



PREPARATION AND CHARACTERIZATION OF WHITE MINERAL TRIOXIDE AGGREGATE (WMTA) USING SILICA FROM RICE HUSK ASH

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Received: December 15, 2021

Accepted: August 15, 2022

Online Published: August 25, 2022

DOI : 10.20961/jkpk.v7i2.57429

ABSTRACT

The preparation of white mineral trioxide aggregate (WMTA) was carried out using a solid technique using silica (SiO₂) from rice husk ash (RHA). The process included extraction of SiO₂ from RHA, the effect of SiO₂ composition on material characterization and material compressive strength. Silica was extracted from RHA using NaOH and washed with distilled water until it was sodium-free. WMTA was prepared by mixing extracted SiO₂, Bi₂O₃ commercial (18.0%), CaO commercial and Al₂O₃ commercial (2.0%), and the mixture was heated at various temperatures (900, 1000, 1100 °C). MTA was made from two variation silica sources: RHA and extracted silica, and each source varied at 15, 20, and 25% by weight, while the CaO content was adjusted. Characterization was carried out by FT-IR spectrophotometry, XRD and UTM for compressive strength measurement conducted after cementation for a day. The RHA extraction results were obtained from sodium-free SiO₂. The peak showed the success of WMTA preparation at wave number 910 cm⁻¹ in FTIR spectra, indicating the presence of C-S-H and C₃S, C₂S, and C₃A bonds both before and after thermal treatment in XRD data. The highest compressive strength test after cementation for one day (3.21±0.13MPa) was given by WMTA using RHA, heating at 900 °C, and the percentage of CaO and SiO₂ were 60% and 20%, respectively.

Keywords: Rice Husk Ash, SiO₂, WMTA, C₃S, C₂S.

INTRODUCTION

The cavity allowed to continue will widen to the root of the tooth. Therefore, the tooth's root needs to be protected using a material that can cover the hole. Another characteristic of dental materials is that they are anti-bacterial. Therefore, root canal

treatment removes infectious bacteria in the pulp and periapical tissues [1].

One of the ingredients used for root canal treatment is mineral trioxide aggregate (MTA). MTA is a synthetic material which contains silica, bismuth, aluminium and calcium. The composition of MTA consists of 80% Portland cement and 20% bismuth

trioxide to give radiopacity [2]. In addition, MTA has biocompatibility and is an effective dental sealant because MTA is unique because it is derived from more than three oxides, can form composites and aggregates, and is more beneficial than other dental materials. [3].

The main composition of MTA is silica. Silica can be obtained from rice husk ash (RHA) through extraction and directs the addition of RHA into the material. The compositions of rice husk ash are SiO₂ 90.50%, CaO 1.48%, MgO 1.23%, MnO 1.094%, Fe₂O₃ 1.54%, Al₂O₃ 1.21%, and other compounds as much as 2.946% [4]. Rice husk has a high silica content and has various oxides in it so that it can be used for dental materials. Rice husk is amorphous, while its ash has different types of crystals depending on ignition temperature. Experiments using temperatures of 700 °C and 800 °C produce similar amounts of silica between 98.7% and 98.9% [5]. A temperature of 700 °C is the transition stage between the amorphous phase to crystalline; and will produce amorphous silica, based on the research carried out.

The composition for producing WMTA is CaO 65%, SiO₂ 15%, Bi₂O₃ 18%, and Al₂O₃ 2% of the total weight percentage, with Ca as the main composition of WMTA. Calcium hydroxide can react with amorphous silica from rice husk ash, forming calcium silicate hydrate gel [6]. ProRoot MTA contains C₃S, C₃A, C₂S, CaSO₄.2H₂O, and Bi₂O₃ which can be used for pulp and bone [7].

The essential ingredient of MTA is bismuth oxide; The lipophilic bismuth oxide in MTA can be used as an antimicrobial and

antibiofilm to fight inflammation after endodontic treatment [8]. The bismuth particles are on the surface of the White MTA and reduce the setting time, the highest Vickers microhardness hardness test, and the compressive strength similar to that of commercial MTA [9]. Bismuth trioxide is added to the material by about 17-18% to change material properties and radiopacity, which reduces particle size and uniform particle size of material when added to Portland cement [10]. Bismuth oxide will combine the gel phase C-S-H, change the cementation reaction and insoluble, and the reaction is unknown under base conditions [11]. Bismuth oxide is an important key for substituting bismuth into the CSH gel phase, affecting chemical stability, physics duration and material cytotoxicity.

