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Effect of silica additions on the structure, phase transformation, and compressive strength on sintered Perlis dolomite mineral

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ABSTRACT

Natural dolomite sedimentary rocks, which can be found in abundance in Perlis, are carbonate minerals rich with calcium and magnesium elements. Both elements are commonly found in bioceramic materials, particularly for hard tissue implants. Even though raw Perlis dolomite powder has other various elemental compositions, it is deficient in important components, such as silica and phosphate, which are commonly used as glass network formers, therefore limiting its potential application as bioceramic materials. This study examines the effects of different percentages of silica powder addition on sintered Perlis dolomite, and the changes in its structure, phase transformation, and compressive strength were analyzed. Perlis dolomite with different ratios of silica (25–45 wt. %) was ball milled, compacted into pellets, and sintered at 1250°C for 4 hours. The results showed that at lower silica content (≤30 wt. %), the MgO phase becomes the dominant phase. However, increasing silica content promotes the formation of akermanite, monticellite, and merwinite phases, although the merwinite phase disappears at 45 wt. %. Higher silica content in dolomite enhances phase stability by increasing the akermanite phase, reduces shrinkage, and increases densification of the sintered samples, which contributes to the improvement in compressive strength.

Keywords: *Perlis dolomite, Silica powder, Bioceramic*

1. INTRODUCTION

The development of advanced ceramic technology has expanded into diverse fields, notably the biomedical industry. Up to this present time, bioceramics have gained significant recognition in the field of regenerative treatment, especially for their vital contribution in bone repair and replacement. The ceramic materials are specially designed and engineered to correspond and interact properly with the living tissues, and exhibit great responses in the biological system [1]. Besides, the high quality of bioceramics is largely determined by their capability to mimic the compositions of natural bone with an absence or minimal complications ahead.

There are possibilities for the damaged bone to show deteriorating conditions because of inefficacy of treatment due to the complication of bone transplant suitability, risk of inflammatory reaction transfer, and adverse immunological response [2]. Currently, prominent bioceramics that are often used in orthopedics and dentistry areas, such as hydroxyapatite and tricalcium phosphate (TCP), are widely known due to their ability to promote osteointegration, provide cell attachment, and encourage new bone formation. However, a major challenge remains in compensating excellent bioactivity performances with appropriate mechanical strength and controllable degradation rate. In the gradual advanced development in bioceramic production, naturally derived materials have received increasing interest as alternative material for the bone regeneration applications, substituting the synthetic materials, which considerably consume high costs.

Dolomite is one of the natural minerals that is widely explored in order to achieve its benefits owing to its unique composition ($CaMg(CO_3)_2$), which is also known as ‘dolostone’ [3–5]. The dolomite mineral plays significant roles as a raw material for bioceramic synthesis due to its calcium (Ca) and magnesium (Mg) content. Both of these elements are found essential in bone regeneration treatment, as Ca is a prime source of bone matrix while Mg allows the bone tissue growth and repair functions [2]. Despite the high level of carbonate compositions in the dolomite, its great thermal stability enables decomposition of carbonate into calcium oxide (CaO) and magnesium oxide (MgO) after the calcination process. Accordingly, the dolomite represents a promising precursor for bioceramic synthesis, offering essential elements for bone regeneration while providing thermally stable oxide phases suitable to form bioactive compounds.

However, there is a scarcity of published reviews and scientific findings regarding the development and application of dolomite-derived bioceramics over the past few years. An investigation by Harrati et al. has used

Moroccan dolomite with natural perlite material with a ratio of 75:25 wt. % [6]. They found that akermanite phase has formed with high bending and tensile strength up to 7.71 MPa and 3.56 MPa, respectively. A formation of a hydroxyapatite layer also was observed on the surfaces of the resulting material, which induces excellent bioactivity performances. Furthermore, another study also came up with an approach of Moroccan dolomite utilization associated with slag waste from the steel industry to prepare akermanite bioceramic [7]. They reported that the bioceramic has a porosity of 41.4%, water absorption of 24.8%, and flexural strength of 13.8 MPa with the non-existence of microcracks on the surface of the final materials.

