The Effect of Acid Treatment Time, Particle Size, and Synthesis Method on the Physical and Mechanical Properties of Dental Materials Produced from Flue Gas Desulfurization (FGD) Gypsum

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Abstract

FGD gypsum, a byproduct of coal-fired power plants, is readily available and relatively inexpensive, which makes it an ideal material for a variety of applications. This study considered the use of FGD gypsum as a substitute for natural gypsum in dental materials. The goal of this research was to investigate how acid treatment time, particle size, and the synthesis method impact the physical and mechanical properties of dental materials to be used in a dental study model for training in dental sciences, and for casting a gypsum model after the removal of the impression material from the patient's mouth. The study used various sulfuric acid treatment times (15, 30, and 60 min), particle sizes (less than 0.1 mm, 0.1-0.35 mm, and 0.4-0.45 mm), and synthesis methods (Method A for dental plaster and Method B for dental stone). From the results, an acid treatment time of 15 min was sufficient for removing impurities from the FGD gypsum while enhancing the compressive strength. The smaller particles provided higher compressive strength than the larger particles. FGD gypsum became lighter in color when treated with sulfuric acid, and the crystal structure had a rough and porous surface. The synthesis methods had a significant influence on the physical properties of dental gypsum. The increased alpha calcium sulfate hemihydrate (α-HH) phase content resulted in improved compressive strength. The gypsum synthesized using Method B exhibited the highest compressive strength due to the presence of the α-HH phase of 65.9%. While gypsum synthesized using Method A contained a α-HH phase of 58.9%. For further study, once the suitable conditions for synthesizing gypsum that meet the compressive strength requirements of the ISO standard for dental materials are achieved, there will be ongoing research and development to improve various properties. Additionally, practical applications will be considered, such as using it in conjunction with modern techniques such as 3D printing instead of traditional die-casting methods.

Keywords: dental material, dental stone, dental plaster, FGD gypsum, compressive strength

Introduction

Flue gas desulfurization (FGD) gypsum is a byproduct of coal-fired power plants. It is obtained through a process where sulfur dioxide (SO_2) is scrubbed from flue gas, which results in the production of gypsum (Guan et al., 2021). This gypsum is often disposed of in landfills and ponds, leading to environmental concerns. The color of FGD gypsum is commonly grayish yellow or grayish white, mainly due to a high content of unburned carbon in the flue gas and a small amount of $CaCO_3$ particles (Yichao et al., 2020). FGD gypsum contains the primary type of gypsum, calcium sulfate dihydrate ($CaSO_4.2H_2O$) (Lee et al., 2008), but it may also contain trace amounts of toxic elements such as Pb, As, Hg, and Cd. Nevertheless, the composition of FGD gypsum varies depending on factors such as the region, the origin of the coal, and the FGD process (Koralegedara et al., 2019). The chemical composition of FGD gypsum is similar to that of natural gypsum (Caillahua & Moura, 2018), making it a viable alternative to conventional gypsum. Additionally, FGD gypsum is readily available and relatively inexpensive, which makes it an ideal material for a variety of applications such as building materials, ceramic

molds, soil amendment (EPA, 2008; Yichao et al., 2020), dental materials (Panpa, 2002), and water treatment processes (Koralegedara et al., 2019).

Dental gypsum is a crucial component in the production of dental materials, appliances, and instruments. It is widely used in creating tooth impressions, mouth and tooth models, and dental restorations (GII, 2023). According to ISO 6873:2013, there are five types of dentistry gypsum products: 1. Dental plaster for impressions, 2. Dental plaster for mounting (Class 1) and for models (Class 2), 3. Dental stone for models, 4. Dental stone (high strength, low expansion) for dies, model bases and CAD/CAM dies, and 5. Dental stone (high strength, high expansion) for dies when this degree of expansion is necessary for shrinkage compensation of some materials used in dental restoration (ISO 6873:2013, 2013). The global market for dental gypsum was valued at US$117.1 million in 2021, which is expected to have about a 5% annual compound growth from 2023 to 2031 (Precision Reports, 2023). This growth can be attributed to several factors, including an increase in dental tourism, the growing demand for cosmetic dentistry, and a rise in oral health awareness. In addition, adults over 65 are a growing demographic for dental practices (GII, 2023).

One possible strategy that has arisen for the production of increased volumes of dental gypsum is the usage of FGD gypsum as a substitute for natural gypsum in dental materials. Several researchers investigated the effects of several factors on the physical properties of dental gypsum, such as synthesis methods (Akinnifesi & Ogunbodede, 2012), setting time (Imelda et al., 2020), additives (Azer et al., 2008), and the amount of silica oxides in the material (O'Brien, 2022). Kostic-Pulek et al. (2005) modified the hydrothermal method for the preparation of the alpha-hemihydrate calcium sulfate with the FGD gypsum and acid solution ratios of 0.125, 0.250, 0.500, and 0.750 g/cm^3. They found that the mixing solution ratios of 0.125-0.500 g/cm^3 had both alpha and beta hemihydrate forms. They concluded that increasing the mixing solution ratios increased the fraction of the alpha form in the final product as well as the reaction rate and the average lengths of the alpha hemihydrate single crystals (Kostic-Pulek et al., 2005).

