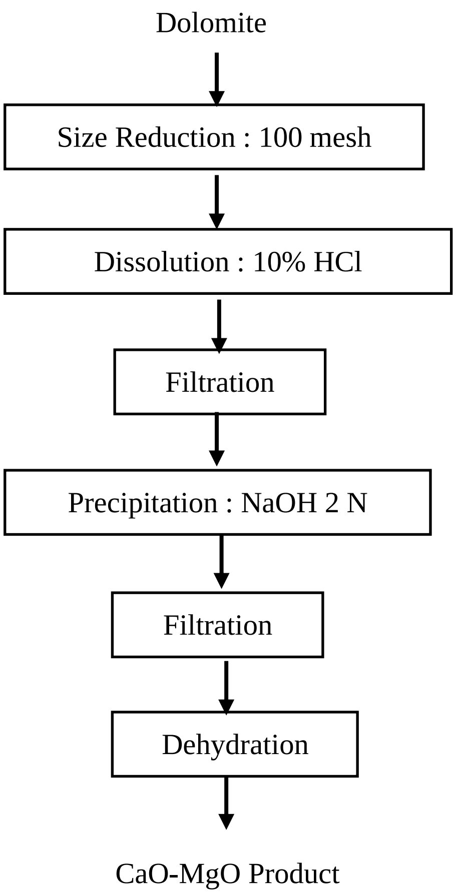


Figure 1. Precipitation-Dehydration Method Diagram

2.2 Characterization

The CaO-MgO obtained are characterized using Scanning Electron Microscope-Energy Dispersive X-Ray (SEM-EDX) to determine the chemical composition and morphology, and BET (Brunauer-Emmett-Teller) to determine surface area, pore volume, pore size (pore diameter), and calculated particle diameter from surface area using Equation 1. The acidity level of the CaO-MgO obtained are analyzed by dissolving 5 g of the CaO-MgO product with 100 mL of distilled water.

$$\mathbf{S} = \frac{\mathbf{6}}{\mathbf{\eta D}} \tag{1}$$

with S = surface area, η = density, and D = particle diameter.

3. RESULTS AND DISCUSSION

3.1 Chemical Composition

The chemical composition of the dolomite raw material used in this research was analyzed using SEM-EDX as shown in Table 1. The SEMEDX results showed that the main component of the dolomite was oxygen, followed by calcium and magnesium, then trace amounts of aluminium and chloride. The ratio of calcium/magnesium (Ca/Mg) was found to be 2.4. Then, the dolomite is categorized as calcareous dolomite [5]

Based on SEM-EDX analysis, dolomite's chemical composition of dolomite after precipitation-dehydration processes is depicted in Table 2. It shows that the chemical composition of CaO-MgO obtained is significantly influenced by dehydration temperature. The percentage of oxygen content of the CaO-MgO is less than 1. This can be attributed to the chloride ions from the solvent used for the dissolution process. As the solubility of the Ca ions is higher than that of Mg ions. During precipitation, Ca ions are more difficult to precipitate, resulting in less Ca content in the material obtained.

MgO is more thermally stable than CaO. A high Mg ratio results in a low Ca/Mg ratio, making it suitable for catalyst applications that are more resistant to sintering at high temperatures. Therefore, catalysts with a high MgO content are better suited for high-temperature reactions, such as reforming or mild cracking.

Table 1. Chemical composition of the raw dolomite

Element	Weight (%)
Calcium (Ca)	29.61
Magnesium (Mg)	12.34
Aluminium (Al)	1.30
Chloride (Cl)	0.31
Oxygen (O)	56.44

Table 2. Chemical Composition of the CaO-MgO after Precipitation-Dehydration Process

