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Effect of Heat Treatment on the Properties of Perlis Dolomite Powder

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ABSTRACT

Dolomite, $CaMg(CO_3)_2$, is a carbonate mineral naturally found in the sedimentary rocks associated with limestone and chalks, which has gained much interest for its potential as a raw material for bioceramic production. Aside from the important natural elements, dolomite is typically composed of organic compounds, carbonates and moisture. Therefore, heat treatment was required for dolomite to decarbonise the carbon content and remove volatiles and impurities, thus improving its purity. However, there were several previous studies on the heat treatment of dolomite minerals, and limited scientific findings have been published specifically for Perlis dolomite. The composition of natural mineral resources always slightly differs with different source locations, which affects some of its properties. Therefore, this study aims to investigate the influence of the heat treatment process on the physical, chemical, and structural properties of Perlis dolomite and evaluate its potential for bioceramic production. The raw and processed dolomite were analysed through XRF, TGA-DSC, BET, XRD and FTIR. The findings showed that the heat treatment process is crucial for Perlis dolomite to improve its purity, enhance the oxide compounds, especially calcium and magnesium oxide, and activate the dolomite by forming hydroxyl groups. These important findings also proved the high potential of heat-treated Perlis dolomite as a promising precursor for bioceramic production.

Keywords: Annealing, bioceramic, heat treatment, material characterisation, Perlis dolomite

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INTRODUCTION

Dolomite is one of the natural resources that can be found easily in most of the reservoirs in the northern area of Peninsular Malaysia, which is in Perlis, specifically at most of the quarries in the vicinity of Chuping and Kangar (Azurahanim et al., 2022). Perlis Dolomite is the second most prevalent carbonate after calcite and is usually associated with chalk and limestone. It can be actively exploited to gain large profits for economic importance for the country through various applications in a wide range of industries. In 2017, the Department of Mineral and Geosciences Malaysia (JMG) proclaimed that Chuping, Perlis possess dolomite reserves with weight assumptions of around 200 million metric tonnes, with an approximate cost of nearly RM 1.3 billion (Samad et al., 2019). Up to the present time, Perlis is still available with an abundance of dolomite, which can be proved by the most recent update by the Ministry of Natural Resources, Environment and Climate Change (NRECC) at the beginning of the year 2024. The aim of executing exploration activities upon the dolomite reservoirs will be implemented progressively in the district of Padang Besar, Perlis, as this mineral is valuable to this state and is worth up to RM 19.2 billion. Perlis Dolomite Industries Sdn often managed the local dolomite. Bhd. as the main manufacturer and supplier where the continuous demands from the domestic and international markets were fulfilled.

Ideally, dolomite is a carbonate resource with main constituents of the mineral, which are calcium and magnesium, with a chemical formula of $CaMg(CO_3)_2$. Generally, dolomite minerals were formed by the dolomitisation process where the magnesium replacement in calcite (Cai et al., 2021; Zahir et al., 2020; Resio, 2024; Shahraki et al., 2009) over long periods. It is naturally found in the dolomitic rocks called dolostone (Fauzi, Osman, Alrashdi et al., 2022; Cai et al., 2021; Resio, 2023). The Perlis dolomite is mostly known as ‘Batu Reput’ by the locals because of its relatively low hardness and easily crushed into fine powders (Azurahanim et al., 2022; Kamarzamann et al., 2022; Kambakhsh et al., 2024; Mohammed et al., 2013; Osman et al., 2022). Dolomite is practically used for soil conditioning and fertiliser making in agriculture, as well as on road pavement and building materials in construction industries (Hussin et al., 2006; Mohammed et al., 2013). The abundance of dolomite minerals in Perlis should be explored broadly to enhance the material development of the dolomite in other applications, particularly for bone tissue engineering practices in the biomedical industry. This is because the dolomite mineral can be used to acquire its dominant components (CaO and MgO) as precursors in the bioceramic process. Both compounds were believed to play vital roles in bone treatments. Practically, CaO is the crucial component in the bone matrix and assists in bone formation by releasing Ca ions to encourage osteoblast proliferation (Liu et al., 2008; Marzban et al., 2019). Meanwhile, MgO contributes to the growth factor of the bone during the beginning phase of osteogenesis, stimulates bone formation and decreases the implications of osteoporosis, as well as is associated with bone mineralisation of calcified tissues (Arokiasamy et al., 2022; Liu et al., 2008; Marzban et al., 2019).

Owing to the unique compositions of dolomite, this mineral has raised researchers' attention to biomedical applications. Moroccan dolomite has been used as the main precursor along with perlite/slag to produce akermanite bioceramic that has great mechanical properties with high formation of apatite layer, indicating its good biocompatibility and bioactivity performances suitable for bone tissue engineering (Arkame et al., 2023; Harrati et al., 2022). Moreover, the addition of calcined dolomite from Qingyang, China, associated with montmorillonite in the hydroxyapatite-based bone adhesive has improved its biocompatibility and degradation performance (Hu, Qu et al., 2024; Hu, Wang et al., 2024). Apart from dolomite from various regions, Perlis dolomite exhibits promising potential for biomedical applications. Hossein Mohammadi et al. performed an investigation by using Perlis dolomite powder along with cockleshell and rice husk ash and successfully produced calcium magnesium silicate (CMS-akermanite) with a relative density of 93.50% and high flexural and tensile strength approximately about 22.16 MPa and 8.21 MPa, respectively which shown potential as bone graft substitutes in the non-load-bearing area (Mohammadi et al., 2020).