Previous research used commercial materials for synthesis so that they are less natural and do not utilize waste from natural materials and the calcination temperature is up to 1100 °C. This research will directly study the synthesis and characterization of MTA material from silica extracted from rice husk and ash. Husk ash to correct the weakness of less natural MTA. The synthesis uses low-temperature sintering so that it can save energy.

METHODS

1. Preparation silika from RHA

Silica source made from 10 grams RHA was added with 60 mL 2M NaOH, then heated to 85 °C and stirred for 1 hour to obtain a clear solution after filtration, and the residue was re-extracted with 30 mL 2M

NaOH [21]. The filtrate was combined and dripped with concentrated HCl until pH 7 and white sediment were obtained. Then the precipitate was filtered and washed with 450 mL of distilled water. The sediment obtained was dried at 400°C for 4 hours to produce silica (SiO₂). The silica was ground and sieved to a size of 200 mesh. The silica powder was characterized using FTIR, XRF, and XRD. With a wash of 450 mL, Silica does not contain sodium and will be used as a raw material to manufacture WMTA.

2. Preparation of WMTA

a. Procedure

Table 1. Composition of WMTA Comparison of Mass Percent with a mass of Bi₂O₃ and Al(NO₃)₃ 1.8 and 0.735 g (18% dan 2%)

WMTA code	CaCO ₃ (g)	SiO ₂ source (g)	
		Extract {e}	RHA {a}
55Ca25Si/e	9.817	2.715	-
60Ca20Si/e	10.710	2.172	-
65Ca15Si/e	11.607	1.560	-
55Ca25Si/a	9.817	-	2.615
60Ca20Si/a	10.710	-	2.092
65Ca15Si/a	11.607	-	1.569

Based on Table 1 above, 10 grams of WMTA was made by mixing the ratio of CaO: SiO₂ in 200 mL of deionized water stirred for 30 minutes until it became homogeneous. Then the solution was concentrated with HNO₃ until pH 6 and stirred again for 30 minutes. Afterwards, Al(NO₃)₃ was added as a source of Al₂O₃ and stirred for 30 minutes, then added three drops of HNO₃ and pH was still constant six then stirred and heated 85 °C until homogeneous for 30 minutes, added Bi₂O₃ 1.8 gram and stirred for 52 hours at a

temperature of 85 °C until gelation process. Furthermore, it remains stirred at 85°C to form a solid gel or MTA to reach the maturation process. Later, the gel was dried at 120 °C for 24 hours until a yellowish-white powder formed. Finally, DTG / TGA thermal analysis was carried out with a range of 25-900 °C and a heating distance of 10 °C / minute and thermal treatment at 900, 1000 and 1100 °C for 1 hour so that WMTA powder was obtained, then crushed and sifted 200 mesh throughout the powder.

b. Compression test

The specimens were presented in cylindrical form with a diameter of 4 mm and a height of 7 mm. The MTA powder-to-water ratio of 2:1 was selected for mixing. The mixture was transferred to a mould with an internal diameter of 4 mm and a depth of 7 mm. The specimens presented in a cylindrical shape with a diameter of 4 mm and height of 7 mm were carried out for compression test by a universal testing machine (Pearson Panke Equipment Ltd.). The compressive strength of each sample was calculated.

RESULTS AND DISCUSSION

Silica from RHA began to settle at pH 11, or the starting of gel formation. HCl was added drop by drop until the precipitated neutral was obtained, and NaOH reacted with HCl to form NaCl. The white precipitate was SiO₂, which was still NaCl to reduce Na ions; it needed to be washed with distilled water because the ion was hygroscopic and soluble in water. If enough Na ions were contained in silica, it would disturb the material and cause

pores in the application of the material due to its hygroscopic nature. Thus, silica was removed by washing it with distilled water.

