

## Research Article

# Preparation and Characterization of Malaysian Dolomites as a Tar Cracking Catalyst in Biomass Gasification Process

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Three types of local Malaysian dolomites were characterized to investigate their suitability for use as tar-cracking catalysts in the biomass gasification process. The dolomites were calcined to examine the effect of the calcination process on dolomite's catalytic activity and properties. The modifications undergone by dolomites consequent to thermal treatment were investigated using various analytical methods. Thermogravimetric and differential thermal analyses indicated that the dolomites underwent two stages of decomposition during the calcination process. The X-ray diffraction and Fourier-transform infrared spectra analyses showed that thermal treatment of dolomite played a significant role in the disappearance of the  $\text{CaMg}(\text{CO}_3)_2$  phase, producing the  $\text{MgO}$ - $\text{CaO}$  form of dolomite. The scanning electron microscopy microphotographs of dolomite indicated that the morphological properties were profoundly affected by the calcination process, which led to the formation of a highly porous surface with small spherical particles. In addition, the calcination of dolomite led to the elimination of carbon dioxide and increases in the values of the specific surface area and average pore diameter, as indicated by surface area analysis. The results showed that calcined Malaysian dolomites have great potential to be applied as tar-cracking catalysts in the biomass gasification process based on their favorable physical properties.

## 1. Introduction

Natural dolomite is the mineral form of the double carbonate of calcium and magnesium,  $\text{CaMg}(\text{CO}_3)_2$ . It occurs as the major constituent of sedimentary formations in association with calcite,  $\text{CaCO}_3$ . Dolomite was first described in 1791 as a rock by the French engineer and mineralogist Deodat Gratet de Dolomieu (1750–1801) when he observed exposures in the Dolomite Alps of Northern Italy [1]. In Malaysia, dolomite is abundantly found in Perlis, the northern part of Peninsular Malaysia, and it is called “Batu Reput” by the local people. The main quarries of dolomite can be found in the surroundings of Chuping City in Perlis. Malaysian dolomite is relatively soft and easily crushed to fine powder. It has a grayish color and when crushed to form the powder, it becomes yellowish [2].

Dolomite in Malaysia is mainly used in the float glass and fertilizer industries, for soil conditioning, for use as building products in construction applications, and to prepare roads before paving with asphalt. Dolomite also has the potential to be used as a catalyst in certain chemical processes such as gasification and pyrolysis.

Gasification and pyrolysis of biomass for the production of fuel gas constitute one of the most promising processes for clean energy based on the substitution of fossil fuel with renewable energy; moreover, it decreases the global emission levels of carbon dioxide ( $\text{CO}_2$ ) [3]. Tar formation is a major drawback when biomass is converted into syngas or fuel gas during the gasification process. The most common method that can effectively reduce the tar content in raw fuel gas is thermal and catalytic cracking. These methods are preferred

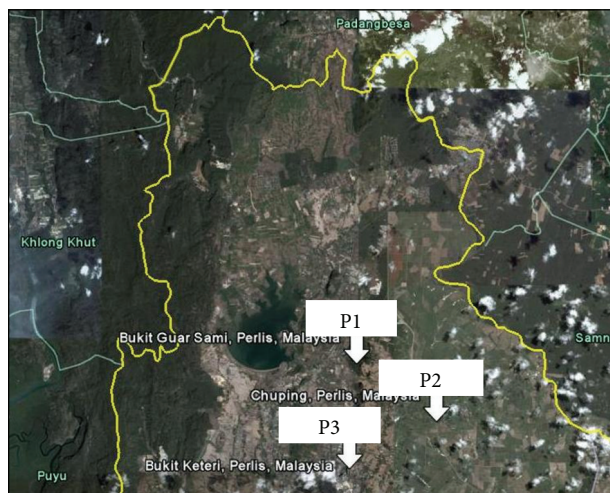


FIGURE 1: Dolomite quarries locations in Perlis State.

because the gas is maintained at a high temperature. This approach has been intensively studied for more than two decades because of its advantages including (1) conversion of tar into useful gases and (2) adjustability of the composition of the gas product. In a review of catalytic tar cracking by Sutton et al. [4], catalysts were divided into three distinct groups, namely, (1) dolomite catalysts, (2) alkali metal and other metal catalysts, and (3) nickel catalysts. Han and Kim [5] added a fourth group, that is, novel metal catalysts, which includes Rh, Pd, Pt, and Ru.