In Malaysia, there is a large abundance of dolomite minerals that can easily be found in natural sedimentary rocks in the vicinity of Chuping, Perlis. The Perlis dolomite is well recognized as 'Batu Reput' by the locals [8–10]. This is because the dolomite is known for its brittle properties, which have a high possibility to crumble under weathering conditions, and lead to the soft and friable texture formation compared to compact limestone. Until now, Perlis dolomites are still explored for limited applications, which focus on soil treatment in the agriculture industry, and road pavement and building in the construction industry [11–13]. In the agriculture field, the dolomite is processed into soil fertilizer in order to neutralize the pH of the soil, boost root growth, and enhance the structure of the soil by providing the Ca and Mg nutrients [14]. Moreover, the dolomite is used as the road base incorporating asphalt aggregate before it is paved due to its durability and stability under heavy loads. Meanwhile, in the building construction, the dolomite acts as reinforcement material associated with the cement to enhance the structure and strength of the concrete.

Thus, the novelty of this study lies on the utilization of Perlis dolomite as an alternative natural material in bioceramic formation for bone regeneration purposes. This is because there are deficient scientific data and findings of Perlis dolomite as raw material in the bioceramic preparation. This study aims to fill in the gap by systematically investigating its structural, phase transformation, and mechanical properties on the sintered Perlis dolomite-derived ceramic with silica addition, and determining its suitability for bone regeneration applications. The use of locally sourced Perlis dolomite not only could provide cost-effective biomaterial development but also decrease the over-reliance on synthetic or imported raw materials.

Although Perlis dolomite is rich with CaO and MgO, and their benefits, both of these elements alone are unable to form high-level performances of bioceramic. This is because it lacks glass network formers, such as silica. Silica (SiO_2) is a well-recognized additive in bioceramic processing, playing a significant role in improving the structural and biological activity of the resulting materials. In the processing and application of dolomite-based ceramics, silica can react together with the CaO and MgO and induce various bioceramics that lie under the $CaO-MgO-SiO_2$ system, such as akermanite, monticellite, merwinite, and diopside [15]. These phases are not only thermodynamically stable at high temperatures but also promote great bioactivity performances by inducing apatite formation, which is promising for biomedical applications. Besides, the addition of silica in the dolomite-based ceramics also can have a significant impact on the structural, phase transformation, and mechanical strength of the final materials.

Therefore, this study will investigate the effect of silica addition with a variety of compositions on the phase transformation, and mechanical properties of sintered Perlis dolomite, particularly concerning bone regeneration applications. The outcomes of this study are expected to enhance the material value of this local dolomite mineral, and support the advancement of locally sourced minerals as the cost-effective alternative materials for the biomedical industry. Besides, the findings are expected to contribute to the development of high-performance bioceramic phases with improved mechanical performances suitable for the biomedical applications.

2. METHODOLOGY

2.1. Raw Materials

Fine powder of raw Perlis dolomite mineral was supplied from the local quarry at Chuping, Perlis, Malaysia. Meanwhile, silica powder, which was acquired from Ipoh Ceramic, Perak, Malaysia was used as an additive material in the preparation of dolomite-derived bioceramic. Heat treatment was implemented on the dolomite powder through a calcination process at 1000°C for 4 hours with a heating rate of 10°C/min. This calcination process was performed for the purpose of decarbonization, and removal of impurities and volatiles substances from the raw dolomite minerals. The chemical compositions of Perlis dolomite and silica powder were analyzed via X-ray fluorescent (XRF) by Philips PANalytical MiniPAL 4 machine, where the percentages expressed the amount of oxides contained in the mineral. The reported findings of the raw Perlis dolomite were CaO: 65.07%, MgO: 23.10%, Na_2O: 8.00%, Ag_2O: 1.46%, and Al_2O: 1.30%, then the chemical compositions were slightly changed after the calcination process executed, where CaO: 54.55%, MgO: 30.50%, Na_2O: 12.00%, Ag_2O: 0.29%, and Al_2O: 1.20%. Meanwhile, the results of the silica powder were SiO_2: 97.74%, and Al_2O_3: 2.00%: The calcined dolomite and silica powder were sieved to achieve homogeneous particles of powders size below 150 μm.