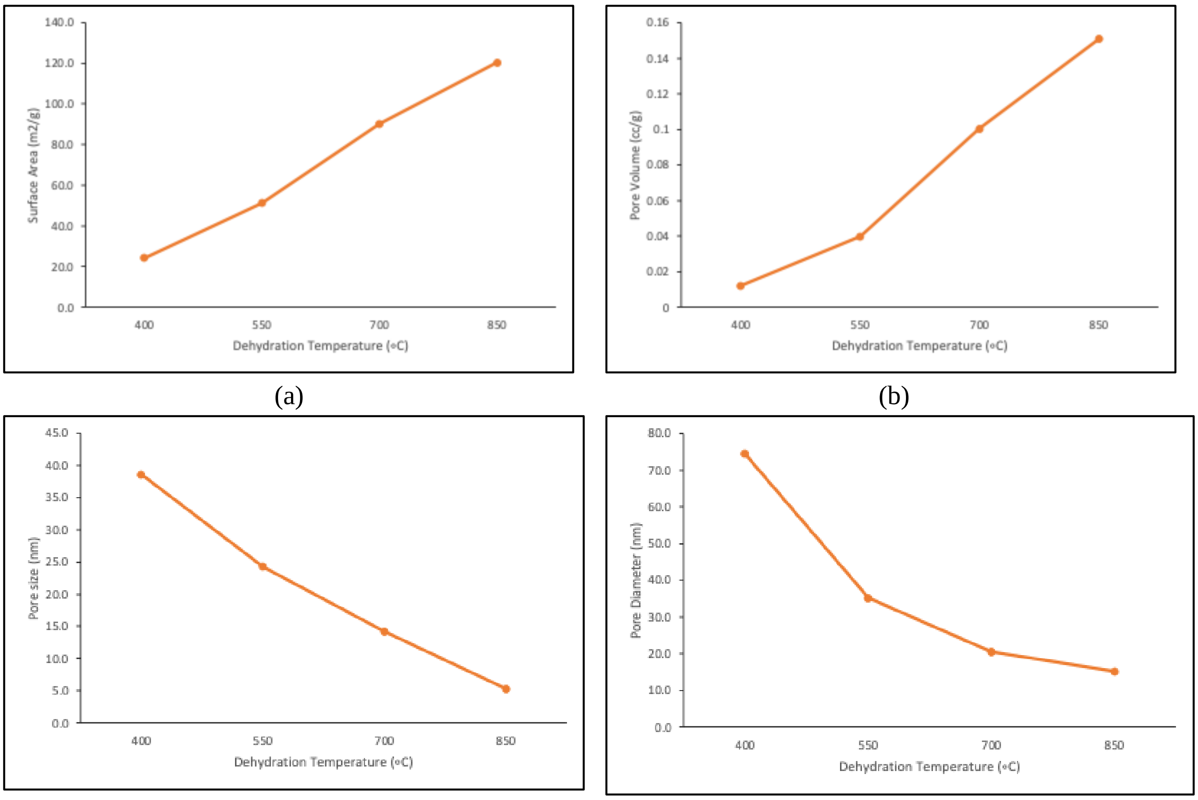
Dehydration temperature of dolomite (C)	Ca (%)	Mg (%)	Ca/Mg
400	10.78	37.44	0.288
550	9.15	37.36	0.245
700	7.49	38.47	0.195
850	6.69	39.55	0.169

3.2 BET Analysis

The BET analysis showed the surface area, pore volume, pore size, and particle size in Figure 2. It shows the characteristics of CaO-MgO after the precipitation-dehydration process.

Generally, sorption capacity depends on the degree of porosity development, high surface area, and pore volume. The surface area and pore volume of 850 ^{o}C dehydration temperature showed the largest result. In contrast, the pore size and particle diameter of 850 ^{o}C dehydration temperature showed the smallest result. Based on the results of the analysis using BET, it shows that CaO-MgO obtained has an average surface area of 71.1213 m^2/g, Another study has a similar result, reporting that CaO-MgO materials have surface areas ranging from 30.99 to 50.72 m^2/g, indicating its comparatively good pore structure, which can provide better adsorption sites for reactants [12].