The application of dolomite as a catalyst in biomass gasification has attracted the interest of many researchers, and dolomite has been proved to have excellent tar-cracking capacity because it is cheap, easily replaceable, and can increase gas yield at the expense of the liquid products. However, the main limitations of using dolomite catalysts are their low activity levels during reforming of the methane present in the product gas and their easy erosion and breaking [4, 10]. The chemical composition and surface area of dolomite varies with its source of supply. Several research groups [6, 8, 9, 11–13] have investigated a system of raw gas cleaning involving different types of dolomites, such as Spanish, Swedish, Finnish, and Chinese dolomites, as catalysts for tar removal.

Dolomite seems to have a significant potential to solve the problems of high tar content in producer gas and will be of significant benefit to the Malaysian economy and environment. Thus, this work was aimed at studying the suitability of Malaysian dolomite for use as a tar-cracking catalyst in the biomass gasification process. To achieve this objective, three types of local dolomites from different locations in Perlis State were prepared and characterized in terms of their properties using different analytical techniques, such as X-ray fluorescence (XRF), thermogravimetric analysis (TGA), X-ray diffraction (XRD), Fourier-transform infrared absorption spectra (FTIR), nitrogen adsorption-desorption, and scanning electron microscopy (SEM).

## 2. Experimental

**2.1. Materials.** Three types of natural Malaysian dolomite (P1, P2, and P3) were used and characterized in this study. Dolomite samples were obtained from dolomite quarries located in Perlis State on the northern part of Peninsular Malaysia (as illustrated in Figure 1). These samples were provided by Northern Dolomite Sdn. Bhd., Perlis; UMPAN Global Sdn. Bhd., Penang; and Kangar Dolomite Sdn. Bhd., Kedah, respectively. The original samples were in the form of stones and had to be crushed and ground until they changed to the powder form; then, they were subjected to size classification using a Retsch Test Sieve (ASTM E11, 200  $\mu$ ). The dolomites were then calcined under nitrogen in a furnace at 1000°C for four hours. The dolomite powders with particle sizes below 0.2 mm for both the natural and the calcined forms were used for characterization.

### 2.2. Analysis

**2.2.1. XRF Study.** The chemical composition of dolomites was estimated using wavelength-dispersive XXRF (WDXRF; model BRUKER, S8 TIGER). The fused-bead technique was used to prepare the dolomite samples for XRF analysis. Specifically, 0.5 g of sample was mixed with 5.0 g of spectra-flux LT-110 ( $\text{Li}_2\text{B}_4\text{O}_7 = 66.5\%$ ,  $\text{LiBO}_2 = 33.5\%$ ) and placed in a platinum crucible. The mixture was then fused at temperatures between 900 and 1250°C and cast in a platinum casting dish. The samples were subjected to precise control of the cooling process to ensure the reproducibility of the flat glass discs and to avoid cracking or crystallization. The samples were then placed in an XRF instrument within nine minutes for analysis.

**2.2.2. Thermal Analysis.** To evaluate the thermal decomposition behavior of dolomites during the calcination process, thermal analysis was carried out in a simultaneous thermogravimetric analyzer (model Mettler Toledo, TGA/SDTA851) for both TGA and differential thermal analysis (DTG). Approximately 50 mg of dolomite powder was placed in an alumina crucible and weighed exactly. The sample was then placed inside the furnace and programmed for continuous heating from room temperature to 1000°C at a heating rate of 10°C/min under nitrogen flow at the rate of 100 mL/min.

**2.2.3. XRD Analysis.** To examine the structural changes induced by the calcination process, the dolomite samples were characterized using a diffractometer-XRD system (model Shimadzu XRD6000), using Cu-K $\alpha$  (at wavelength 1.5406 Å) radiation, fitted with a Cu filter on the secondary optics, and operated under 40 kV power and 30 mA current. The scanning  $2\theta$  range was from 20° to 90°, at a scan rate of 3°/min.

**2.2.4. FTIR Spectral Analysis.** FTIR spectra (model Perkin-Elmer, 100 series) were used to confirm the structural transformation of the dolomites during the calcination process.

TABLE 1: Analysis results of the compositions of dolomite (%).