Table 2. RHA XRF Test Result and Various Washed Silica (mL)

Component	RHA (%)	270 (%)	360 (%)	450 (%)
SiO ₂	95.6	79.9	81.7	92.1
Na ₂ O	-	10.2	7.9	-
MgO	-	2.3	2.3	-

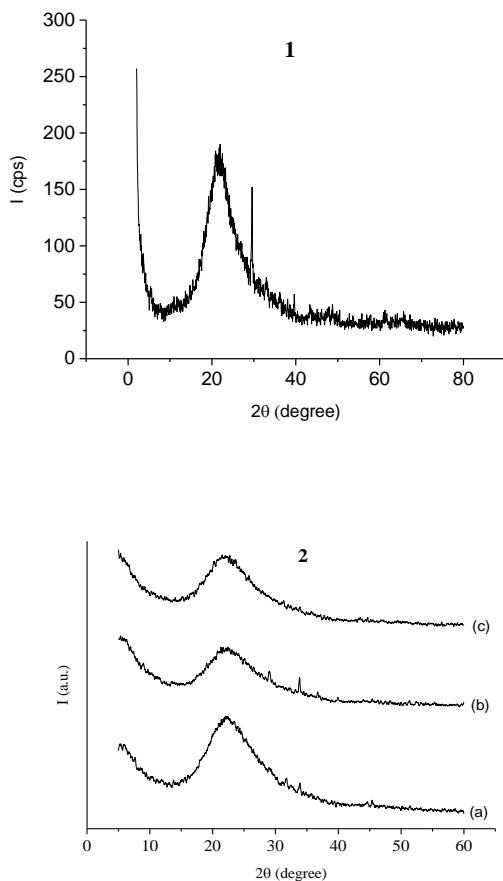


Figure 1. XRD RHA (1) and (2) Silica RHA Washing (a) 270 mL (b) 360 mL (c) 450 mL

The extracted silica phase was determined by the ignition temperature and the character of SiO₂ at an angle of (2θ) 22. There was more. At the same time, the presence of sodium was shown at (2θ) 30. The results of XRD analysis were performed

on either washed or not extracted silica, as seen in Figure 1.

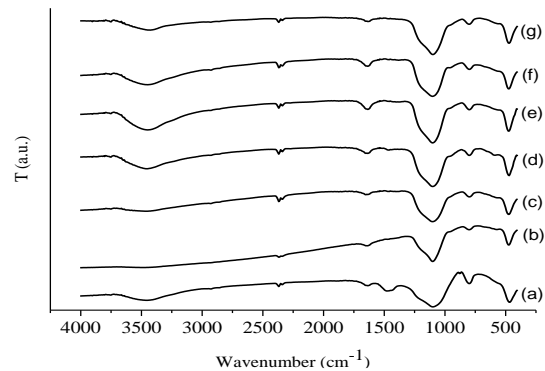


Figure 2. FTIR spectra (a) RHA, washing silica (b) 0 mL, (c) 90 mL, (d) 180 mL, (e) 270 mL, (f) 360 mL, and (g) 450 mL

FTIR patterns in Figure 2 shows that at wave number 470 cm⁻¹ was the bending vibration Si-O-Si≡, 802 cm⁻¹ stretching vibration Si-O symmetry of Si-O-Si≡, 1103 cm⁻¹ stretching vibration Si-O asymmetry of ≡Si-O-Si≡, 1635 cm⁻¹ -OH bending vibration of H₂O, 3448 cm⁻¹ -OH stretch vibration of ≡Si-OH, and 3873 cm⁻¹ was the -OH stretching vibration of H₂O.

DTA/TGA test was used to determine the temperature of thermal treatment or calcination in the manufacture of WMTA, and the shrinkage of sample mass could be seen. The reduction in mass or total weight loss of MTA gel by 4.42% of its original weight or 0.9773 mg by 22.113 mg is aluminium nitrate dehydration. Loss of water from portlandite (calcium hydroxide), and decomposition of calcium carbonate with the formation of portlandite from carbonation (Figure not shown). DTA data was used to determine endothermic and exothermic events. The DTA data above showed that the percentage of endothermic effects, namely enthalpies,

was positive [19] [21]. The calcination started from 900 °C (variations of 900, 1000, and 1100 °C).