Besides, the dolomite has also been used as a mineral filler in the Poly(ethylene-co-vinyl acetate) composite to substitute the non-recyclable silicone elastomer in biomedical applications, and the findings showed that the dolomite has improved the mechanical performances, where the tensile strength and elongation at break of the copolymer matrix were enhanced (Fauzi, Osman, Alosime et al., 2022; Chong et al., 2021). These studies show that dolomite has significant potential as a biomaterial for various biomedical applications. Thus, heat treatment would be required on the dolomite minerals to achieve better structural properties and a high purity grade before further bioceramic production procedures, leading to great performances and responses in biomedical applications.

Heat treatment is an initiative through a controlled process involving thermal reactions to alter the initial properties of the material. The calcination process is one of the heat treatments carried out at a significantly lower temperature than its melting point for certain durations. Besides, the heat treatment process also aims to decarbonise the carbon content and remove any other insignificant compounds from the impurities apart from the materials. Technically, the ideal thermal decompositions of dolomite would be achieved at a temperature range from 700°C to 1000°C. The dolomite mineral could directly decompose completely and produce CaO and MgO at 1000°C (Vaganov et al., 2017). In addition, the decomposition of dolomite can also be divided into two phases where the applied heating temperature contradicts each other.

According to Jablonski (2015), the first decomposition of the dolomite happened at low temperatures (below 700°C), where it forms MgO and $CaCO_3$ while releasing CO_2

gases. Meanwhile, the second decomposition of the $CaCO_3$ at higher temperatures above 700°C creates CaO and again releases the excess gases of CO_2. This statement meets in agreement with Carlos et al., who have stated that the decomposition of dolomite can occur in two different approaches, which are full decomposition (one stage) and half decomposition (two stages) (Rodriguez-Navarro et al., 2012). The dolomite undergoes full decomposition upon the heat applied to the thermal reaction, as in Equation 1. Meanwhile, half of the decompositions are reported in Equations 2 and 3. The exact temperatures of the dolomite decompositions are not stated as they may vary in their scientific data. This is because the thermal mechanism of the dolomite minerals is poorly resolved and obscured (Rodriguez-Navarro et al., 2012; Shahraki et al., 2009) as the findings were varied and inconsistent, occasionally regarding different compositions of impurities in the dolomite carbonate from different quarry sources, followed with different experimental conditions applied during the decomposition process, including contrast atmosphere, heating rate, prevailing pressure of CO_2, sample mass, size of crystal particle and more else (Jablonski, 2015; Rodriguez-Navarro et al., 2012). Thus, Perlis dolomite powder may require a heat treatment process to subsequently proceed to the sintering process for the preparation of bioceramic samples to avoid samples cracking. This is because the applied sintering temperature may lead to thermal shock on the samples, which is caused by the sudden high temperature and excessive gas burn-off.

$$MgCO_3 \rightarrow MgO + CO_2 \quad [1]$$

$$CaMg(CO_3)_2 \rightarrow CaO + MgO + 2CO_2 \quad [2]$$

$$CaMg(CO_3)_2 \rightarrow CaCO_3 + MgO + CO_2 \quad [3]$$

$$CaMg(CO_3)_2 \rightarrow CaCO_3 + MgCO_3 \quad [4]$$

$$CaCO_3 \rightarrow CaO + CO_2 \quad [5]$$

According to previous literature, the physical, chemical and structural properties of dolomite are commonly changed by heat treatment and significantly affected by the source location of the dolomite mineral. Even though some research has been carried out on Perlis dolomite recently, the specific knowledge regarding its heat treatment properties, thermal decomposition, and reactions is still relatively limited compared to dolomite minerals in other countries. Therefore, heat treatment studies carried out on the Perlis dolomite are becoming crucial in determining this local natural resource's important properties and characteristics. Furthermore, the scientific discussion which correlates Perlis dolomite and the production of bioceramics is still not well-established, as this local mineral still focuses on agriculture and construction industries. Thus, the novelty of this study lies in investigating the impact of the heat treatment process (1000°C for 4 hours) on local Perlis dolomite and reveals its suitability and potential as a promising material for bioceramics in biomedical applications. This study aims to characterise the raw Perlis dolomite powder for its physical, chemical, and structural properties and compare it with the heated Perlis dolomite (a post-heating treatment process has been implemented). The testing of samples includes X-Ray Fluorescent (XRF), Thermogravimetric Analysis (TGA) and Differential Scanning Calorimetry (DSC), Brunauer-Emmett-Teller (BET), X-ray diffraction (XRD) and Fourier transform infrared spectroscopy (FTIR).