This is obviously attributed to the sintering effect [13]. Increases in surface area show that the product re-arrangement has taken place and that the impregnation procedure was successful. A larger surface area can provide additional active pore pathways and improve the interaction between reactants and active sites, which produces better products. On the other hand, only tiny molecules can be adsorbed if the pore diameter is small [14].



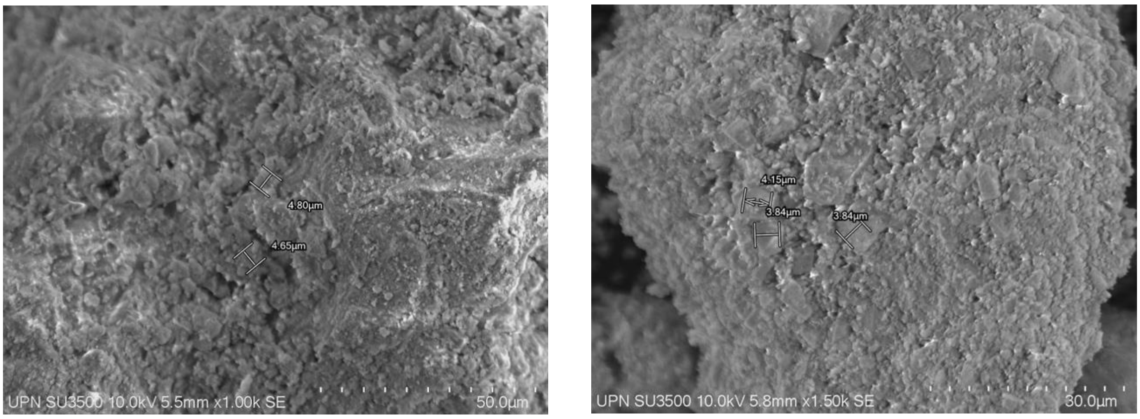
(a) (b)

(c) (d)

Figure 2. BET Analysis of CaO-MgO (a) Surface Area (b) Pore Volume (c) Pore Size (d) Particle Diameter

3.3. Morphology Analysis

Utilizing scanning electron microscopy (SEM), the surface shape and morphology of the CaO-MgO material were investigated. SEM captures and processes secondary electrons released from the catalyst surface to create a high-resolution image displaying the form of the substance. Figure 3 displays the SEM characterization at 1500× magnification.



(a) (b)

Figure 3. SEM Analysis of CaO-MgO with (a) 400ºC (b) 850ºC of Dehydration Temperature

The surface morphology of the CaO-MgO product made from dolomite using the precipitation-dehydration process exhibits uneven particle distribution and variable particle sizes. The dehydration temperature improved the particle size of the resulting product. By increasing the dehydration temperature from 400 to 850 ºC, the particle size has decreased from 4.80 to 3.84 μm. In addition, there are pores and spaces that show that CaO is present in the product and that MgO and CaO particles from dolomite have aggregated [6,15].

Comparable outcomes have been documented, the morphological structure of CaO-MgO obtained revealed aggregates of a particular shape. The increased crystallinity indicated that it might be used as a catalyst. The high crystallinity of CaO-MgO eliminates contaminants from the product and might enhance its physical characteristics, including its surface area, stability at high temperatures, and catalytic capabilities [14].

3.4. Acidity Level

Acidity level analysis is shown in Figure 4. In this analysis, dehydration temperature affects the pH of the CaO-MgO obtained. The increasing temperature causes the rise of acidity level. The dehydration process using the precipitation-dehydration method releases OH^- or interlayer water and produces acid-active structural defects. Dehydration at high temperatures can make the surface structure more acid-active, with an optimal increase around 700 °C. Therefore, the resulting material can be applied as an acid catalyst.