However, few studies on the production of dental materials from FGD gypsum have been published. Panpa (2002) studied the production of dental stone by treating FGD gypsum using ultrasonication and acid-leaching methods. They reported that FGD gypsum treated with sulfuric acid could remove more impurities, and the dental stone produced from treated FGD gypsum had properties equivalent to natural gypsum with a compressive strength of 28.75 MPa. Although a previous study by Panpa (2002) demonstrated the potential use of FGD gypsum in dental stone production, there has been limited investigation into the different factors that influence the properties of dental materials made from FGD gypsum.

The objective of this research was to undertake a preliminary study of the effect of acid treatment time, particle size, and synthesis method on the physical and mechanical properties of dental materials produced from FGD gypsum. The different sulfuric acid treatment times (15, 30, and 60 min), particle sizes (small for less than 0.1 mm, medium for 0.1-0.35 mm, and large for 0.4-0.45 mm), and synthesis methods (Method A for the synthesis of dental plaster gypsum and Method B for the synthesis of dental stone gypsum) were carried out in the study. The synthesized materials were tested for compressive strength, crystal morphology, and calcium sulfate hemihydrate form. The investigation determined the feasibility and suitability of selecting the size and synthesis method for production. Therefore, other variables, such as setting time and water/powder ratio, were controlled following the ISO standard and kept constant. As there are no reports that confirm the safety of FGD gypsum when it comes into direct contact with humans, there are concerns about the presence of trace toxic elements in FGD

gypsum associated with its direct use. To address this issue, FGD gypsum was utilized as a dental study model for training in dental sciences and for casting a gypsum model after the removal of the impression material from the patient's mouth. Future analysis, after identifying suitable synthesis conditions, other physical properties of setting time, setting expansion (%), flexural strength, etc., will be further examined according to the ISO standard for dental materials before practical applications.

Methods and Materials

Sample preparation of FGD gypsum particle sizes

The FGD gypsum sample was obtained from the Mae Moh power plant in Lampang province. For sample preparation, the FGD gypsum was placed in a tray and dried in an oven at 80 °C for 5 h. The dried FGD gypsum was sieved and separated into three different particle sizes: small (less than 0.1 mm), medium (0.1-0.35 mm), and large (0.4-0.45 mm). The portion weight of FGD as received for small particles was about 55%, for medium particles, 25%, and large particles accounted for approximately 20%.

Method A was used to synthesize dental plaster gypsum. The treated FGD gypsum was placed in a furnace, and a two-stage heating process was employed. The FGD gypsum was first heated from room temperature to 120 °C, and the temperature was gradually increased to 165°C. This process took 2 h and 30 min (modified from Kostic-Pulek et al., 2005; Panpa, 2002). The resultant powder was intended to be used as dental plaster gypsum. Method B was used to synthesize dental stone gypsum. The treated FGD gypsum was autoclaved at 130 °C with a pressure of 1.8 bar for 1 h (modified from Panpa, 2002). The materials were then dried in an oven at 80 °C, resulting in a powder that could be used for dental stone gypsum. The schematic diagram of the research method is shown in Fig. 1.

Determination of the optimal time for sulfuric acid treatment

In this section, the small-sized FGD gypsum was selected for treatment with sulfuric acid at different times to determine the optimal treatment time. The small-sized FGD gypsum was used as it accounted for 55% of the sample and had a high surface area that required a longer treatment time than other sizes. The determination of the optimal time consisted of four steps, as follows:

FGD gypsum treatment with sulfuric acid

A 100-gm sample of the small-sized FGD gypsum was mixed with 100 cm^3 of distilled water and 100 cm^3 of 3 M sulfuric acid at room temperature. The solution was stirred for 15, 30, and 60 min to ensure proper mixing. After the solution was thoroughly mixed, it was filtered and rinsed with distilled water until the pH was within the range of 6.5-6.7. The filtered product was then dried in an oven at 80 °C for 5 h. The final product was the treated FGD gypsum in the form of a fine powder.

Compressive strength test

Method A was chosen for the preliminary step because the preparation process was simple. The water/powder ratio of dental plaster was 0.72. The resulting mixture was poured into a mold with dimensions of 8 mm in diameter and 16 mm in height. After 48 h, the hardened samples were removed from the mold and polished using 600- and 1200-grit silicon carbide paper. Three specimens were prepared for each sample. All specimens were subjected to compression testing using a universal testing machine with a load rate of 5000 N/min until they fractured. The

compressive strength was determined by dividing the applied load at the time of failure by the cross-sectional area of the specimen and expressed in megapascals (MPa).

Selection of the optimal time for sulfuric acid treatment

FGD gypsum, which was treated with sulfuric acid at the optimal time, was selected for further processing based on the results of compressive strength.

Determination of the optimal particle size

Different sizes of FGD gypsum (small, medium, and large) treated with sulfuric acid for the optimal time were calcined using Methods A and B to change gypsum from dihydrate to hemihydrate form.

Testing and characterization

Dental stone made using Method B often has a lower water/powder ratio than dental plaster made using Method A (ISO 6873:2013, 2013; McCabe & Walls, 2008). However, the water/powder ratio for Method B in this study was equal to 0.72, consistent with the ratio used for Method A. This was because the material produced from Method B could not be molded at a lower water/powder ratio due to the mixture being too dry. Then, the resulting mixture was poured into a mold with dimensions of 8 mm diameter and 16 mm height. After 48 h, the hardened samples were removed from the mold and polished using 600- and 1200-grit silicon carbide paper. Three specimens were prepared for each sample.