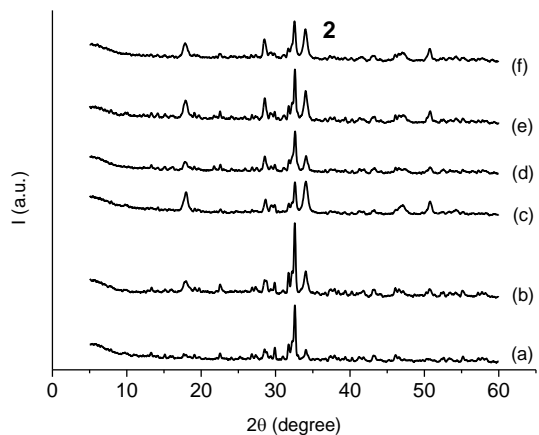
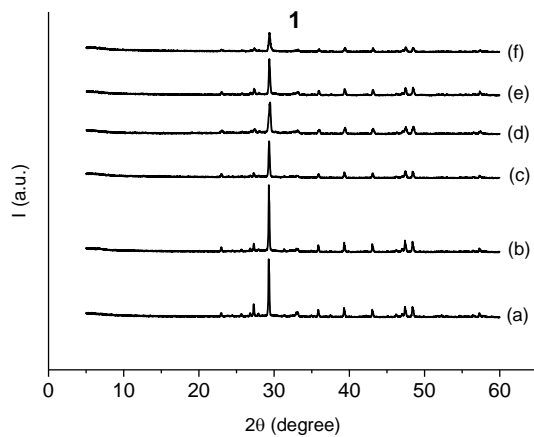


Figure 3. XRD (1) Gel-WMTA (a) 55Ca 25Si/e, (b) 60Ca 20Si/e, (c) 65Ca 15Si/e, (d) 55Ca 25Si/a, (e) 60Ca 20Si/a, (f) 65Ca 15Si/a and (2)WMTA 900 °C (a) 55Ca 25Si/e, (b) 60Ca 20Si/e, (c) 65Ca 15Si/e, (d) 55Ca 25Si/a, (e) 60Ca 20Si/a, (f) 65Ca 15Si/a

Calcination temperature affects the crystallinity of MTA, as seen from XRD 65% CaO, 15% SiO₂ from thermal temperatures ranging from 900, 1000 to 1100°C. XRD test results for various thermal temperatures can be seen in Figure 4. XRD test results for various thermal temperatures can be seen in Figure 4.

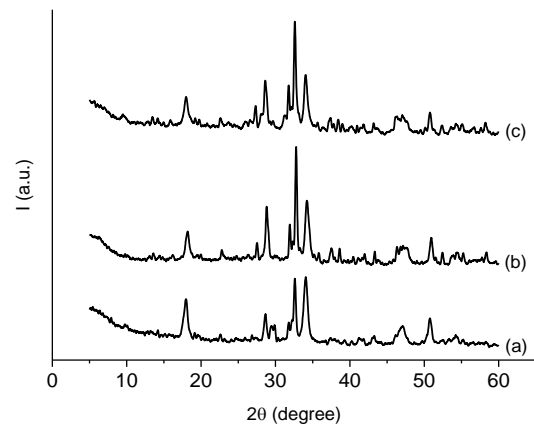


Figure 4. XRD WMTA 65%Ca 15%Si Thermal Temperature (a) 900 °C, (b) 1000 °C, (c) 1100 °C

Before calcination, it could be seen that there was a peak at (2θ) 22.98° and 27.25°, which showed SiO₂ and Bi₂O₃, respectively, with high intensity at 29.34° was CaCO₃ calcite (ICDD 05-0586) and C₃S, 35.87° was the peak of C₃S and C₂S (ICDD 09-0351), 43.04° was Ca nitrate, 47.36° was C₃S and C₂S, and 57.52° was C₃A. After calcination, there was a peak with a small intensity of 22.57°, which indicated the presence of silica, 29.3° was the peak of C₃S, 32.1° was C₃S and C₂S, 34.3° was Ca (OH)₂, C₃S and C₂S, 37.3° was CaO, 41.2° was C₃S and C₂S, 46.1° was C₃A, 47.1° was Ca(OH)₂, 51° was the peak of C₃S (ICDD 31-0301 and JCPDS 42-0551), and 18 and 28.6° were peaks of Ca(OH)₂ portlandite (ICDD 44-1481) formed due to calcination. However, XRD could not detect the C-S-H bond because the crystallinity was very low [23].