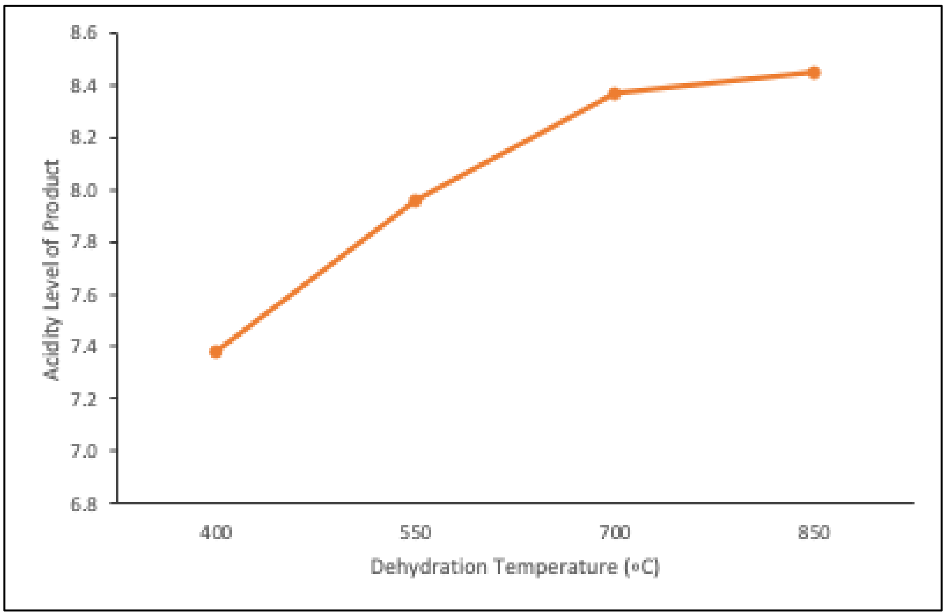


Figure 4. Acidity Level of Cao-MgO

4. CONCLUSION

In this study, the CaO-MgO material was successfully prepared from dolomite via precipitated-dehydration. The formation of MgO in the mixed oxide CaO-MgO derived from dolomite can be controlled by dehydration temperature. Chemical composition showed that the ratio of Ca/Mg was 2.4. Morphology analysis showed that the product has uneven particle distribution and variable particle sizes, indicating that the MgO and CaO particles from dolomite have aggregated. Based on the results of the analysis using BET, CaO-MgO obtained has the average surface area 71.1213 m^2/g, average pore volume: 0.1081 cc/g, average pore size: 20.5165 nm and average particle diameter: 36 nm. The large surface area can provide additional active pores and improve the interaction between reactants and active sites for catalyst application. The measured pH of the resulting product ranged from 8.0 to 8.5. It is concluded that precipitation-dehydration can be utilized for CaO-MgO recovery and further catalyst application.

ACKNOWLEDGEMENTS

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REFERENCES

[1] W. Jindapon, C. Ngamcharussrivichai, "Heterogeneously catalyzed transesterification of palm oil with methanol to produce biodiesel over calcined dolomite: The role of magnesium oxide," *Energy Convers Manag*. 171 1311–1321 (2018). https://doi.org/10.1016/j.enconman.2018.06.068.

[2] A. Buasri, K. Rochanakit, W. Wongvitvichot, U. Masa-Ard, V. Loryuenyong, The Application of Calcium Oxide and Magnesium Oxide from Natural Dolomitic Rock for Biodiesel Synthesis, in: Energy Procedia, Elsevier Ltd, 2015: pp. 562–566. https://doi.org/10.1016/j.egypro.2015.11.534.

[3] Solihin, Indriani, M.Z. Mubarok, Dissolution profile of dolomite in chloric acid solution: The effect of chloric acid concentration and pulp density, in: AIP Conf Proc, American Institute of Physics Inc., 2018. https://doi.org/10.1063/1.5038304.

[4] W. Widayat, T. Darmawan, H. Hadiyanto, R.A. Rosyid, Preparation of Heterogeneous CaO Catalysts for Biodiesel Production, in: J Phys Conf Ser, Institute of Physics Publishing, 2017. https://doi.org/10.1088/1742-6596/877/1/012018.