Compounds	Malaysian dolomite (P1) This study	Malaysian dolomite (P2) This study	Malaysian dolomite (P3) This study	Chinese dolomite (Zhejiang) [6]	Swedish dolomite (Sala) [6]	Indian dolomite (D <sub>01</sub> ) [7]	Spanish dolomite (Malaga) [8]	Finnish dolomite [9]
SiO <sub>2</sub>	0.07	0.09	15.37	1.35	2.21	0.18	—	2.5
Al <sub>2</sub> O <sub>3</sub>	0.04	0.08	1.69	0.14	0.11	0.25	0.4	0.6
Fe <sub>2</sub> O <sub>3</sub>	0.07	0.12	0.51	0.03	0.54	0.63	0.01	2.1
CaO	30.0	32.0	23.0	30.72	30.5	30.24	30.6	26.6
MgO	21.0	21.0	17.2	20.12	20.2	21.33	21.2	18.3
K <sub>2</sub> O	0.014	0.026	0.195	0.02	0.04	0.03	—	—
Na <sub>2</sub> O	0.013	0.013	0.013	0.01	0.03	0.23	—	—
P <sub>2</sub> O <sub>5</sub>	0.013	0.017	0.019	0.01	0.01	—	—	—
MnO	0.01	0.011	0.013	0.002	—	21.33	—	—
SrO	0.008	0.009	0.009	—	—	—	—	—
TiO <sub>2</sub>	0.006	0.007	0.015	0.00	0.01	0.18	—	0.3

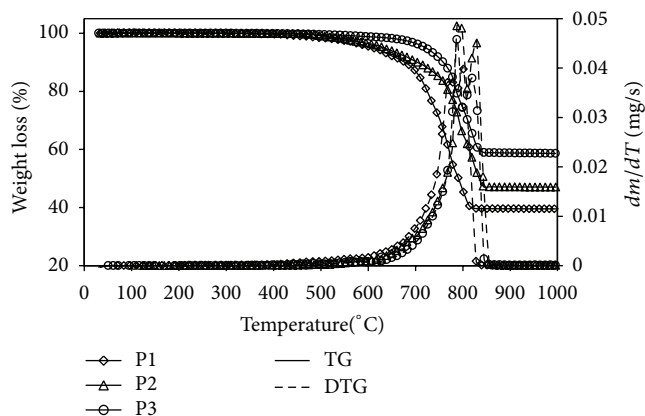


FIGURE 2: TG and DTG curves of Malaysian dolomites.

The spectral data were used to identify the sample compounds by matching the fingerprints of the samples with those in the database. In addition, the functional groups and structural characteristics of a compound enable us to elucidate the possible structure types. The spectrum range of the FTIR study was the mid-infrared region, which covers the frequency ranges of 500–4000  $\text{cm}^{-1}$ , at a resolution of 8  $\text{cm}^{-1}$ .

**2.2.5. SEM Analysis.** The surface and microstructure analysis of dolomites was carried out using an SEM instrument (model HITACHI, S-3400N). The dolomite powder was mounted onto the SEM stubs (layered with sticky carbon tape). The stub was then placed in a sputter coater (EMITECH, K550X) for five minutes for coating with gold to provide high reflectivity during the scanning process. The samples were then placed at 40°C in an oven before SEM analysis.

**2.2.6. Physical Properties Analysis.** The physical properties of the dolomites, such as specific surface area, total pore

volume, and pore size distribution, were obtained by measuring their nitrogen adsorption-desorption isotherm at 77 K using a Quantachrome instrument (model Autosorb-1). The Brunauer-Emmet-Teller (BET) surface area was estimated from the adsorption-isotherm data using the relative pressure ( $p/p_o$ ) range of  $10^{-6}$  to 1. The total pore volume was assessed by converting the amount of nitrogen gas adsorbed at relative pressure to the volume of liquid adsorbate. The analytical method consisted of three steps, including dehydration, degassing under low vacuum pressure, and nitrogen gas adsorption at 77 K. The resulting isotherm was analyzed using the BET adsorption method, and the pore-size distributions were derived by the Barrett-Joyner-Halenda (BJH) desorption method.