Mechanical properties test

The cylindrical specimens, each with dimensions of 8 mm diameter and 16 mm height, underwent compressive strength testing using a universal testing machine. The machine was operated in load control mode with a loading rate of 5000 N/min until failure occurred. The force was measured and reported in megapascal (MPa) units. Three replicates were conducted for each sample, and the results were reported using the average value and standard deviation.

Physical properties test

The crystal morphology and surface of the specimens were examined using scanning electron microscopy (SEM). In this study, SEM was selected to investigate the preliminary physical properties resulting from the impact of different particle sizes and synthesis methods on the morphology of gypsum crystals and surface porosity. The objective of the investigation was to determine the feasibility and suitability of selecting the size and synthesis method for production. Therefore, other variables were held constant. For future consideration, once suitable synthesis conditions are obtained, usability and other physical properties, including setting time and setting expansion (%), will be further studied to meet the ISO standard for dental materials.

Determination of calcium sulfate hemihydrate form

The synthesized gypsum powder obtained from Methods A and B was analyzed using X-ray diffraction (XRD) to determine the presence of calcium sulfate hemihydrate (HH) and calcium sulfate dihydrate (DH) phases. The characteristic XRD patterns of HH and DH were identified at 2 theta (2θ) locations. The areas under the crystalline peaks related to HH and DH were integrated. These areas were then compared with the areas obtained from standard samples of HH and DH for both the alpha and beta phases. The percentage of each phase was calculated by dividing the area of each phase peak by the total area of all relevant peaks and multiplying by 100%, as seen in Equation 1.

$$\text{Percentage of phase} = \left(\frac{\text{Area of phase peak}}{\text{Total area of relevant peaks}}\right) \times 100\% \quad (1)$$

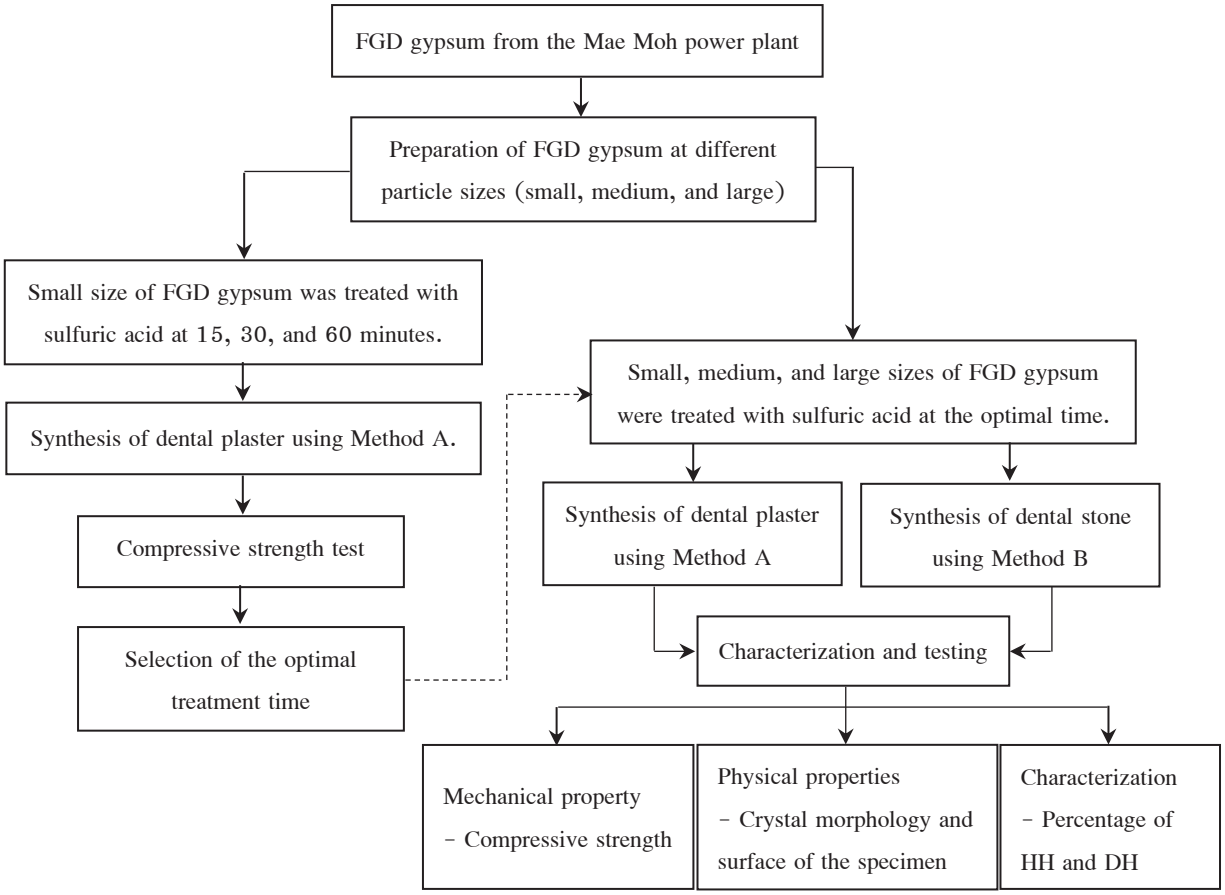
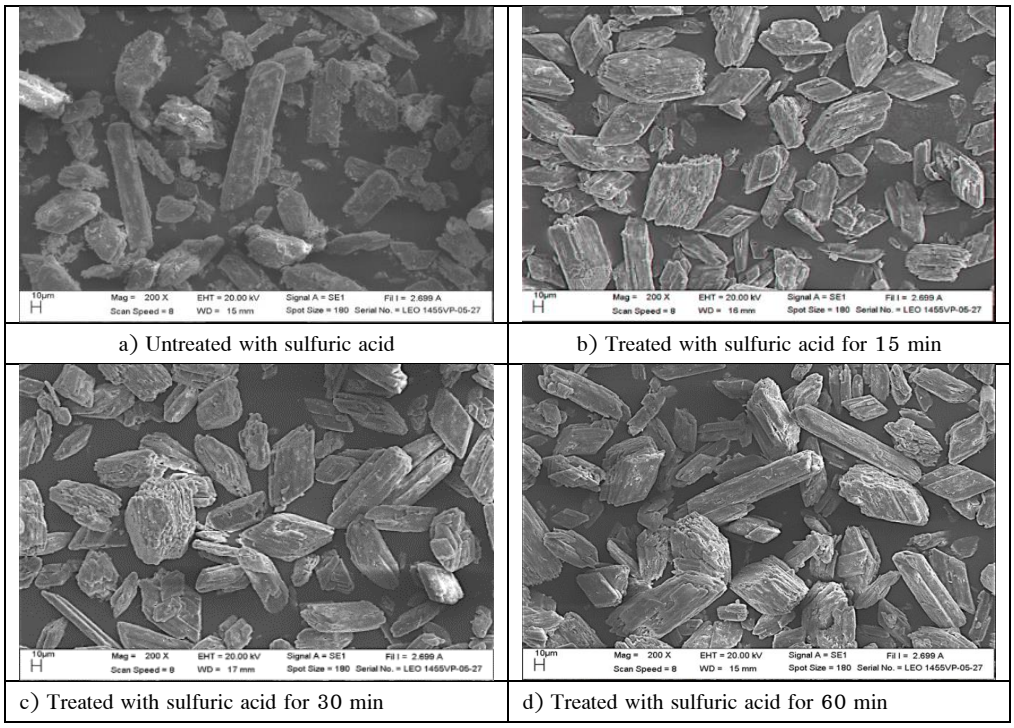