[5] S. Long, Z. Huang, Q. Du, P. Tang, X. Xian, F. Du, Y. Li, "Glucose isomerization into fructose with CaO-MgO mixed oxides and dolomite(ore)-derived base CaO/MgO catalysts in water under mild conditions," *Fuel*. 370 (2024). https://doi.org/10.1016/j.fuel.2024.131754.

[6] Widayat, H. Satriadi, P.W. Setyojati, D. Shihab, L. Buchori, H. Hadiyanto, F.A. Nurushofa, "Preparation CaO/MgO/Fe3O4 magnetite catalyst and catalytic test for biodiesel production," *Results in Engineering*. 22 (2024). https://doi.org/10.1016/j.rineng.2024.102202.

[7] Y.S. Erchamo, T.T. Mamo, G.A. Workneh, Y.S. Mekonnen, "Improved biodiesel production from waste cooking oil with mixed methanol–ethanol using enhanced eggshell-derived CaO nano-catalyst," *Sci Rep*. 11 (2021). https://doi.org/10.1038/s41598-021-86062-z.

[8] W. Widayat, N.T. Maheswari, W. Fitriani, L. Buchori, H. Satriadi, K. Kusmiyati, N. Ngadi, "Preparation of MgO-CaO/SiO2 catalyst from dolomite and geothermal solid waste for biodiesel production," *International Journal of Renewable Energy Development*. 12 541–549 (2023). https://doi.org/10.14710/ijred.2023.51573.

[9] D. Murguía-Ortiz, I. Cordova, M.E. Manriquez, E. Ortiz-Islas, R. Cabrera-Sierra, J.L. Contreras, B. Alcántar-Vázquez, M. Trejo-Rubio, J.T. Vázquez-Rodríguez, L. V. Castro, "Na-CaO/MgO dolomites used as heterogeneous catalysts in canola oil transesterification for biodiesel production," *Mater Lett*. 291 (2021). https://doi.org/10.1016/j.matlet.2021.129587.

[10] X. Yan, Y. Li, X. Ma, J. Zhao, Z. Wang, H. Liu, "CO2 capture by a novel CaO/MgO sorbent fabricated from industrial waste and dolomite under calcium looping conditions," *New Journal of Chemistry*. 43 5116–5125 (2019). https://doi.org/10.1039/c8nj06257a.

[11] M.H. Sorour, H.A. Hani, H.F. Shaalan, G.A. Al-Bazedi, "Schemes for salt recovery from seawater and RO brines using chemical precipitation," *Desalination Water Treat*. 55 2398–2407 (2015). https://doi.org/10.1080/19443994.2014.946720.

[12] M. Hu, J. Pu, E.W. Qian, H. Wang, "Biodiesel Production Using MgO–CaO Catalysts via Transesterification of Soybean Oil: Effect of MgO Addition and Insights of Catalyst Deactivation," *Bioenergy Res*. 16 2398–2410 (2023). https://doi.org/10.1007/s12155-023-10580-z.

[13] F.D.M. Daud, K. Vignesh, S. Sreekantan, A.R. Mohamed, "Improved CO2 adsorption capacity and cyclic stability of CaO sorbents incorporated with MgO," *New Journal of Chemistry*. 40 231–237 (2016). https://doi.org/10.1039/c5nj02081f.

[14] W. Widayat, N.T. Maheswari, W. Fitriani, L. Buchori, H. Satriadi, K. Kusmiyati, N. Ngadi, "Preparation of MgO-CaO/SiO2 catalyst from dolomite and geothermal solid waste for biodiesel production," *International Journal of Renewable Energy Development*. 12 541–549 (2023). https://doi.org/10.14710/ijred.2023.51573.

[15] H. Khaleghi, H. Esmaeili, N. Jaafarzadeh, B. Ramavandi, "Date seed activated carbon decorated with CaO and Fe3O4 nanoparticles as a reusable sorbent for removal of formaldehyde," *Korean Journal of Chemical Engineering*. 39 146–160 (2022). https://doi.org/10.1007/s11814-021-0972-4.