### 3. Results and Discussion

**3.1. Chemical Composition of Dolomite.** The chemical compositions of the various Malaysian dolomite samples, which were determined using XRF analysis, are listed in Table 1. The main contents of dolomite are CaO and MgO, followed by low levels of undesirable impurities, such as SiO<sub>2</sub>, K<sub>2</sub>O, Na<sub>2</sub>O, and Al<sub>2</sub>O<sub>3</sub>. The chemical composition of P3 dolomite differed significantly from that of P1 and P2. As shown in Table 1, the chemical composition analysis indicated that the CaO and MgO contents of the P1 and P2 dolomites were very close to those of other types of dolomites, whereas in the case of P3, these contents were relatively low. Furthermore, P1 and P2 had a much lower content of impurities than the other types of dolomites, whereas P3 had a higher level of impurities, especially silica (SiO<sub>2</sub>, 15.37%) and alumina (Al<sub>2</sub>O<sub>3</sub>, 1.69%). These impurities combine with CaO at elevated temperatures to form a slag, reducing the pore volume and the amount of available active lime [7]. Many research groups have reported that the chemical composition of dolomites play an important role when used as a catalyst for tar reforming in gasification processes [6, 8, 9, 11–13]. Myren and others reported that a mixture of silica and dolomite was not as effective as the same

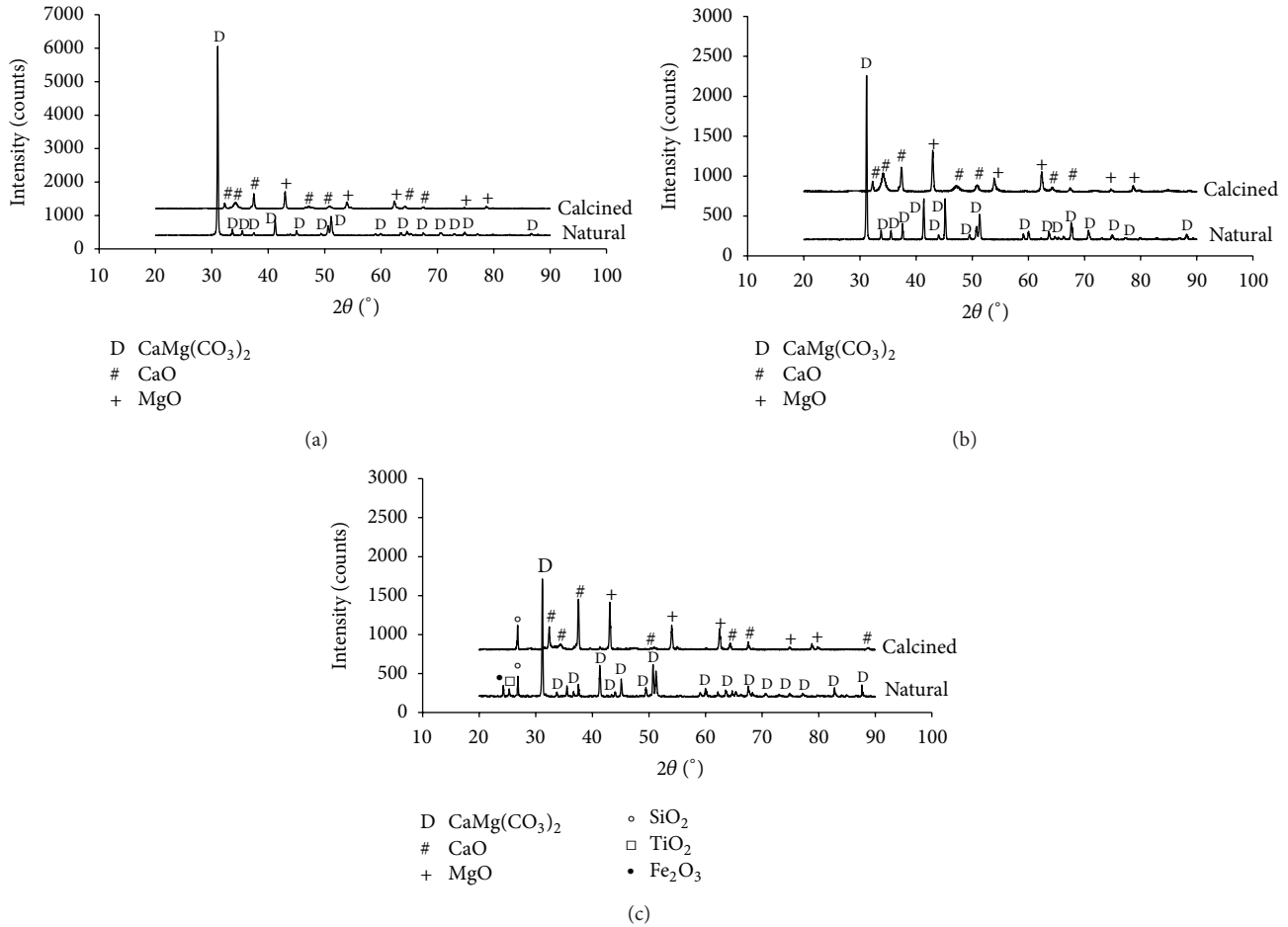
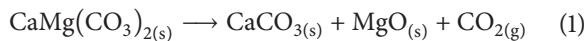


FIGURE 3: XRD analysis before and after calcination (a) P1 dolomite, (b) P2 dolomite, and (c) P3 dolomite.