Figure 1 Schematic diagram of the research method

Results and discussions

Effect of sulfuric acid treatment times on the color and morphology of FGD gypsum crystals

It was observed that the colors of the FGD gypsum treated with sulfuric acid for 15, 30, and 60 min were lighter than the untreated FGD gypsum. This could be attributed to the removal of impurities such as silicon dioxide, fly ash, and iron compounds that commonly cause the gypsum to turn brown, so the elimination of these impurities resulted in a lighter and cleaner appearance of the gypsum, as seen in Fig. 2. The results indicate that increasing the treatment time with sulfuric acid resulted in the removal of more impurities from the FGD gypsum crystals. A treatment time of 15 min was determined to be sufficient for effectively cleaning the surface of the FGD gypsum crystals. Furthermore, the crystal structure of the treated FGD gypsum remained unchanged, appearing as a bar shape, and no size alteration was observed compared to untreated FGD gypsum samples.



a) Untreated with sulfuric acid | b) Treated with sulfuric acid for 15 min

c) Treated with sulfuric acid for 30 min | d) Treated with sulfuric acid for 60 min

Figure 2 Morphology of the untreated and treated FGD gypsum crystals

Effect of sulfuric acid treatment times on the compressive strength

The Fig. 3 illustrates the compressive strength results of dental plaster gypsum materials derived from untreated and treated FGD gypsum with sulfuric acid at varying time intervals. The results indicate that treating FGD gypsum with sulfuric acid before synthesizing dental plaster gypsum led to a significant improvement in compressive strength. In this case, the decrease in silicon dioxide content in FGD gypsum could be attributed to the acid treatment, as reported in a previous study by Panpa (2002). However, no XRD analysis of silicon dioxide was conducted in the current research. As the level of silicon oxide increased, the compressive strength of the material decreased (O'Brien, 2022). This occurs because silicon oxide is insoluble in water, which disrupts the interconnection of the growing gypsum crystals during the formation process (O'Brien, 2022). The untreated FGD gypsum exhibited the lowest compressive strength, with a value of 2.40 MPa. Furthermore, there were minor variations observed in the compressive strength values with increasing acid treatment time. As a result of examining the compressive strength and SEM analysis, it was determined that the FGD gypsum treated with sulfuric acid for 15 min was the most suitable for further processing.

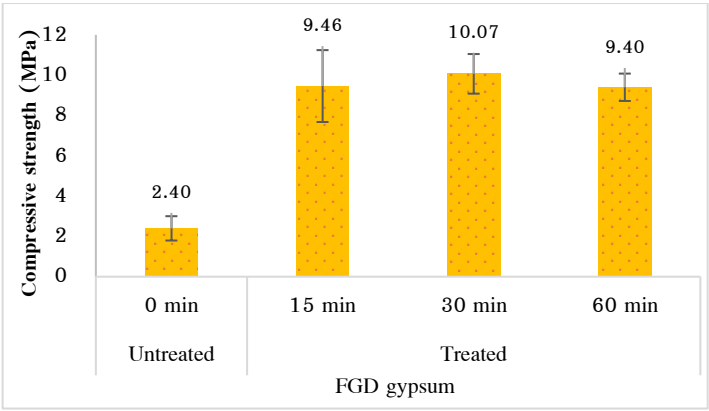


Figure 3 The compressive strength of untreated and treated FGD gypsum with sulfuric acid at different times

Effect of particle sizes on the compressive strength

The results indicate that the particle sizes of FGD gypsum had an impact on the compressive strength of the materials. It was observed that the materials synthesized using small particles through both Methods A and B exhibited higher compressive strength than other particle sizes, as seen in Fig. 4. This was because using smaller particle sizes during the casting process resulted in a denser arrangement with less space between crystals (Fu et al., 2017). In addition, the synthesis methods also affected the percentage of phase change transitions to calcium sulfate hemihydrate (HH), which influenced the compressive strength of materials, as discussed later in the results of the synthesis methods on the calcium sulfate hemihydrate form.