MATERIALS AND METHODS

Material

The local quarry at Chuping supplied Perlis dolomite, and Perlis was used as it is in this study without sieving or any other mechanical processing. An annealing process was carried out to determine the effect of heat treatment on the properties of this dolomite. The initial dolomite powder with an average particle size distribution of 150 µm was heated in a muffle furnace at 1000 °C for 4 hours with a 10°C/min heating rate. The temperature set for the annealing process was chosen according to the previous researchers' literature study and the author's lab work findings. Then, the annealed powder was ground with a pestle and mortar to obtain uniform fine powder. The purpose of the heat treatment process is to decarbonise the carbon contents and decompose the main compound in this mineral. Other than that, it also aimed to remove moisture and organic impurities from the dolomite composition. The raw and heat-treated dolomite powders were then sieved under 150 µm to obtain uniform fine powders.

Characterisations of Raw and Heated Perlis Dolomite

Chemical compositions of the raw and heated Perlis dolomite were determined by the analysis via X-ray fluorescent (XRF) by Philips PANalytical MiniPAL 4 machine. For sample preparation, 10 g of powders with an average size of about 75 µm were pressed using a hydraulic machine to create pellets. The prepared pellet was loaded into the spectrometer chamber. The X-ray was emitted by operating the machine with the maximum voltage and current at 30 kV and 1 mA, respectively, for 10 minutes of pre-set duration. The data of the detected elements was analysed using MiniPal software. The thermal properties of the materials have also been studied using the Linseis Thermal Balances instrument for Thermogravimetric Analysis (TGA) and Differential Scanning Calorimetry (DSC). The powders with a sample mass of approximately 30 mg were placed in an alumina crucible and inserted into the furnace with heating temperature initiated from room temperature up to 1200°C with a heating rate of 10°C/min under the flow of nitrogen at the rate of 100 mL/min. Analysette 22 Next Nano (FRITSCH) was used to perform precise Laser Particle Size Analysis (LPSA) to characterise the grain particles' size distribution on the sample powders. It has used the measurement range within 0.01 µm to 3800 µm with the Fraunhofer diffraction theory to calculate the grain size distribution, where the entire grain particles were assumed to have a spherical shape (Houghton et al., 2024).

Next, the surface area of the powders was analysed via Brunauer-Emmett-Teller (BET) analyser by Anton Paar for Nova. The analysis was implemented at room temperature with a thermal delay of 3 minutes. The adsorption of nitrogen gas, N_2, was set at 77.35 Kelvin, and the samples were degassed in a vacuum condition for 90 minutes at 250 °C with a 10 °C /min heating rate. The phase transformations and the crystallinity between the raw and heated dolomite were identified by X-ray diffraction (XRD) using the Test Instrument Machine Bruker D2 Phaser, German (2010). The powder samples were scanned using Cu Kα radiation at a wavelength of 1.5406 Å, operated under 40 kV voltage and 30 mA current for 10 minutes loading time with the angle diffraction of step-scanning was adjusted within 10° to 80° (2θ) with step size of 0.02°. The obtained diffractograms were then analysed by X'pert Highscore Plus software to distinguish the formed phases and crystal structures in the powders that have matched peak positions and intensities with the crystallographic database's ICCD Powder Diffraction File (PDF). The Perkin Elmer machine was used to perform the Fourier transform infrared spectroscopy (FTIR) analysis to identify the characteristic bands of the functional groups that transform the dolomite powder following the applied heat treatment. For the testing preparation, each sample of raw and heat-treated dolomite powders was mixed with potassium bromide powder (KBr) with a dolomite to KBr ratio of 3:7 and then pressed into pellet form. The prepared pallet was scanned from 450 cm^{-1} to 4000 cm^{-1} with 4 cm^{-1} resolution, and the transmission percentages of infrared radiation at each wavenumber were recorded.

RESULTS AND DISCUSSION

Chemical Composition Analysis

The chemical compositions of raw and heated dolomite analysed by XRF are shown in Table 1, where the weight composition was expressed in percentage of oxides. It showed that the raw Perlis dolomite was mainly composed of CaO and MgO, with 65.07% and 23.1%, respectively. Na_2O, Ag_2O, and Al_2O_3 were also found to be 1.0% to 8.0%. Other minor compounds are found to be below 1.0%, such as Sc_2O_3, SiO_2, SnO_2 and others. It was found that the percentages for all chemical compositions were slightly changed after the heat treatment. CaO has reduced from 65.07% to 54.55% because CaO is more reactive with CO_2 and tends to easily separate from the impurities and volatile substances from the dolomite during heat treatment. Meanwhile, the amount of MgO and Na_2O has increased by approximately 32% and 50%, respectively, as both compounds are less volatile due to high stability in thermal conditions and are able to maintain stability during the heat treatment. Since the carbonate contents decreased after the heat treatment, the remaining oxides with stable phases, such as MgO and Na_2O, became more concentrated. They appeared higher in the XRF measurement regarding the overall mass reduction. The increase of Na_2O contents after the heat treatment is suggested due to the applied thermal treatment that causes sodium ions to become more mobile and cause the sodium migration and diffusion on the material's surface, which leads to this compound being easily identified in the XRF detection. Subsequently, the percentages of the insignificant compounds have mostly decreased after the heat treatment.