2.2. Sample Preparation

The fine calcined Perlis dolomite and silica powder were mixed together uniformly through mechanical activation approach via planetary ball milling (Mono Mill Pulverisette 6-classic line, Fritsch, Germany). Six mixtures of calcined dolomite were prepared with different ranges of ratio within 25, 30, 35, 40, and 45 wt. % of silica powder, and labeled in the results and discussion as 25S, 30S, 35S, 40S, and 45S respectively. The deionized water was used as the milling medium during the wet ball milling with the ratio of deionized water to powder was 3:1. Meanwhile, the ratio of balls to mixture powder was 10:1. The milling operation was adjusted with a milling speed of 450 rpm for 4 hours in total. Each cycle of the milling spent an hour, and continued for further 3 repetitions with regular direction in reverse for each cycle.

Then, the mixtures were dried in an oven at 80°C overnight for drying purposes. The dried powders were ground, and sieved to obtain fine uniform powders. Next, 0.5 g sodium alginate powder (Sigma-Aldrich, Germany) was used during the preparation of binder solution, where it was diluted with 20 mL deionized water. It was manually stirred until the solid substances completely dissolved until no visible granules remained in the solution. Each 1.0 g of dolomite-silica powder mixture sample used 0.5 wt. % amount of binder solution. Subsequently, the powder mixtures were pressed into pellet samples via hydraulic press machine (Nichi T-61230A). Cylindrical carbon steel mold with diameter of 12 mm was used during the compaction process with pressure set up to 200 MPa for 5 minutes loading. Finally, the pellets were sintered at 1250 °C for 4 hours with a heating rate of 5 °C/min in the furnace (LT Furnace SIC4-1600).

2.3. Characterization of Samples

For structural analysis, the samples were characterized by using X-Ray Diffractometer (XRD) (Test Instrument Bruker, D2 Phaser Germany 2010). XRD was performed to investigate the phase transformations of the samples. The XRD scans were conducted with a step scanning of the angle diffraction (2θ) at the range of 10° to 80° with each sample scanned over a duration of 10 minutes. The resulting data of XRD diffractogram were analyzed using X'pert-HighScore Plus software in order to identify the phase compounds that occur in the samples. The phases of the experimental XRD were matched referencing the crystallographic database from the International Centre for Diffraction Data – Powder Diffraction File (ICCD PDF). The XRD analysis was performed on the raw and calcined Perlis dolomite. It was also executed on the sintered ceramic to investigate the effect of silica on the sintered Perlis dolomite.

For Fourier Transform Infrared spectroscopy (FTIR) analysis, it was implemented by using the Perkin Elmer machine to identify the chemical bonds, and functional groups that are affected by varying ratios of silica addition in the dolomite. The potassium bromide powder (KBr) pellet preparation was chosen in this study owing to its superior infrared transparency, and suitability for analyzing solid powdered samples. Each sample was prepared by mixing 1.0 wt. % of the sample with KBr powder, and compacted into thin pellets. The prepared pellets were scanned in the wave length within the range of 400–4000 cm^{-1} with a spectral resolution of 4 cm^{-1}. The obtained spectra were analyzed to observe vibrational bands associated with the functional groups of the materials.

For the mechanical analysis, the powder samples were compacted under 200 MPa loads by using 9 mm diameter of cylindrical carbon steel mold with the ratio of diameter to thickness is 1:2 according to standard of ASTM C1424. The samples were sintered at 1250°C for 4 hours, and readily for the compressive strength testing. The testing was performed by using 50kN pressure load of Universal Machine Testing (UTM) (Shimadzu AG-X Plus). Each sample was located at the centre between the upper and lower compression plates of the UTM. The upper plate was lowered until a minimal gap remained between the sample and the plate, as thin as a piece of a paper. Calibration was performed from zero prior to initiate the compressive strength test. The compressive load was applied at a constant crosshead speed of 0.5 mm/min until failure was observed on the sample. The recorded data were analyzed by using Trapezium X software.