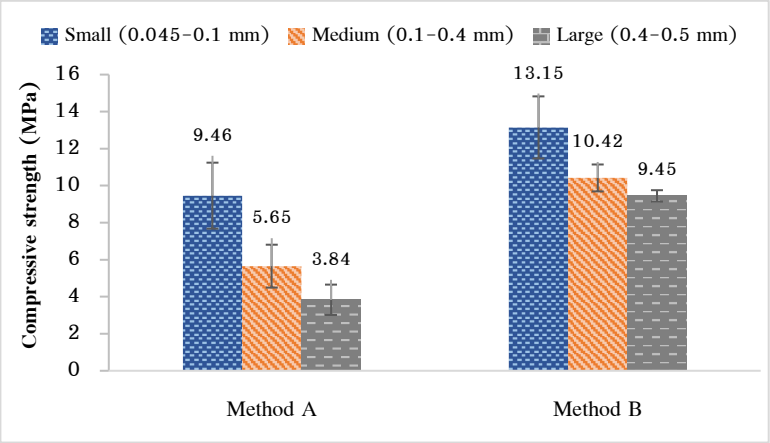


Figure 4 The compressive strength of materials at different particle sizes synthesized using Methods A and B

Effect of particle sizes on the morphology of gypsum crystals

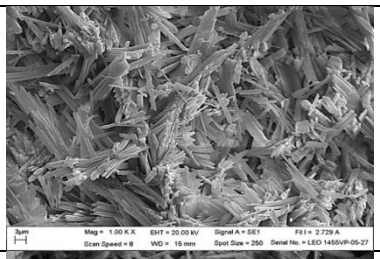
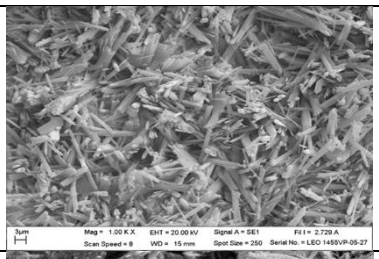
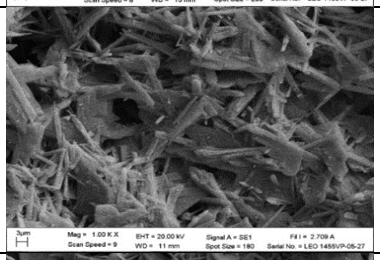
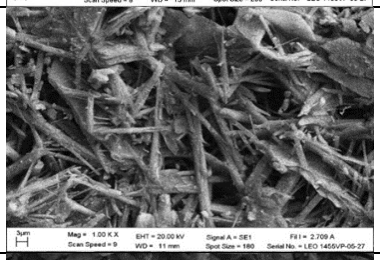
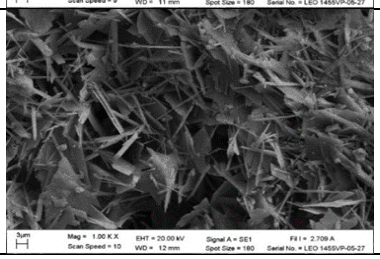
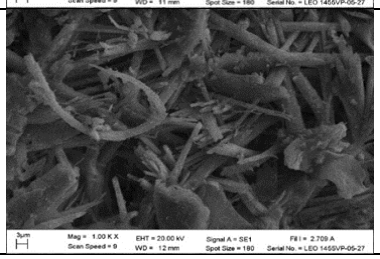
The morphology of gypsum crystals at various particle sizes obtained from synthesis Methods A and B are depicted in the SEM images, as seen in Table 1. The results show that gypsum crystals formed from small particles exhibited greater dispersion and denser arrangement than the larger particle sizes. This is primarily due to the tendency of water to be drawn into the gypsum structure during the casting process, leading to crystal agglomeration. As a result, these crystals were tightly packed, forming a needle-like crystal structure with high density.

Effect of synthesis methods on compressive strength

In this study, unsynthesized FGD gypsum was used as a control sample for comparison with the results obtained from synthesized FGD gypsum. It is important to note that the study did not use a commercial product as a control sample due to a lack of available information regarding the production methods and additives used in the commercial product. These unknown factors could potentially have influenced the properties that we intended to investigate. The commercial products used in dental schools meet the compressive strength requirements of the ISO standard for dental materials, with a minimum of 9 MPa for dental plaster and 20 MPa for dental stone for models (Whip Mix Corporation, 2021). The results showed that the synthesis methods influenced the compressive strength, as seen in Fig. 4. Method B, which applied pressure to the system, compressed the interlayer space in the crystal that contained water molecules. Consequently, the gypsum crystals synthesized by Method B had a higher density and compressive strength than those synthesized by Method A. However, the materials produced by Method B did not meet the standard for dental stones as they had a compressive strength of less than 20 MPa. On the other hand, the materials synthesized using Method A were gypsum plaster, containing DH with irregular particle size and porosity (O'Brien, 2022), resulting in a lower compressive strength. Despite this, the materials produced by Method A still