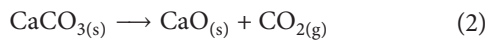
amount of pure dolomite for tar cracking [14]. In general, dolomites with the lowest content of CaO and MgO show the lowest tar cracking efficiency [6].

**3.2. Thermal Decomposition of Dolomite.** To evaluate the thermal decomposition behavior of dolomites under calcination temperatures, thermal analysis was conducted in a simultaneous TG-DTA analyzer. The thermal decomposition characteristics of dolomites with reference to the TGA and DTG curves are displayed in Figure 2. Dolomite is the mineral form of the double carbonate of calcium and magnesium,  $\text{CaMg}(\text{CO}_3)_2$ . Under heating, dolomite is often assumed to be decomposed in two separate stages as follows [2, 15].

First stage:



Second stage:



The first thermal decomposition stage occurs after 600°C and is followed by the second stage after 800°C. From Figure 2,

the observed weight loss below 600°C for P1, P2, and P3 was 4.59%, 3.42% and 1.24% respectively, whereas, the weight loss between 600 and 830°C was 55.77%, 49.26% and 39.84% respectively. The main weight loss in the 600–830°C range can be attributed to the decomposition of the carbonates mineral that exists in dolomite and the release of carbon dioxide as explained by (1). Therefore, the weight loss for P1 and P2 in this temperature range was higher compared to that of P3 due to the higher content of carbonates. In Figure 2, the DTG curves of the three types of dolomites show two peaks. For P1, the onset of the first peak begins at 600°C, reaches a maximum at 767.7°C, and ends at 790.3°C, whereas the second peak begins at 790.3°C, reaches a maximum at 801.3°C, and ends at 830°C. For P2, the first peak begins at 600°C, reaches a maximum at 787.5°C, and ends at 809.5°C, whereas the second peak begins at 809.5°C, reaches a maximum at 829.5°C, and ends at 830°C. For P3 also, the first peak begins at 600°C, reaches a maximum at 787.5°C, but ends at 799.3°C, whereas the second peak begins at 799.3°C, reaches a maximum at 819.6°C, and ends at 830°C. As shown in (1) and (2), the peak at the lower temperature represents the decomposition of the dolomite structure, with the release of carbon dioxide from the carbonate ion associated with the magnesium part of the structure, accompanied by the formation of calcite