In addition, the loss of ignition (LOI) of both samples in a vacuum was consistently about 10%, where these low values were considered an accurate measurement and precise detection of light elements in the samples. In contrast with most of the studies, the LOI of the dolomite was measured under the heat treatment temperature applied (maximum at 1000°C) and has a higher value around 40%–50%, which leads to decomposition of the carbonate contents and volatile substances and causes the increasing LOI (Dursun & Coşkun, 2020; Khashbaatar et al., 2022; Khoshraftar et al., 2023; Resio, 2023; Srinivasan et al., 2020). Yang et al. (2024) have mentioned that the low LOI is attributed to a few thermal decompositions on the resulting products.

This result shows that the heat treatment process has an average effect on the chemical composition of Perlis dolomite. Apart from that, the raw and heated Perlis dolomite is free from hazardous compounds, has zero toxicity and is safe for bioceramic production. Plus, the dominant compounds in the dolomite, which are CaO and MgO, were already common and mostly used as precursors in bioceramic production since they are able to play vital roles in biomedical applications. Comprehensively, the CaO is the main source of calcium in the matrix of human bone, teeth and skull (Fiume et al., 2020). Meanwhile, the MgO promotes bone growth modulation, repairs and regenerates bone tissues, assists in osteoblast proliferation, mineralises calcified tissue, and performs bone metabolism activity (Arokiasamy et al., 2022; Fiume et al., 2020; Nabiyouni et al., 2018).

Table 1
Chemical compositions of dolomite by XRF analysis

Compound	**CaO**	**MgO**	**Na_2O**	**Ag_2O**	**Al_2O_3**	**Sc_2O_3**	**SiO_2**	**SnO_2**	**Fe_2O_3**	**Others**	**LOI**
Raw Dolomite	65.07	23.10	8.00	1.46	1.30	0.48	0.25	0.24	0.18	0.12	10.35
Heated dolomite	54.55	30.50	12.00	0.29	1.20	0.23	0.10	0.38	0.10	0.17	10.97

Thermal Analysis

The thermal properties of the dolomite powder were carried out from room temperature to 1200°C, as TGA and DSC illustrated in Figure 1. Based on Figure 1(a), TGA curves of raw dolomite show a significant mass loss process with the total mass loss at 50.24% in the range of 423°C to 844°C. It is expected that mass loss will occur during the heat treatment process due to the decomposition of carbonates, water and organic matter. The major mass loss was attributed to the decomposition of carbonate contents from the raw dolomite by releasing CO_2 gases at an increasing temperature. The decomposition of raw dolomite between 500°C and 800°C has created high heat-resistant compounds CaO and MgO (Harrati et al., 2022). This result indicates that the raw Perlis dolomite powder requires a high annealing temperature of up to 850°C to eliminate the volatile or organic substances, similar to the findings by Shahraki et al. (2009). The constant slope of the TGA curve at the temperature above 840°C indicated the absence of any decomposition, which could support the sintering process at high temperatures over 900°C (Hafriz et al., 2018).

Meanwhile, the TGA curve of heated Perlis dolomite still shows multiple stages of mass losses due to residues and impurities being removed from the sample. However, for the heated dolomite powder, an apparent mass loss occurred approximately twofold lower than the value measured for the raw dolomite. The total mass loss was reduced by 24.98%, and no more significant mass loss was observed after 750°C. These results suggest that the heated dolomite has reached partial decomposition to form primary CaO and MgO compounds (during heat treatment) and that longer heating time is needed. The heat treatment process is suspected of readily activating the dolomite powder, lowering the decomposition and phase transformation temperature, and making it more reactive during heating than raw dolomite. Also, improper packing of the heated dolomite would be another factor for the small mass loss that occurs below 400°C, which is affected by the moisture contents.

Meanwhile, the mass loss above 400°C is due to the decomposition of the residue of calcite into pure CaO and organic matter, which agrees with the exothermic reactions on DSC curves. This result also indicates that the heated dolomite has greater thermal stability when applied at high temperatures compared to the raw dolomite. In order to achieve complete decarbonisation, decomposition, and phase transformation of raw dolomite powder, it is suggested that the heat treatment process be set to a longer soaking time of more than 4 hours. Besides, fine-sized particles of the powders normally promote good decomposition due to more exposure to heat on the surface area, which allows the carbonates to break down easily from the mineral. Complete heat treatment is important to avoid any gaseous burn-off during a powder sintering process, which can lead to deterioration of mechanical properties, thus causing cracking and fracture to the sintered bodies of the ceramic sample.