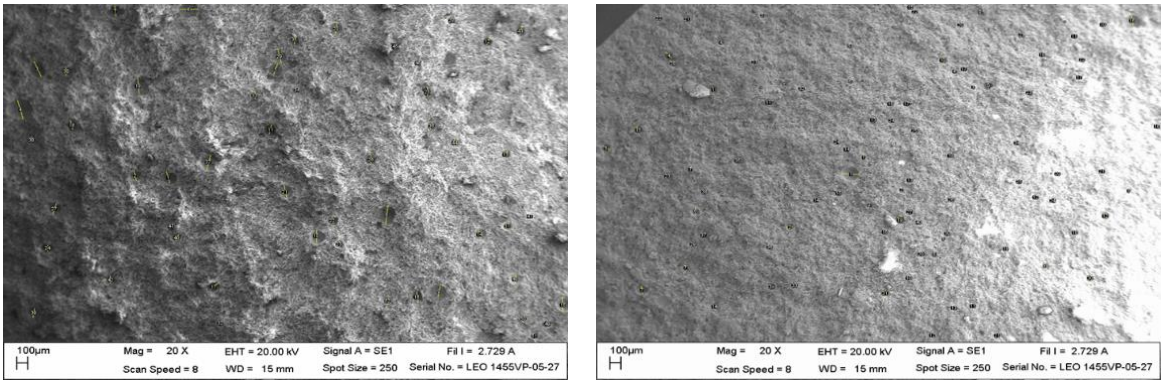
had a compressive strength greater than 9 MPa, which was sufficient for use as dental plaster for impressions and dental plaster for mounting, as they met the standards for dental plaster materials (ISO 6873:2013, 2013; Satameth et al., 2014).

Table 1 The morphology of gypsum crystals at different particle sizes synthesized by Methods A and B

Particle size	Method A: dental plaster	Method B: dental stone
Small (Less than 0.1 mm)		
Medium (0.1–0.35 mm)		
Large (0.4–0.45 mm)		

Effect of synthesis methods on the surface of specimens

The results show that the surface porosity of the specimen synthesized by Method A was larger and the surface density was lower than the specimen synthesized by Method B, as shown in Fig. 5. These surface porosity results corresponded with the compressive strength results, where materials synthesized by Method A had a lower compressive strength than the materials synthesized by Method B. This was because the gypsum crystals synthesized by Method A were large and non-uniform. Furthermore, the crystals were not densely agglomerated, which caused difficulties in connecting with the surrounding crystals. These results created a space between the crystals, resulting in the porosity of the specimens.



(a) (b)

Figure 5 The cross-sectional area of the specimens exhibiting surface porosity synthesized by a) Methods A and b) Method B

Effect of synthesis methods on the calcium sulfate hemihydrate form

Table 2 presents the percentages of calcium sulfate hemihydrate (HH) phases observed via XRD analysis for unsynthesized and synthesized gypsum through Methods A and B. The XRD analysis reveals that synthesis methods and particle sizes significantly affect the phase changes of gypsum. The HH value for unsynthesized gypsum is the lowest among all, at 42.5%. In contrast, the gypsum synthesized using Method A had an HH value of 91.1% and the gypsum synthesized using Method B had an HH value of 100%. The HH content was directly proportional to compressive strength. Accordingly, the gypsum synthesized by Methods A and B exhibited higher compressive strength than the unsynthesized gypsum. Additionally, gypsum synthesized using Method B exhibited the highest compressive strength value because it contained a higher percentage of the α-HH phase, which had a uniform hexagonal shape and was dense (Guan et al., 2021). This contributed to higher strength and surface hardness than the material synthesized using Method A, which contained a mixture of amorphous and crystalline β-HH with non-uniform crystal sizes. In addition, there was no CDS content found in the gypsum synthesized using Method B, while the amount of β-HH remained high at 34%-37%. This may explain why the compressive strength of the gypsum synthesized using Method B was not high enough to meet the standard for dental stone. To address this issue, increasing the pressure in Method B should be taken into consideration in future studies to enhance the compressive strength of the dental stone. It was found that the particle sizes of FGD gypsum also had an impact on the HH phases. Smaller particles had a higher proportion of α-HH phase than larger particles. Although synthesized using the same method, particles of three different sizes showed comparable amounts of HH phases.

Table 2 The percentage of calcium sulfate hemihydrate (HH) and calcium sulfate dihydrate (DH) forms at different synthesis methods

Synthesis method	DH (%)	HH (%)	
		β-HH (%)	α-HH (%)
Unsynthesized gypsum	57.5	14.8	27.7
Synthesized gypsum			
Method A	7.6-8.9	33.5-37.9	53.2-58.9
Particle size:			
Small	7.6	33.5	58.9
Medium	8.2	36.5	55.3
Large	8.9	37.9	53.2
Method B	0	34.1-37.0	63.0-65.9
Particle size:			
Small	0	34.1	65.9
Medium	0	34.8	65.2
Large	0	37.0	63.0

The results of XRD patterns for unsynthesized and synthesized gypsum using Methods A and B with small particle sizes are provided in the Supplementary materials in Fig. 6 – Fig. 11.