The XRD test results in Figure 4 show that C₃S increased with increasing thermal temperature, so crystallinity also increased. For example, at a temperature of 1000 °C, peaks appeared at 27.4°, which showed bismuth oxide that had not appeared

in calcination at 900 °C and 31.889°. Whereas at 1100 °C, peaks appeared at 31.75 and 33.98° and began to appear in bismuth peaks oxides at 27.4 and 28.0° (ICDD 41-1449). Shifting the value 2θ in a larger direction indicates that particles are getting smaller because of d-spacing increases. SEM analysis can prove that calcination will reduce and homogenize particle size [20].

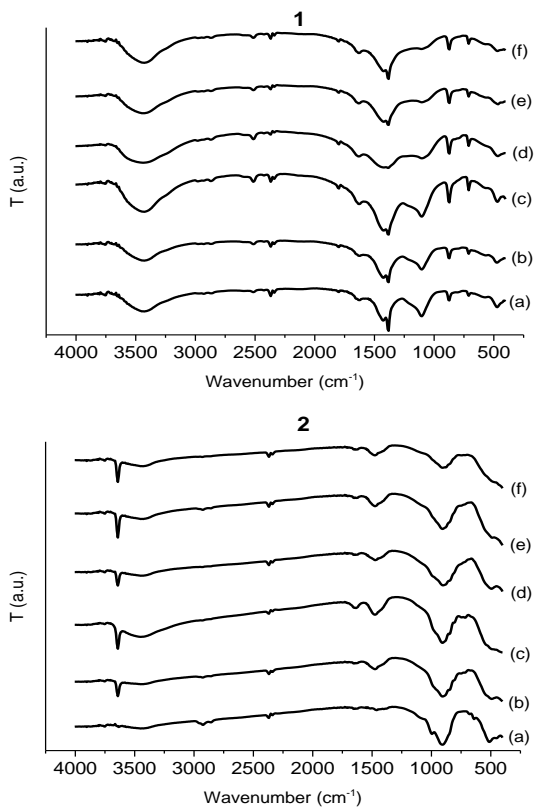


Figure 5. Spectra FTIR (1) Gel-WMTA (a) 55Ca 25Si/e, (b) 60Ca 20Si/e, (c) 65Ca 15Si/e, (d) 55Ca 25Si/a, (e) 60Ca 20Si/a, (f) 65Ca 15Si/a and (2)WMTA 900 °C (a) 55Ca 25Si/e, (b) 60Ca 20Si/e, (c) 65Ca 15Si/e, (d) 55Ca 25Si/a, (e) 60Ca 20Si/a, (f) 65Ca 15Si/a

As a comparison, calcination was carried out in three temperature variations, and FTIR performed the analysis. The results of the FTIR test of WMTA at various temperatures can be seen in [Figure 6](#).

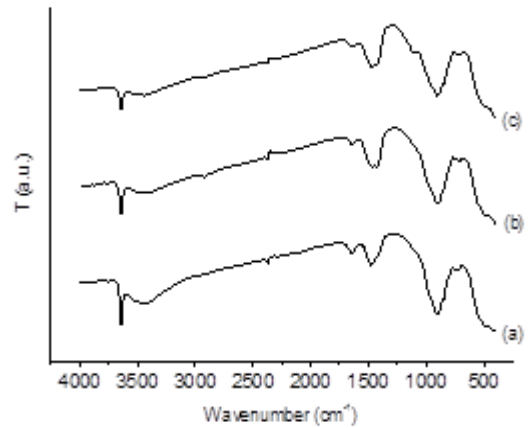


Figure 6. FTIR Test Results for Various Thermal Temperatures (a) 900 °C, (b) 1000 °C, (c) 1100 °C