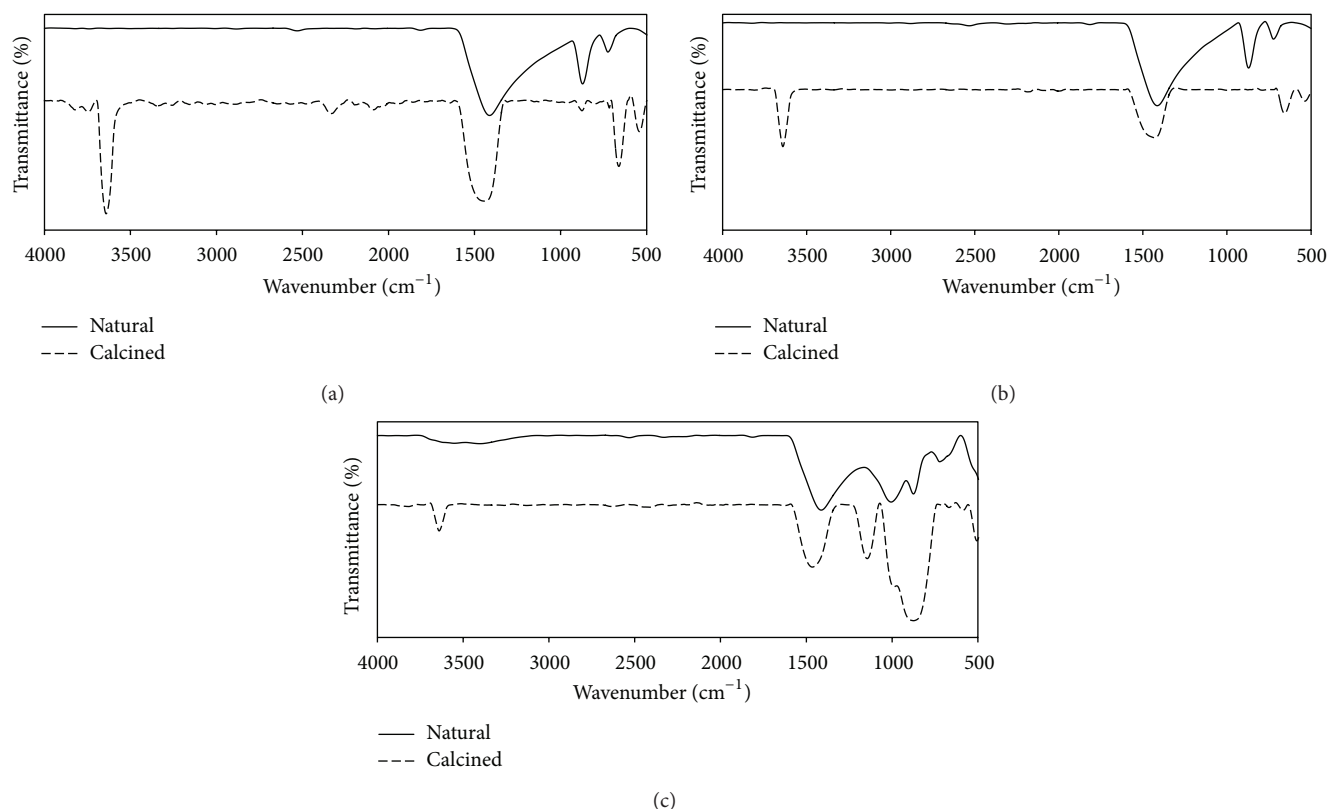


FIGURE 4: FTIR spectra before and after calcination (a) P1 dolomite, (b) P2 dolomite, and (c) P3 dolomite.

and magnesium oxide. The peak at the higher temperature corresponds to the decomposition of calcite, with the release of carbon dioxide. As shown in Figure 2, the decomposition process for P1 is initiated at lower temperatures compared to the other types of Malaysian dolomites. This probably is due to its higher content of CaO, as presented in Table 1. These results are in agreement with the previous works by McIntosh et al. [15] and Gunasekaran and Anbalagan [7]. In addition, Farizul et al. [2] have also found that the DTG plot of Malaysian dolomite shows two peaks at 750.0 and 930.0°C. The temperature of the second peak is higher compared to that in the present study, which can be attributed to the partial pressure of carbon dioxide in the dolomite samples, whereby the temperature of the second peak increases with increasing partial pressure of carbon dioxide. The peak temperature also depends on other procedural variables, such as the heating rate and the sample mass, because these, in turn, influence the actual atmosphere around the decomposing sample [15]. As a result, the thermal treatment process (calcination) of dolomite produced a catalyst with very high activity, due to disappearance of the  $\text{CaMg}(\text{CO}_3)_2$  phase and producing of the MgO-CaO form of dolomite.

**3.3. XRD Analysis.** The dolomites were characterized before and after calcination using XRD to analyze the effect of calcination on the dolomite structure. The results are shown in Figure 3. The XRD patterns for natural dolomites show that all peaks correspond to those of  $\text{CaMg}(\text{CO}_3)_2$  (JCPDS

36-0426). All these peaks also have been observed in other research works [3, 7], indicating that the Malaysian dolomite samples have almost the same properties as the other dolomites. The XRD pattern for calcined dolomites, however, changes drastically compared to that of natural dolomites. New dominant peaks which belong to CaO (JCPDS 04-0777) and MgO (JCPDS 87-0652) are observed. Natural P3 sample (Figure 3(c)), on the contrary, shows three additional peaks between  $2\theta = 20^\circ$  and  $2\theta = 30^\circ$ , which are assigned to  $\text{Fe}_2\text{O}_3$  (JCPDS 26-1319),  $\text{TiO}_2$  (JCPDS 71-1169), and  $\text{SiO}_2$  (JCPDS 83-0539), occurring at  $2\theta = 25.2^\circ$ ,  $26.7^\circ$ , and  $28^\circ$ , respectively. The changes in the peaks illuminate the fact that calcination of Malaysian dolomites results in the formation of the MgO-CaO form, which is the active catalytic component in tar conversion.