3. RESULTS AND DISCUSSION

3.1. Phase Transformation Analysis

Figure 1 illustrated the phase transformation of raw and calcined dolomite. Figure 1(a) presented that the XRD diffractogram of raw Perlis dolomite was mainly dominated by the calcium magnesium carbonate, $CaMg(CO_3)_2$ phase (JCPDS file no. 01-081-8227), with a crystal system of hexagonal structure. These $CaMg(CO_3)_2$ phases formed at the most outstanding peak were observed at 31.148°, and also most likely at the entire peaks in the diffractogram of Perlis dolomite, which is found to be similar to the previous studies. [16, 17]. Furthermore, the peaks in the diffractogram of raw Perlis dolomite were also attributed to minor 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 1(b) displayed the diffractogram of calcined dolomite that exhibited apparent changes in the XRD patterns because of the calcination process that caused the phase transformation. The phases of $CaMg(CO_3)_2$ from the previous raw dolomite have decomposed into some individual single phases, where most of the significant peaks were assigned by CaO (JCPDS file no. 04-005-4398) and MgO (JCPDS file no. 04-016-2776) phases, where their crystal systems were both formed by cubic structures. This finding verified that the major constituents of the calcined dolomite were CaO and MgO, which agrees with the findings from Abdullah et al. [16]. The CaO phases were obviously found to be located at 32.508°, 37.642°, and 54.166° while the MgO phases were at 43.184° and 62.564°. Besides, the calcined dolomite powder also has a small amount of other phases, including 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 were found in the calcined dolomite, which is well-correlated with the previous statement of the XRF analysis in the 2.1. However, these minor phases can be disregarded, as they are expected to further decompose upon exposure to higher sintering temperatures in the fabrication of sintered ceramics derived from calcined dolomite and silica.

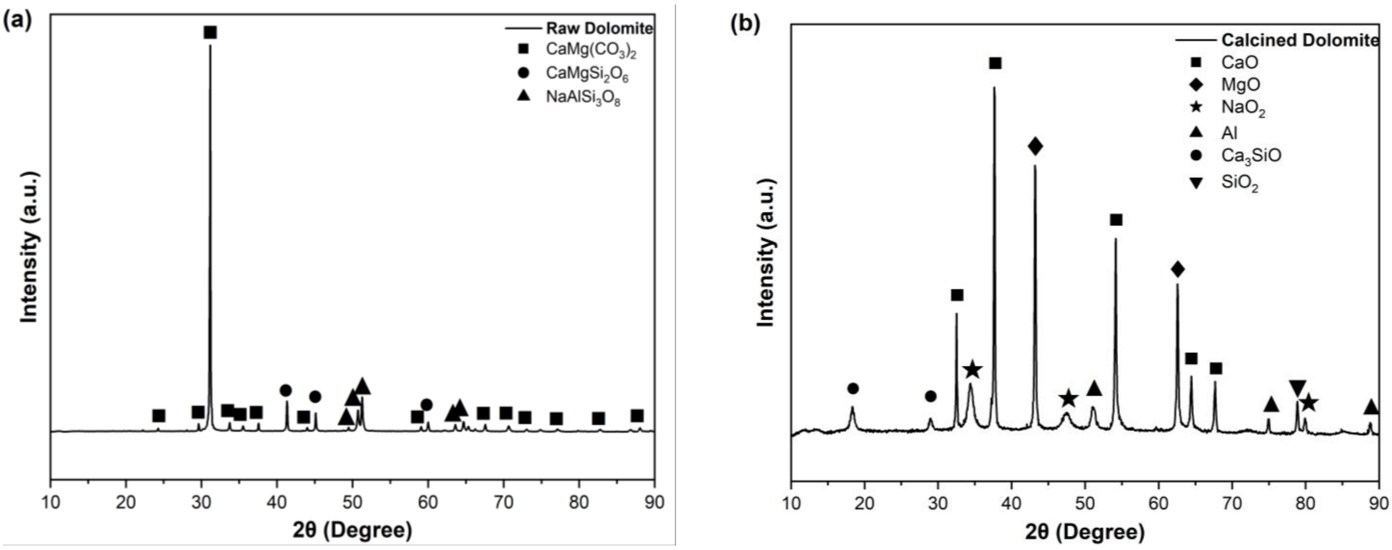

Figure 1. XRD diffractogram of (a) raw dolomite and (b) calcined dolomite

According to this phase analysis, it can be emphasized that the calcination process at 1000°C is significantly necessary, and already sufficient to allow the dominant carbonate phases of raw dolomite, $CaMg(CO_3)_2$ to transform into more significant and more stable CaO and MgO phases in the calcined Perlis dolomite powder. This statement can be supported by the previous findings from Abdullah et al. [16], where they reported that the phases of dolomite presented significant transformation at high temperatures around 800°C and 1000°C, while no obvious changes were observed at low temperatures, such as 400°C and 600°C. The calcination process on the raw dolomite powder promotes the phase transformation of the main compound of the carbonate matrix into CaO and MgO phases due to the breaking of bonds between the chemical atoms in the dolomite structures caused by the high thermal energy [18].