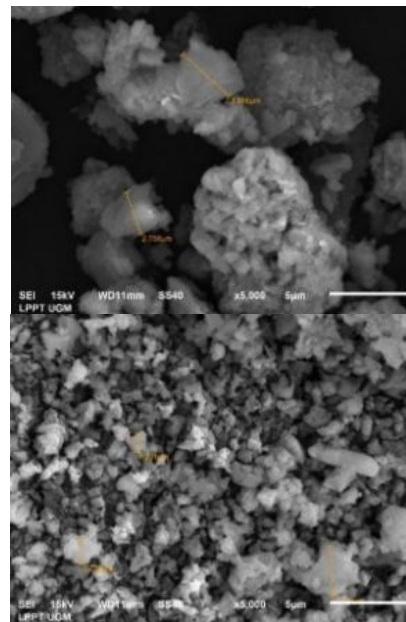


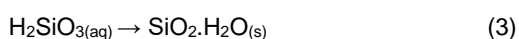
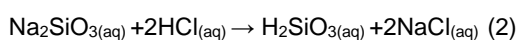
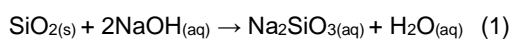
Figure 7. SEM 65% Ca 15% Si (A) MTA gel magnification 5000x, (B) WMTA Thermal 900 °C 5000x magnification.



Figure 8. Radiopacity Characterisation (left) Calcium silicate cement, (middle) WMTA, (right) WMTA Comercial
Table 3. Results of MTA Compressive Strength Ratio WMTA: Water (2:1)

No.	Material	Compressive Strength (MPa)
1	ProRoot MTA	3.31±0.15
2	45Ca 35Si e 900	0.92±0.30
3	50Ca 30Si e 900	1.59±0.38
4	55Ca 25Si e 900	3.21±0.13
5	60Ca 20Si e 900	3.07±0.13
6	65Ca 15Si e 900	3.12±0.42
7	55Ca 25Si a 900	3.49±0.18
8	60Ca 20Si a 900	4.60±0.14
9	65Ca 15Si a 900	1.77±0.09
10	65Ca 15Si e 1000	1.88±0.13
11	65Ca 15Si e 1100	2.52±0.11

XRD could detect this free Na at (2θ) 30° (NaCl JCPDS No. 5-0628). When there was no peak in the area, indicating that there is no Na in silica which may dissolve in washing with distilled water, so the material to be made from silica is not hygroscopic and does not cause pores in the application. About 6-8 grams of silica was obtained from RAH extraction. The reactions that occur in the process of making silica [13] are as follows:



The content of silicon dioxide in RHA was 95.6% showed that the crystalline phase in RHA was amorphous silica. The crystallinity test using XRD. Furthermore, XRF test in Table 2 showed that the extracted silica by washing 450 mL of distilled water did not have sodium. Therefore, sodium will dissolve or disappear by washing using distilled water so that silica by washing 450 can be used as raw material to make WMTA which is unlikely to cause pores in the material when applied and has good biocompatibility.

The more distilled water used in washing, the lower the sodium content and mass of silica produced because the impurities and some metals found in the extracted silica dissolve, increasing the silica content. The way to purify silica or remove sodium from solid silica is to wash it with demineralized water, then dry and obtain silica purity of 98% [14]. Silica crystallinity is influenced by the ignition temperature, where the temperature of 700°C is the transition temperature from the amorphous phase of rice husk to a crystal with a high quartz phase in the form of cristobalite and tridymite.

Diffraction test results showed that silica washed with 270 mL still contained Na peaks at (2θ) 30° , while the silica washed with 360 mL decreased intensity of Na; and silica washed with 450 mL of distilled water, no Na peak was not found, and amorphous silica was produced. It can be concluded that washing with distilled water can reduce and even eliminate Na content. Thus, the silica by washing 450 mL of distilled water can be used as a basic ingredient in making WMTA.

As shown in Figure 2, it was found that the same wave number between RHA and SiO_2 with the washing of 90, 180, 270, 360, and 450 mL of water or not at 438 cm^{-1} was a buckling vibration. Bend $\equiv\text{Si}-\text{O}-\text{Si}\equiv$ (siloxane), about 800 cm^{-1} stretching symmetry Si-O from $\text{Si}-\text{O}-\text{Si}\equiv$, 1103 cm^{-1} stretching vibration Si-O asymmetry from $\text{Si}-\text{O}-\text{Si}\equiv$, 1635 cm^{-1} was the -OH buckling vibration of H_2O , 2337 cm^{-1} was a Si-O bending vibration of $\equiv\text{Si}-\text{O}-\text{Si}\equiv$ which distinguished silica from RHA, 3448 cm^{-1} - OH stretching vibration from Si-OH, and about $3750-3900\text{ cm}^{-1}$ was the -OH stretching

vibration of H₂O [15] [18] [19]. The influence of pH caused gel formation on the mixture, and the presence of CSH (Ca-Si-Hydrate) bonds or between calcium-silicon hydrate catalysed by nitric acid would be maintained at pH 6. The gel could be analyzed using FTIR after thermal treatment, and there was a peak at wave number 885-997cm⁻¹ on FTIR [20].