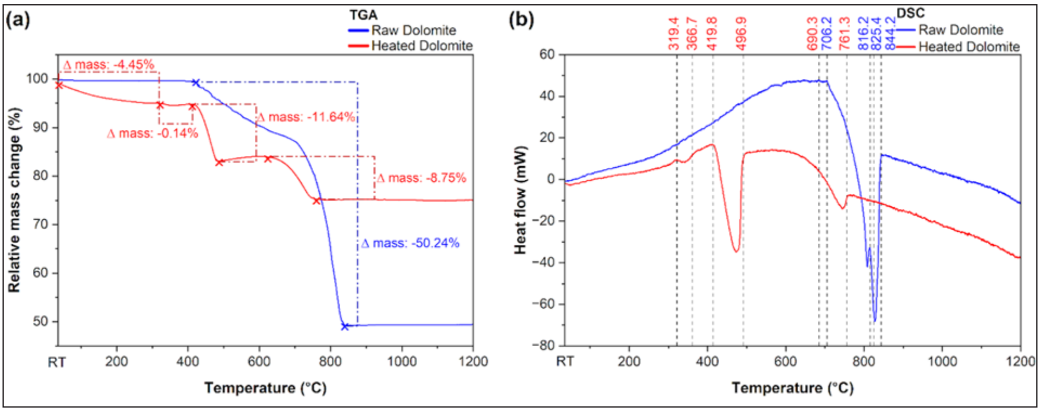

*Figure 1***.** (a) TGA and (b) DSC curves of raw and heated dolomite

Figure 1(b) presents the analysis of DSC curves of the raw and the heated dolomite. The curve of raw dolomite displayed two stages of thermal reactions, which are endothermic and exothermic. The endothermic peak sharply at 706.2°C indicated the thermal decomposition of calcite, $CaCO_3$ into CaO and CO_2 (C. Li et al., 2024; Zhang et al., 2017). Besides, it also could have occurred due to the thermal decomposition of $Ca(OH)_2$ resulting from the reaction between CaO and moisture content (Mandrino et al., 2018). The energy absorbed during this reaction reached 173.98 J/g. Then, two exothermic reaction peaks at 816.2°C and 825.4°C with a negative enthalpy of 152.09 J/g were attributed to the crystallisation of MgO and CaO, respectively. Next, the DCS curve of heated dolomite exhibits three obvious exothermic reactions, which agree well with the results discussed on TGA. The first exothermic peak at 319.4°C to 366.7°C was due to the crystallisation of small amounts of calcite from the raw dolomite. Next, both exothermic peaks shifted to an earlier temperature than the raw dolomite curve. The peak at 419.8°C to 496.9°C was due to the crystallisation of MgO and the decomposition of organic matter. Meanwhile, the last exothermic peak was due to the crystallisation of the calcite into pure CaO. This is because the raw dolomite has released more energy during the crystallisation process at high temperatures.

In the end, the heated dolomite powder consisted of a few residues that could crystallise at lower temperatures by involving the discharge of slight energy to create better thermal stability with steady reactions towards heat applied. The significant exothermic reactions by the heated dolomite promote favourable crystallisation with other precursors during a sintering process. This is because the exothermic reaction assists in the cross-linking between the components, forms a better crystalline structure and leads to stronger and solid materials in terms of their mechanical strengths. From these results, it has been shown that thermal treatment through heat treatment on the dolomite mineral is compulsory to provide high thermal stability and a high purity grade of powder. It shows that the applied heat treatment on the raw dolomite is able to decompose most of the organics, impurities, carbonates, and moisture from the mineral powder. Besides, the heated dolomite's constant slope after 750°C indicates thermal stability, whereas the raw dolomite required higher temperature up to 900°C.

Surface Area Analysis

Figure 2 presents the BET analysis by N_2 adsorption-desorption isotherms on the raw and heated dolomite. This specific surface area analysis is important and beneficial for dolomite powder. Technically, the raw material obtained from the local dolomite quarry is already in fine milled form, and this powder was used as it is. In addition, the processing of bioceramic by solid state sintering is commonly involved in the use of fine powder for the process and the preparation of samples, and it is rarely executed by using bulk or solid rock form. In the case of possible future work using this fine milled dolomite powder for bioceramic sample preparation, the porous characteristics of this mineral need to be clarified. Besides, the surface area, size, and pore volume of raw dolomite powder definitely affect the structural, morphological, and strength of the bioceramic. Hence, performing specific surface area analysis on this dolomite powder is important. The entire isotherm curves were categorised in type IV according to the IUPAC classification, which indicated the presence of mesoporous in the internal structure of the powders (Algoufi et al., 2017) and hysteresis loops commonly caused by H_2 (Chen et al., 2019). The results demonstrated that the heated dolomite has extremely high volume adsorbed during adsorption and desorption conditions compared to the raw dolomite. It shows that the N_2 adsorption of the capacity by the heated dolomite powder was a slackening increase at low pressure and displayed