Therefore, the formed phases of CaO and MgO after the calcination process have induced, and confirmed their potential and positive perspective of Perlis dolomite for bioceramic-based material fabrication, particularly for calcium- and magnesium-based bioceramics. These phases have been promising candidates for essential roles and play a critical part in biomedical applications, especially in the treatment of bone engineering tissues [19–21]. From this finding, it can be supported that the Perlis dolomite with further incorporation of silica could enhance these properties by promoting the development of stable silicate phases, which improve both the structural integrity and mechanical performances of the material for bone regeneration practices.

Figure 2 showed the phase changes observed in the sintered dolomite samples under varying ratios of silica addition. At lower amounts of silica addition (25S and 30S) remarkable MgO phases (JCPDS file no. 00-045-0946) with strong diffraction peaks at 42.95°, 62.36°, and 78.65° have been created. In the 25S, it was found that there is a minor existence of akermanite ($Ca_2MgSi_2O_7$) and calcium silicate (Ca_2SiO_4) phases, while the 30S showed the presence of gehlenite-akermanite ($Ca_2Mg_{0.08}Al_{1.84}Si_{1.08}O_7$) and Ca_2SiO_4 phases. Furthermore, at higher silica contents (≥35 wt. %), the diffraction patterns indicated significant phase transformations. The MgO phase has been transformed into other oxide phases, including akermanite ($Ca_2MgSi_2O_7$) (JCPDS file no. 98-002-7381), monticellite ($CaMg(SiO_4)$) (JCPDS file no. 98-001-7233) and merwinite ($Ca_3Mg(SiO_4)_2$) (JCPDS file no. 98-001-1933), where the crystal systems of these phases were built by tetragonal, orthorhombic, and monoclinic structures, respectively.

Based on these findings, it showed that the synthesis of dolomite at 1200°C with low content of silica leads to the formation of MgO and Ca_2SiO_4 phases only without any presence of $CaMgSiO_2$-related phases yet. This is because the silica content in the dolomite mixture is insufficient to produce Ca-Mg-Si compounds. In this case, the available silica primarily reacts first with CaO from the dolomite powders due to its higher thermodynamic stability before the silica reacts with the MgO [22–24]. Thus, it explained the existence of residual MgO as distinct phases in the XRD diffractogram. Meanwhile, with increased silica addition in 35S and 40S, phases such as akermanite, monticellite, and merwinite were initiated to develop. This is due to the higher availability of SiO_2, which reacts with CaO and MgO derived from dolomite decomposition during sintering, thereby facilitating the formation of calcium–magnesium silicate phases. These phases are thermodynamically favorable at elevated silica content, and contribute to the stabilization of bioactive ceramic structures.

However, as the silica content increased further to 45S, the merwinite phase was diminished, and was eventually replaced by the akermanite and monticellite. Notably, in the 45S sample, the merwinite phases were completely absent due to their lower stability at high silica concentrations. This leads to the remarkable formation of the akermanite and monticellite phases resulting in a multiphase ceramic material. Additionally, an outstanding peak at 31.24° indicated a well-defined, crystallized, and prominent presence of akermanite. These findings confirmed that the addition of silica in the sintered dolomite leads to the formation of biocompatible compounds, such as akermanite and calcium silicate. Therefore, Perlis dolomite has