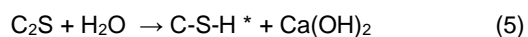
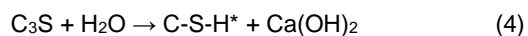
MTA gel still had much oxide due to the presence of hydrated water before thermal treatment and imperfect drying, so the silica content is not comparable. At the same time, aluminium is too small because the source used was aluminium nitrate instead of aluminium oxide. Increased CaO levels were caused by water loss and the formation of a portlandite which then changed to CaO due to calcination. The XRD peak height of tricalcium silicate would increase with increasing thermal treatment temperature and in component or crystallinity [21]. In addition, the calcination temperature affected the presence of silanol and siloxane [22]. The FTIR test could further prove this.

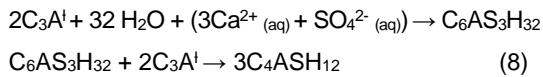
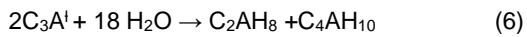
Increasing the calcination temperature caused the intensity at (2θ) 32.1 and 34.3°. The concentrations of C₃S and C₂S also increased. In addition, there was also an increase in intensity at (2θ) 41.2°, namely C₂S and (2θ) 37.3°; CaO proved that calcination increased the CaO concentration given from various compounds such as Ca(OH)₂, CaO, C₂S, and C₃S in MTA material [25]. Before the thermal treatment or dry MTA gel, 1381 cm⁻¹ was a NO₃⁻ vibration, 1427 cm⁻¹ was a stretching vibration of CO₃²⁻, while vibrations of 2368 and 3873 cm⁻¹ were vibrations of

CSH bonds from MTA gel, as seen in Figure 5.

Whereas after the thermal treatment, stronger bonds on Si-O-Ca or CSH at ~ 910-918 cm⁻¹, Si-O-Si buckling vibration at 493 cm⁻¹, 717 cm⁻¹ was the vibration of AlO₄⁻, 1473 and 2924 cm⁻¹ was the stretching vibration of Ca-O, 1635 and 3448 cm⁻¹ was the -OH bending vibration of H₂O, 2337 cm⁻¹ was Si-O bending vibration from Si-O-Si, the vibrations of 2854 cm⁻¹ was Si-O from Si-OH or silanol, vibrations of 2368 and 3873 cm⁻¹ was vibrations of the CSH bond from the MTA gel. After calcination, peaks appeared at 3641 cm⁻¹ Ca-O vibrations from portlandite or calcium hydroxide. In addition, NO₃⁻ and CO₃²⁻ vibrations were eliminated after calcination. Before calcination, there was still much water and other ionic vibrations, such as carbonate and nitrate, so the peak formation of C-S-H bonds (calcium silicate hydrate) could not be observed at 910 cm⁻¹ [10] [20] [23].

While as seen in Figure 6 that an increase in thermal temperature made the spectra at wave numbers less than 500 cm⁻¹ be tilted. In addition, the Ca-O-Si bond, as an indication of C-S-H at 910 cm⁻¹, had increased the intensity, and the Ca-O portlandite bond at 3641 cm⁻¹ also experienced an increase in intensity. Whereas at wave number 2283 cm⁻¹ Si-O vibration from Si-OH and 2854 cm⁻¹ Si-O vibration from Si-O-Si decreased. The Silanol (Si-OH) and siloxane (Si-O-Si) groups will decrease in intensity with increasing calcination temperature [22]. The reaction of cementation or cement hydration is:





Calcium silicate hydrate amorphous hydrogels with a variable composition ratio of Ca: Si and H₂O: SiO₂ tC4AF [(CaO) 4.Al₂O₃.Fe₂O₃], which indicates the C₃A reaction producing C₆(A, F)S₃H₂₂ (C: CaO; S: SiO₂; A: Al₂O₃) [29].