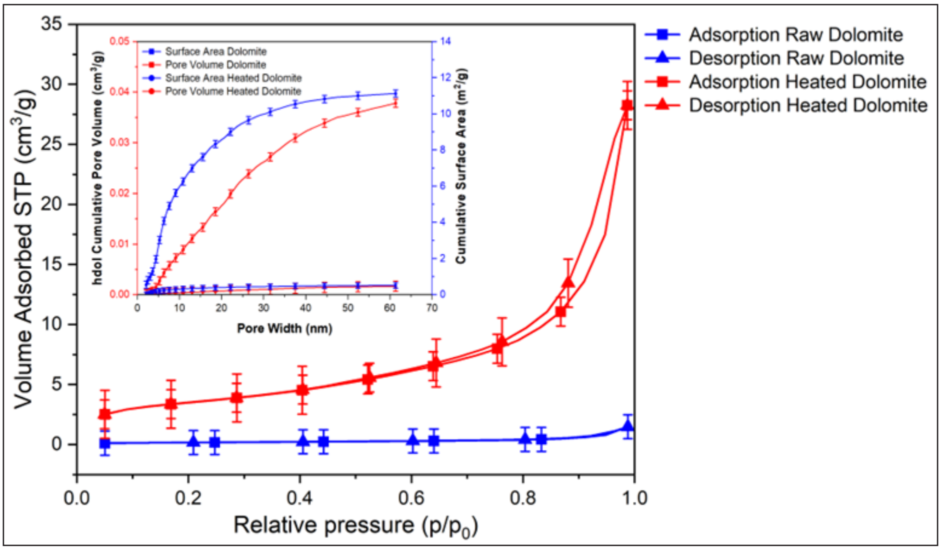

Figure 2. N_2 adsorption-desorption isotherms and pore size distribution of raw and heated dolomite

an expedited increase during P/P_0> 0.8. A study by Chen et al. (2019) has proclaimed that it happened due to the mesoporous formation with size particles around 4 nm based on the pore size distribution curves. Besides, high partial pressures are required in the N_2 adsorption due to the pore constrictions of the powder at the large pore diameter. The results show that the calcined dolomite has high adsorption performance and bioceramic production potential. This is because high adsorption performances are preferable mainly for protein adsorption, drug delivery, and bone growth factors to recover rapidly.

The findings on the specific surface area, pore volume and average pore diameter of Perlis raw dolomite and heated dolomite are summarised in Table 2**.** It shows that the heat treatment at 1000°C has increased the value of the specific surface area up to 13.3790 m^2/g, which is twenty times larger than the raw dolomite, which meets agreement with most studies (Kamarzamann et al., 2022; Hafriz et al., 2018; L. Li et al., 2024). The drastic increase in the surface area found for calcined dolomite is because the heat treatment process on the dolomite powder released CO_2 and removed volatile substances and organic matter, apart from the material, and created porous structures of CaO-MgO particles. The dense and well-packed crystalline structure of the raw dolomite has changed to higher porosity, followed by an increase in surface area after the heat treatment due to the crystal breakdown and loose bonds between the CaO and MgO particles. Subsequently, the size of the particle reduces with the increasing surface area. Besides, the heat treatment process has increased the pore volume and reduced the pore diameter average with values of 0.0438 cm^3/g and 14.1556 nm, respectively. It has been assumed that the increase in surface area and pore volume after the heat treatment was attributed to the large formation of CaO and MgO in the powder and the elimination of undesirable impurities, followed by other volatile contaminants (Algoufi et al., 2017; Hafriz et al., 2018). Meanwhile, a slight reduction in average pore diameter was due to the decomposition of carbonate contents and the release of CO_2 gases during the heat treatment process (Hafriz et al., 2018). The higher value in the specific surface area, pore size and larger pore volume can promote a significant improvement in adsorption performances (Kamarzamann et al., 2022). Besides, the larger specific surface areas also improve bioactivity performances, have excellent mechanical properties, and control degradation as intended to complement human cancellous bone. Thus, it can be supported that calcined dolomite can be used as an alternative material in bioceramic production for bone tissue engineering applications.

Table 2
Pore characteristics of raw and heated dolomite

Samples	BET surface area (m^2/g)	Pore volume (cm^3/g)	Average pore diameter (nm)
Raw dolomite	0.6010	0.0023	15.3193
Calcined dolomite	13.3790	0.0438	14.1556

Phase Transformation Analysis

Figure 3 displays the phase transformation of raw and heated dolomite. Figure 3(a) shows that the XRD pattern of raw Perlis dolomite was mainly formed by calcium magnesium carbonate, $CaMg(CO_3)_2$ phase, with a crystal system of hexagonal structure. The phases of $CaMg(CO_3)_2$ (JCPDS file no. 01-081-8227) occurred at the most intensified peak located at 31.148° and nearly entire peaks in the diffractogram of Perlis dolomite, which is similar to previous studies. (Abdullah et al., 2021; Sompech et al., 2016). Furthermore, the minority of the peaks in the diffractogram of raw Perlis dolomite were assigned to the phases of calcium magnesium silicate, $CaMgSi_2O_6$ (JCPDS file no. 04-008-8060) and sodium aluminium silicate, $NaAlSi_3O_8$ (JCPDS file no. 04-021-1621), where the crystal structures of the phases were monoclinic and anorthic, respectively.