**3.4. FTIR Spectral Analysis.** The typical transmittance FTIR spectra of the Malaysian dolomites before and after the calcination process are shown in Figure 4. The FTIR spectra for all natural dolomite samples indicate that the most prevalent component present in dolomite is carbonate. The carbonate mineral group (magnesium and calcium carbonates) shows a strong and broad band near  $1412\text{ cm}^{-1}$ , whereas the other main components of dolomite are clearly differentiated by characteristic bands near 872 and  $724\text{ cm}^{-1}$  [16]. In the case of P3 (Figure 4(c)), in addition to the band corresponding to the carbonate group, the FTIR spectra show a band at  $1005\text{ cm}^{-1}$ ,

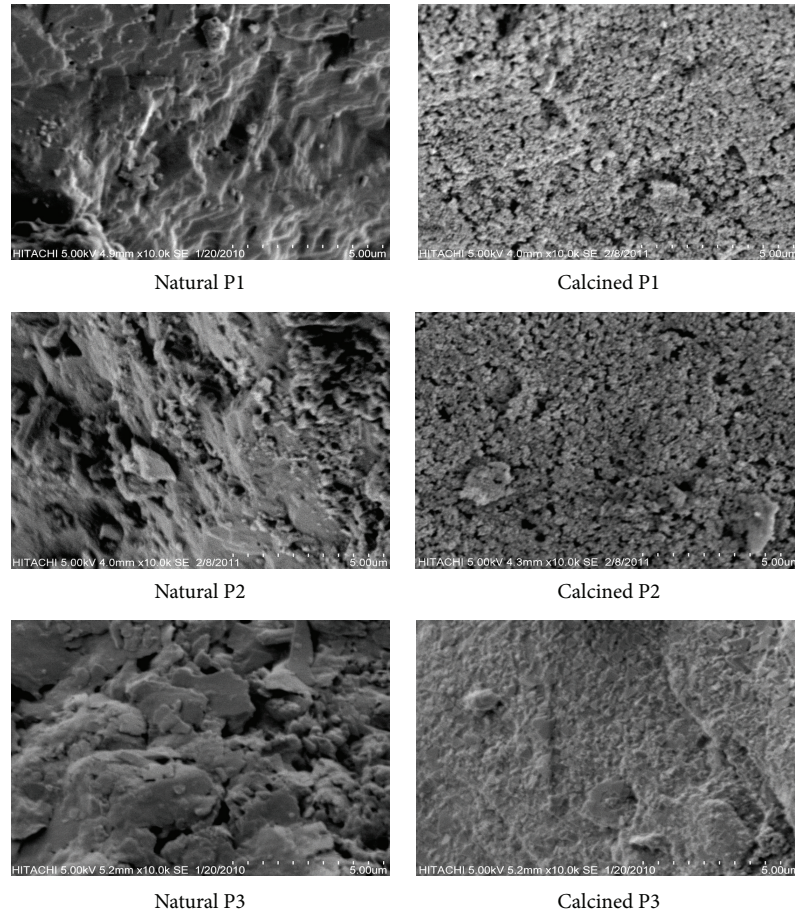


FIGURE 5: SEM microphotograph of natural and calcined Malaysian dolomites.

TABLE 2: Pore characteristics of the calcined dolomites.

	P1 (this study)	P2 (this study)	P3 (this study)	Sala [6]	Zhejiang [6]	Malaga [8]
BET surface area ( $\text{m}^2/\text{g}$ )	15.25	16.85	6.16	7.3	5.1	12
Pore volume ( $\text{cm}^3/\text{g}$ )	0.32	0.104	0.09	0.09	0.03	0.07
Average pore diameter ( $\text{\AA}$ )	416	233	215	496	269	144

referring to the presence of Si–O vibration of the silicate phase, which is in agreement with the chemical analysis data.

The FTIR spectra for dolomites after calcination clearly show the structural transformation of natural dolomite to calcium and magnesium oxides. At this stage, the main broad band at  $1412\text{ cm}^{-1}$  shifts to  $1430\text{ cm}^{-1}$ , and the intensity of the band decreases, showing the presence of calcium and magnesium oxides [17]. Moreover, strong and intense bands are observed at  $3641$  and  $660\text{ cm}^{-1}$  due to calcium oxide, in addition to the band at  $540\text{ cm}^{-1}$  attributable to magnesium oxide. The weak bands at  $875$  and  $1005\text{ cm}^{-1}$  for natural P3 (Figure 4(c)) combine together to form a strong and broad band after calcination, which can be attributed to the effect of impurities such as silicates on the dolomite structure at

elevated calcination temperatures. Moreover, another strong band is observed at  $1144\text{ cm}^{-1}$  for calcined P3 dolomite due to the presence of silica, whereas the intensity of the calcium oxide bond at  $3641\text{ cm}^{-1}$  is weak compared to the bands of the calcined samples of P1 and P2. These results are in complete agreement with the results obtained from XRD analysis.