The compressive strength of WMTA was 2.09 until 3.92 MPa, so the calcination variation of WMTA 900 °C qualified as a dental drug problem. Based on the results of the SEM analysis in Figure 7, the calcination minimized and equalized particle size was observed. Moreover, agglomerates lump-shaped particles, disks and a small portion of the cavity or pore in the agglomeration section. The anhydrous portion before thermal was found in the gel structure of the C-S-H (tobermorite gel). An increase in thermal temperature causes the solid phase to interlock better than the solid mass of the MTA.

A high C-S-H bond will result in faster setting time and better cement hydration. Hydration or cementation reactions are exothermic, and low cement porosity will be formed with high thermal temperatures. The incorporation of the C-S-H gel phase is affected by the presence of bismuth oxide. In addition, the function of bismuth oxide is to change cementation reactions and is insoluble and not yet known reactions in alkaline conditions. Furthermore, bismuth oxide is an essential key as a substitute for bismuth in the C-S-H gel phase, affecting chemical stability, physics duration and material cytotoxicity. The particle size of MTA

before calcination was 11.04 µm, but the particle size after calcination was reduced to 1.61 µm or even size less than 1 µm [10] [12] [20].

In addition, SEM analysis showed that smaller particle size changes could be strengthened from XRD shifting to greater 2θ or right after calcination. Uniform particle size also could be known from the peak width of XRD. The narrower the width of the peak, the more uniform particle size is. MTA was affected by a storage temperature of 40 °C, so the leakage or cavities in the material are very small compared to those stored at room temperature. White Mineral Trioxide Aggregate was successfully synthesized with nitric acid catalyst from silica rice husk ash and had comparable characteristics with commercial MTA.

The radiopacity of MTA is influenced by bismuth oxide or substances that affect radiopacity, such as zircon oxide. WMTA, which contained a bismuth oxide, will appear brighter when tested using X-rays than calcium silicate cement without additional bismuth oxide. Based on Figure 8, adding bismuth oxide affected the radiopacity properties. WMTA added by bismuth oxide appeared brighter than calcium silicate without the addition of bismuth oxide. Commercialized MTA and synthesized WMTA had almost the same appearance or bright properties, but calcium silicate cement not added by bismuth oxide appeared darker. This proves that adding bismuth oxide enhances the radiopacity properties of the material. Bismuth oxide can improve radiopacity due to its large molecular weight; Bi₂O₃ added in dicalcium silicate cement is

the best choice for dental root canal treatment material [30].

From this study, adding bismuth oxide reduces the calcination temperature in making MTA. The addition of bismuth oxide, which was added directly to calcium silicate and then calcined, had the best compressive strength at 900 °C. Therefore, the presence of Bismuth oxide will induce the process of forming cement at low temperatures during calcination. In addition, bismuth oxide has a low melting point of around 825 °C, so the material sintering process does not require high temperatures and a lower sintering temperature. As a result, the material fabrication process does not require high temperatures, impacting energy use and processing time and reducing the cost of making materials [31]. Accordingly, bismuth oxide is essential in making WMTA material, calcination, cementation when applied, and radiopacity properties.

Based on the research and characterization, WMTA products were obtained with physical and chemical characteristics per the comparison in commercial MTA. Therefore, WMTA that has been made has the potential to be utilized as a substitute for commercial MTA. First, however, it is necessary to add several tests directly to living things as an ingredient for treating root canals or teeth.

CONCLUSION

White Mineral trioxide aggregate was successfully made from silica rice husk ash at low temperature (900 °C), which was characterized by the presence of C₃S, C₂S, and C₃A peaks in the XRD diffractogram and

the presence of C-S-H peaks in FTIR. The best characterization from MTA in a variation of calcium oxide and silica was 60:20%. Moreover, it was characterized by compressive strength similar to a ProRoot after cementation for one day with the composition of calcium oxide and silicon oxide, which affected the compressive strength. The brightness level of the radiopacity test with X-rays showed similarities with commercial WMTA after adding bismuth oxide. Furthermore, it is necessary to examine its application on teeth as a problem for root canal treatment.

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