Figure 3(b) presents the diffractogram of heated dolomite that demonstrated apparent changes in the XRD patterns because of the phase transformation after the heat treatment. The phases of $CaMg(CO_3)_2$ from raw dolomite have decomposed into some single phases individually, where most of the significant peaks were accredited by cubic structures of CaO (JCPDS file no 04-005-4398) and MgO (JCPDS file no 04-016-2776) phases. This result proved that the primary constituents of the heated dolomite were CaO and MgO, which were similar to the findings from Abdullah et al. (2021). The CaO phases were observed at 32.508°, 37.642° and 54.166° while the MgO phases were at 43.184° and 62.564°. Moreover, there were also found in the heated dolomite powder, involving sodium oxide, NaO_2 (JCPDS file no 04-005-4449), calcium silicon oxide, Ca_3SiO (JCPDS file no 04-021-0148), aluminium, Al (JCPDS file no 04-014-0129) and silica, SiO_2 (JCPDS file no 04-015-7161). These phases, formed in the heated dolomite, correspond well with the previous results and discussions made for the XRF analysis.

According to the phase analysis, it can be highlighted that the heat treatment at 1000°C is particularly required and sufficient to allow the transformation of dominant carbonate phases of raw dolomite, $CaMg(CO_3)_2$ into more significant and more stable CaO and MgO phases in the heated Perlis dolomite powder. This statement can be supported by the findings from Abdullah et al. (2021) where the phases of dolomite showed significant changes at 800°C and 1000°C, while no apparent changes were found at 400°C and 600°C. Therefore, the phase transformation of this Perlis dolomite is found to be equivalent and develops the thermal decomposition and reaction mechanism, as shown previously in Equations 1 to 5. The heat treatment on the raw dolomite powder is able to change the phase transformation of the main compound of the dolomite carbonate matrix into CaO and MgO phases because of the high thermal energy that breaks the bonds between the chemical atoms in the dolomite structures (Ramli et al., 2022). Therefore, the formed phases of CaO and MgO after the heat treatment have arisen and affirm the potential and positive perspective for producing bioceramic-based material, especially for calcium and magnesium-based bioceramics.

These phases have been manifested to be significant and play a crucial part in biomedical applications, especially in the practices of bone engineering tissues (Choudhary et al., 2015; Collin et al., 2021; Zadehnajar et al., 2021).

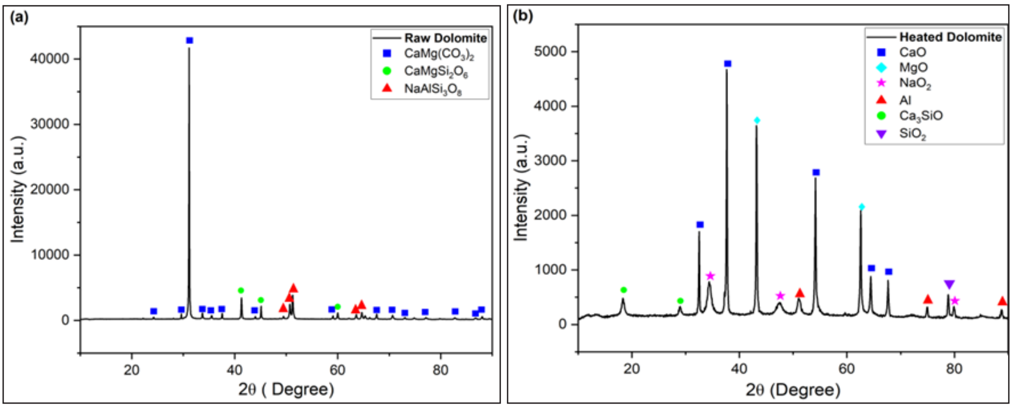

Figure 3. Diffractogram of (a) raw dolomite and (b) heated dolomite

Functional Group Analysis

Figure 4 illustrates the infrared spectral band of raw and heated Perlis dolomite. For raw dolomite, an outstanding and wide asymmetric stretching band at 1421 cm^{-1}, narrow bending bands around 875 cm^{-1} and 726 cm^{-1} have proved the existence of complex carbonate mineral group proportional with the v_3, v_2 and v_4 vibration modes of $(CO_3)^{2-}$ groups (Harrati et al., 2022; Shahraki et al., 2009) which incorporates with CaO and MgO compounds as its prime absorption components in the dolomite (Abdullah et al., 2021). The intense bands at 668 cm^{-1} and 3642 cm^{-1} were attributed to the appearance of CaOH bonds in the raw dolomite powder. Meanwhile, the weak bands at 875 cm^{-1}, 1005 cm^{-1} and 1080 cm^{-1} indicated the Si-O vibration bond, which affirmed the statement of silicate occurrence as the small impurities in the raw dolomite as found in the chemical composition analysis (Mohammed et al., 2013). Moreover, the band at 2895 cm^{-1} represented organic matter (C-H bond), while the bands at 3400 cm^{-1} and 3875 cm^{-1} were distinguished as stretching bonds of O-H caused by the moisture contents in the carbonates of dolomite (Buyang et al., 2023; Harrati et al., 2022; Kurtulbaş et al., 2020).