**3.5. Scanning Electron Microscopy (SEM) Analysis.** As illustrated in Figure 5, the SEM micrographs of the calcined dolomites are significantly different from those of the dolomites before calcination. The SEM micrographs of natural dolomites show a rough and disordered surface with low-porosity grains, whereas the surfaces of calcined dolomites show clusters of tidy and porous grains, confirming the thermal decomposition.

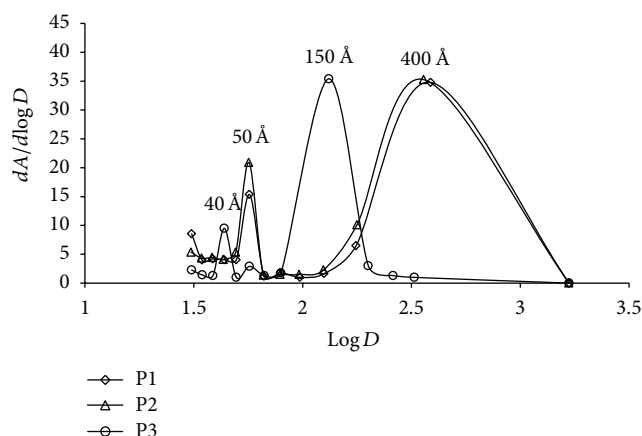


FIGURE 6: Pore size distribution of calcined Malaysian dolomites:  $D$ : pore diameter (Å),  $A$ : BET surface area ( $\text{m}^2/\text{g}$ ).

**3.6. Surface Area and Total Pore Volume Analysis.** The BET surface area, the BJH pore volume, and the average pore size for calcined Malaysian dolomites are summarized in Table 2. A comparison of the surface area and pore volume (17–3000 Å) shows that P1 and P2 dolomites have the highest surface area and pore volume compared to P3 and other published dolomites. This is possibly due to the high CaO and MgO contents in P1 and P2 dolomites and the lower contents of impurities, such as  $\text{SiO}_2$ ,  $\text{K}_2\text{O}$ ,  $\text{Na}_2\text{O}$ , and  $\text{Al}_2\text{O}_3$ . Slag is produced when these impurities combine with CaO at high temperatures, leading to a reduction in both the pore volume and the amount of available active lime [18]. The surface properties, apart from the chemical composition of dolomite, are the most important factors for selecting dolomite as the cracking catalyst in many thermochemical processes.

The pore size-distribution curves of the calcined Malaysian dolomites are shown in Figure 6. The pore-size distribution of P1 and P2 dolomites differs from that of P3. The pore sizes of P1 and P2 dolomites are mainly in the 400 Å region, whereas the major pore size of P3 is in the 150 Å region.

## 4. Conclusions

The natural Malaysian dolomites, after undergoing thermal treatments at high temperatures ( $1000^\circ\text{C}$ ), produced activated dolomite, which could be used as a cracking catalyst in the biomass gasification process. Malaysian dolomites P1 and P2 contained higher amounts of CaO and MgO, with lower amounts of  $\text{SiO}_2$ ,  $\text{K}_2\text{O}$ ,  $\text{Na}_2\text{O}$ , and  $\text{Al}_2\text{O}_3$ , compared to P3 and other dolomites (Table 1). The DTG curves showed two stages of decomposition during the calcination process. The first thermal decomposition occurred after  $600^\circ\text{C}$ , followed by the second step after  $800^\circ\text{C}$ . The XRD and FTIR results confirmed the structural transformation of natural dolomite to the calcium and magnesium oxide form, and the results of the BET and BJH analyses showed that P1 and P2 dolomites had higher surface areas and pore volumes compared to P3 and other types of dolomites. This suggests that P1 and P2 dolomites have great potential for

use as tar-cracking catalysts. The most important factors that contribute to the selection of dolomites with highly efficient tar-cracking capacity are calcination balance and chemical composition in addition to geometrical surface area and average pore diameter.

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