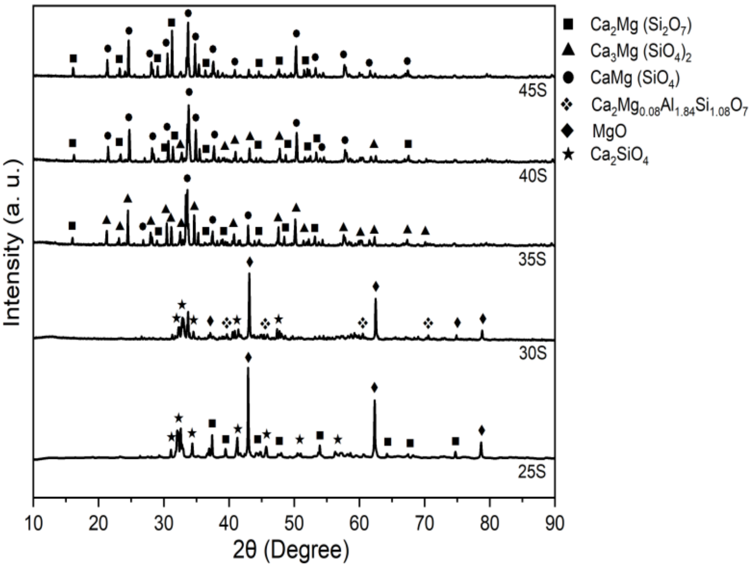

Figure 2. XRD diffractogram of sintered dolomite with varying ratios of silica

demonstrated its strong potential as a raw material for bioceramic production associated with the silica as an additive material to form these bioactive silicate phases, making it promising for bone regeneration applications.

3.2. Functional Group Analysis

Figure 3 presented the FTIR spectra of the sintered dolomite/silica samples with varying silica concentrations. Despite the difference in silica content, all samples exhibited similar spectral patterns. A distinct absorption band was observed at 680 cm^{-1}, which corresponded to the vibrational mode of O─Si─O [25, 26]. Additionally, peaks at 848 cm^{-1} and 932 cm^{-1} were attributed to Si─O stretching vibrations, meanwhile, a prominent peak at 1021 cm^{-1} occurred due to a symmetric stretching vibration of Si─O─Si bonds [6, 25]. These bands confirmed the presence of silicate bonding and the formation of silicate structural networks in the samples, which tally with the findings from XRD analysis that has been discussed previously.

In the sample of 25S, additional bands appeared at 3638 cm^{-1} and 3700 cm^{-1} attributed to the weak absorption related to hydroxyl groups, corresponding to O─H stretching mode vibrations [6]. Furthermore, the absorption peak at 1480 cm^{-1}, linked to the H─O─H bending mode of absorbed water molecules [2, 26], was found to gradually decrease as the silica content increased in the dolomite mixture. This reduction happens due to the CaO and MgO components from the dolomite that reacted with the higher silica levels, promoting the formation of denser ceramic phases, such as akermanite, merwinite, and monticellite. As a result, the surface area available for water absorption diminished. Notably, a sharp peak at 594 cm^{-1} appeared in the high silica samples (35S, 40S, and 45S), corresponding to the Ca═O stretching vibration [6], which is consistence with the presence of akermanite, merwinite, and monticellite. The formation of these phases is further supported by the existence of bands at 462 cm^{-1} and 520 cm^{-1}, represented the bending mode of O─Mg─O [6] and O─Ca─O [2], respectively.

Overall, the obtained FTIR result aligns well with the XRD analysis, relatively confirming the formation of akermanite, merwinite, and monticellite phases under the influence of silica addition in the sintered dolomite samples. This complementary agreement between FTIR and XRD provides strong validation of the structural and compositional transformations occurring during the sintering process. The presence of these silicate phases is particularly significant, as they are well-documented for their bioactivity, osteoconductivity, and compatibility with bone tissues. Hence, the results not only verify the successful phase development but also emphasize the potential of silica-modified dolomite as a promising precursor for high-performance bioceramics in bone regeneration applications.

3.3. Mechanical Strength

The mechanical strengths of the sintered samples are presented in Figure 4. It can be observed that the addition of silica in the dolomite mixtures has a positive impact on