According to the FTIR spectrum analysis of heated dolomite, the heat treatment does not cause any apparent changes compared to the raw dolomite, but there were a few transmission bands that have decreased or disappeared (Resio, 2023; Yuliya et al., 2023), and there was also the formation of new peaks observed. The complex bonds of raw dolomite at 1421 cm^{-1} turn into a sharp band after the heat treatment introduces the decomposition of dolomite, $CaMg(CO_3)_2$, into CaO and MgO formation in the heated

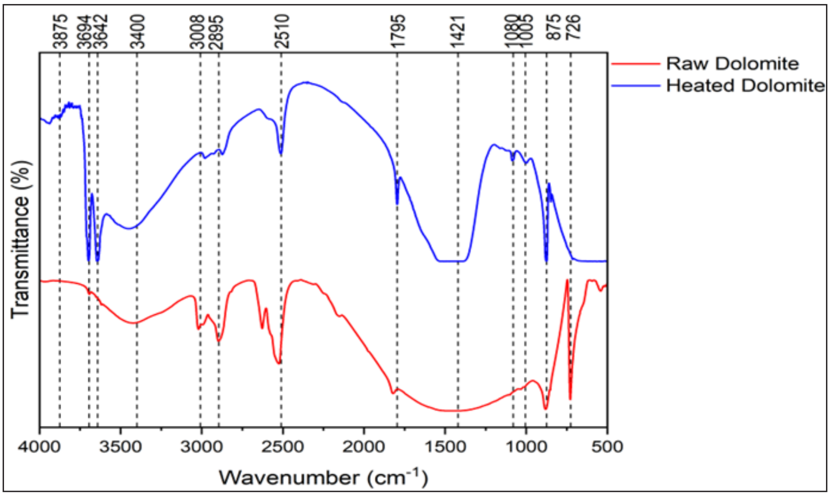

Figure 4. FTIR spectrum of raw and heated dolomite

dolomite powder. This is because the broad band of raw dolomite with overlaying vibrational modes has decomposed into simpler oxide bonds caused by the applied thermal decomposition. Besides, the bond segregation between the main absorbing functional groups of the carbonates from raw dolomite $(CO_3)^{2-}$ has caused the low concentration of carbonate ions subsequently confronted by the concentration of CaO and MgO, which lessens the carbonate intensities and causes the formation of the new peak exhibiting the oxide compounds in the heated dolomite powder. The strong and intense bands at 3642 cm^{-1} and 3694 cm^{-1} existed after the heat treatment, particularly due to $Ca(OH)_2$ formation from the hydroxyl bonds (Abdullah et al., 2021; Shahraki et al., 2009).

This spectral band transformation introduces the decomposition of the carbonate group of dolomite into the oxide compounds of CaO and MgO. The weak bands at the range of 875-1080 cm^{-1} have merged together to create a strong band indicating the presence of silica, Si-O bond as an impurities substance in the dolomite in the form of silicates vibration mode (Abdullah et al., 2021). The reduced intensity of the bands at 2895 cm^{-1} and 2510 cm^{-1} in the heated dolomite was assigned as the residue of the C-H stretching bonds from organic matter and O-H stretching bonds from the hydroxyl groups, respectively. The insignificant presence of H-bonds can be decomposed completely with high temperatures during the sintering process during bioceramic production. It has been clarified by Shahraki et al. that there is a tendency for the formation of bioceramic, which is wollastonite, due to the decomposition between calcite and quartz in the dolomite mineral after heat treated at 950°C, where its bands centred at 1005 cm^{-1} and 1080 cm^{-1} (Shahraki et al., 2009).

This result shows that the raw dolomite assigned to the changes occurred on the functional groups of Perlis dolomite after the heat treatment. The heat treatment has segregated the functional group of dolomite matrix bonds by decomposing the carbonate groups and creating CaO and MgO bonds individually. In addition, it also showed the existence of hydroxyl groups in the heated dolomite, as they were important in enhancing the absorption performance, especially for bioceramic production purposes. Hydrogen bonds have significant roles in interacting with biological tissues, providing biocompatibility reactions and accelerating cell proliferation and tissue growth in bone tissue engineering applications. Thus, Perlis dolomite with applied heat treatment has very good potential as the main precursor and is suitable for use in the production of bioceramics.

CONCLUSION

The investigation of the effect of the heat treatment process on the Perlis dolomite was completed, and the objective was successfully achieved. It can be summarised that the heat treatment process done on the raw dolomite at 1000°C for 4 hours had a significant impact and made significant changes to the physical, chemical and structural properties of Perlis dolomite. However, this process is partially decarbonised, and the dolomite phase has been decomposed into multiple oxide phases. The heat treatment process is proposed to be extended to more than 4 hours to complete the reaction. The heat-treated dolomite was very suitable and has potential as a main precursor for bioceramic production. The dolomite was predominated by CaO and MgO, vital in bone tissue engineering applications. The heat treatment at 1000°C for 4 hours, as carried out in this study, is necessary and has shown significant positive impacts on the Perlis dolomite powder. The utilisation of dolomite in bioceramic production should be explored widely to maximise its potential.

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