Supplementary materials

The effect of acid treatment time, particle size, and synthesis method on the physical and mechanical properties of dental materials produced from flue gas desulfurization (FGD) gypsum

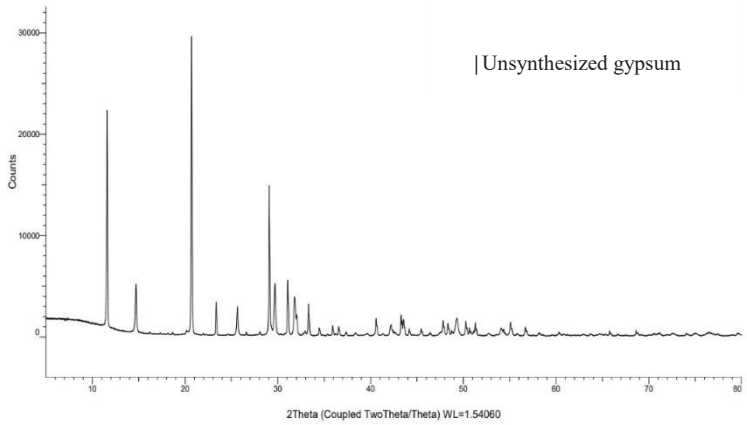


Figure 6 The X-ray diffraction patterns of unsynthesized gypsum

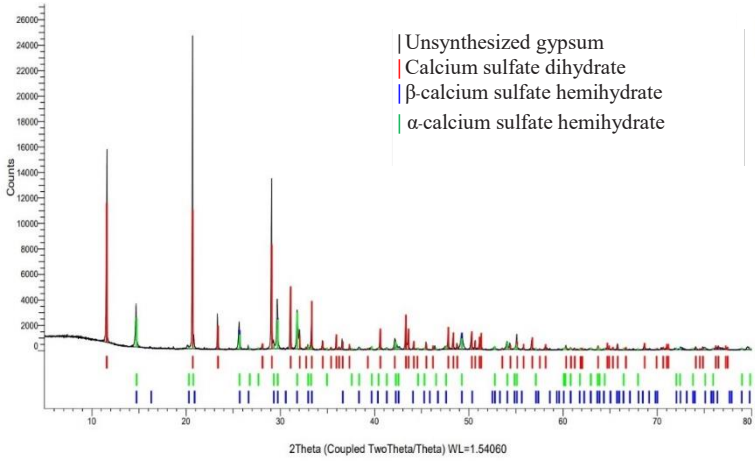


Figure 7 The X-ray diffraction patterns of unsynthesized gypsum, relative to the reference diffraction peaks of calcium sulfate dihydrate, β-calcium sulfate hemihydrate, and α-calcium sulfate hemihydrate phases identified

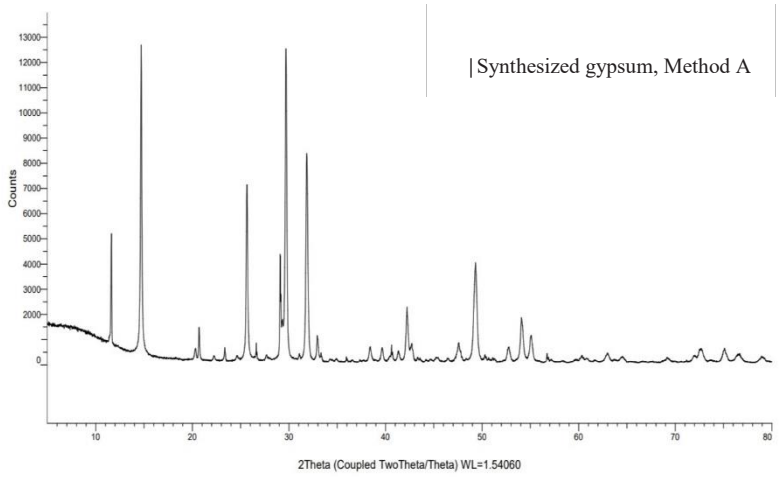


Figure 8 The X-ray diffraction patterns of synthesized gypsum with small particle sizes using Method A

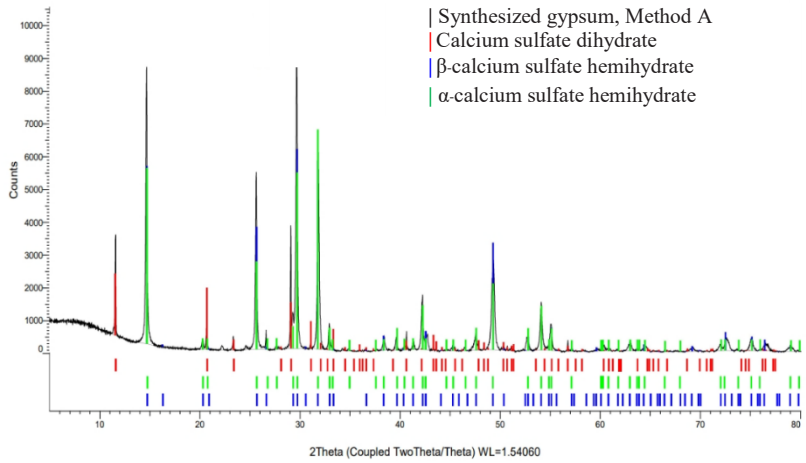


Figure 9 The X-ray diffraction patterns of synthesized gypsum with small particle size using Method A, relative to reference diffraction peaks of calcium sulfate dihydrate, β-calcium sulfate hemihydrate, and α-calcium sulfate hemihydrate phases identified