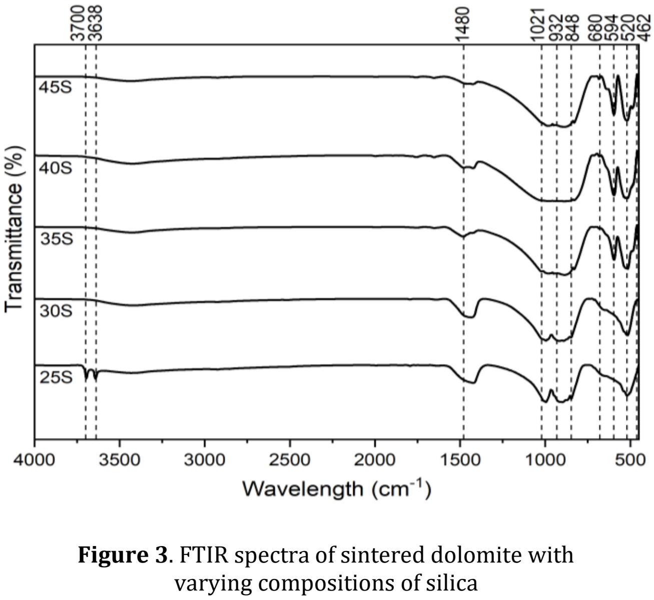

Figure 3. FTIR spectra of sintered dolomite with varying compositions of silica

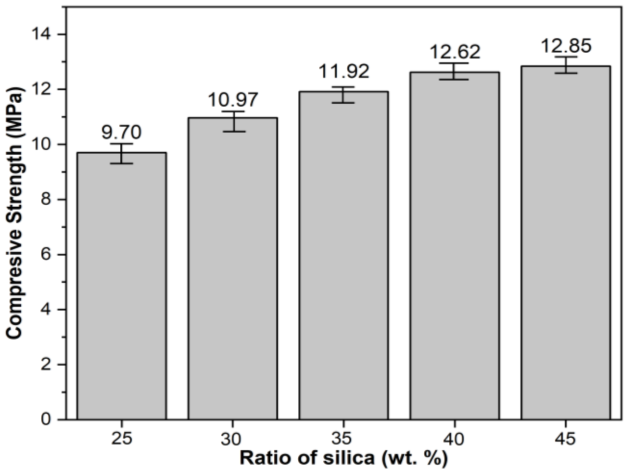

Figure 4. Mechanical strength of the sintered dolomite with varying compositions of silica

the compressive strength of the samples. In general, the compressive strength of the samples increased relatively with the increasing silica addition. The values recorded for each sample meet the minimum requirement of human cancellous bone, which is in the range of 2–12 MPa [21, 27–29]. The sample of 45S showed the highest value in the compressive strength (12.85 MPa) compared to the other samples, which is in agreement with the discussion in XRD analysis regarding the presence of dense akermanite, and monticellite in this sample. These phases contribute to its mechanical properties.

Additionally, the compressive strength of the 45S has exhibited about twofolds higher than akermanite scaffold and tricalcium-phosphate (β-TCP) from previous studies, which is approximately about 5.92 MPa [30] and 6.89 MPa [31], respectively. Although the compressive strength of the sintered Perlis dolomite associated with silica in this study falls within the range of hydroxyapatite, it is much more considered to lean towards the lower range of hydroxyapatite (2–100 MPa) [32]. This improvement highlights the beneficial role of silica addition in enhancing the mechanical stability of sintered Perlis dolomite. Consequently, the material shows strong potential for non-load-bearing biomedical applications, particularly in bone regeneration scaffolds, where moderate strength and bioactivity are crucial at the highest silica content.

Overall, this result showed that the silica additions have significantly affected the mechanical properties of the samples by improving barrier particles through the reactivity of oxygen saturated [33]. This is closely related to the stable phases of akermanite and monticellite. The silica addition tends to strengthen the grain boundaries, and enhance the mechanical properties of the samples. The increasing silica contents support the formation of stable silicate phases, contributing to improved strength, confirming the critical role of silica in phase stability and microstructural integrity. These findings highlight the feasibility of utilizing locally sourced dolomite as an alternative precursor as well as silica as an additive material for cost-effective and functional bioceramics. Besides, optimizing the silica composition is essential to tailor the performance of dolomite-based bioceramics for specific biomedical applications. This study provides a foundation for advancing locally sourced dolomite into high-value biomaterials.

4. CONCLUSION

In conclusion, an investigation on the effects of varying silica contents on the structure, phase transformation, and mechanical properties of sintered Perlis dolomite at 1200°C was successfully accomplished. The results demonstrated that the increasing silica contents led to the significant formation of akermanite, merwinite, and monticellite phases, as confirmed by both chemical structure and functional group analysis. Additionally, the silica addition in the sintered dolomite promoted great mechanical strength of the samples, which lay in the range of strength values of natural cancellous bone. It has been found that the higher silica levels enhanced the compressive strength of the samples corresponding to the dense crystalline structures and grain boundaries due to oxygen-saturated reactivity that contributes to the enhanced mechanical properties, making them more suitable for bone regeneration applications. Further investigation on the bioactive properties via in vitro analysis is essential, particularly to mimic and reach the optimum performances of human cancellous bone in future work. Increasing silica content and refining the sintering parameters are also recommended to optimize the bioceramic phase development and performances.

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