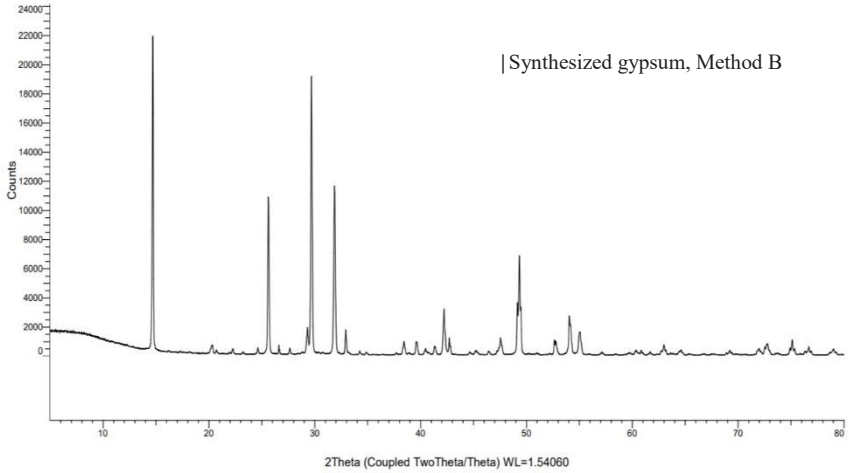


Figure 10 The X-ray diffraction patterns of synthesized gypsum with small particle size using Method B

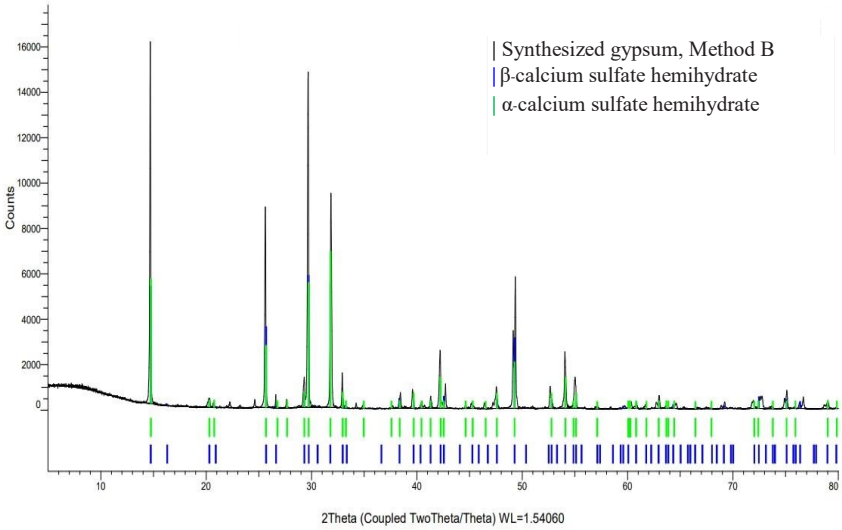


Figure 11 The X-ray diffraction patterns of synthesized gypsum with small particle size using Method B, relative to reference diffraction peaks of β-calcium sulfate hemihydrate, and α-calcium sulfate hemihydrate phases identified

Conclusion and Suggestions

This research investigated the impact of acid treatment times, particle sizes, and synthesis methods on the physical and mechanical properties of dental materials derived from FGD gypsum. The results showed that an acid treatment time of 15 min can be recommended for removing impurities from FGD gypsum. This suitable treatment time also improved the compressive strength of the material. Additionally, the particle sizes and synthesis methods significantly affected the compressive strength, physical properties, and phase changes of the gypsum. The materials synthesized using small-sized particles through both Methods A and B exhibited higher compressive strength and a greater proportion of α-HH phase than larger particle sizes. The gypsum crystals synthesized by Method B had a higher density and compressive strength than those synthesized by Method A due to the application of pressure during the synthesis. Conversely, materials synthesized using Method A contained DH with irregular particle size and porosity, resulting in lower compressive strength.

The findings of this research provide valuable information for improving the physical and mechanical properties of dental materials derived from FGD gypsum. This could help in the development of more effective and durable dental materials, which can improve the quality of dental models. Additionally, the results have implications for the field of materials science, as they provide insights into how different factors, such as acid treatment times, particle sizes, and synthesis methods, can influence the properties of crystalline materials.

The objective of this research was to find suitable conditions for synthesizing materials from FGD with sufficient compressive strength for use in dental applications. However, the synthesized material had a low compressive strength level, classified as types 1 and 2, and was referred to as dental plaster. Even though its application in dentistry has some limitations, it is quite useful for basic training purposes because it is inexpensive and available for making dental plaster for constructing models and dies. This is beneficial for institutions where dental plaster is often used for mounting stone models onto articulators and sometimes for preparing study models, making it a valuable resource for beginner dental training programs.

However, our study had a limitation in that the compressive strength of the gypsum synthesized using Method B did not meet the standard for dental stones classified as type 3. To address this limitation, further studies should consider increasing the pressure in Method B to meet the compressive strength standard required for dental stones. Additionally, further study should be conducted to investigate the optimal properties for both laboratory and clinical use after the completion of testing according to the ISO standard for dental materials.

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Author Contributions

Author 1 (Noppawan Motong): Design research and implement plans, collect and analyze data, summarize research findings, provide recommendations, and write first draft and revised manuscript.

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Conflict of Interests

The authors declare no conflicts of interest.

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