

Evaluation of Mechanical Activation and Chemical Synthesis for Particle Size Modification of White Mineral Trioxide Aggregate

 Mohammad Ali SAGHIRI,  Hamed KAZERANI,  Steven M. MORGANO,  James L. GUTMANN

ABSTRACT

Objective: Initial setting time is one of the most important properties of calcium silicate cements (CSCs) such as white mineral trioxide aggregate (WMTA). This study aimed to evaluate the effect of two methods used to reduce the particle size of WMTA, mechanical activation and chemical synthesis.

Methods: WMTA without bismuth oxide (WMTA-B) was provided and divided into four groups (n=5) including: WMTA-B, WMTA-B+10 min milling, WMTA-B+30 min milling, and sol-gel. In groups 2 and 3, the milling was performed by using tungsten carbide balls in a ratio 1:15 (w/w) and a vibration frequency of 30 Hz together with absolute ethanol. For the fourth group, polyethylene glycol (PEG), calcium acetate ($\text{Ca}(\text{C}_2\text{H}_3\text{O}_2)_2$), SiO_2 , and aluminum oxide (Al_2O_3) were used for the sol-gel process. After preparation, sample powders were mixed with distilled water and placed in cylindrical molds, covered with water-moistened gauze, and incubated at 37°C for 24 hours. The Vicat needle test analyzed the initial setting time. Data were analyzed by ANOVA and Tukey tests at a significance level of $P < 0.05$. The correlation between particle size and setting time was determined.

Results: Initial setting time of the sol-gel and WMTA-B+30 min milling was significantly lower than in the other two groups ($P < 0.05$). A significant correlation was noticed between particle size and initial setting time ($P < 0.05$).

Conclusion: Sol-gel process introduces a promising alternative strategy for the reduction of initial setting time of CSC materials. While both methods increased surface area, mechanical activation was not as successful in reducing surface area and initial setting time as effectively as the sol-gel process.

Keywords: Mechanical activation, mineral trioxide aggregate, particle size analysis, setting time, sol-gel, surface area

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HIGHLIGHTS

- There is an inverse relationship between the surface area and the initial setting time. As the surface area of the powder increased, the tendency of the particles to react with water increased, which in turn, resulted in a faster hydration process and reduced of initial setting time.
- Mechanical activation was not successful in reducing the initial setting time.
- The sol-gel method resulted in finer powder with a narrower range of particles and uniform powder particle distribution compared with milling and control groups, which would naturally result in higher compressive strength, higher pH and enhanced calcium release rate.

INTRODUCTION

White mineral trioxide aggregate (WMTA) has advantages when used as root-end filling material, including biocompatibility, sealing ability, and antibacterial properties (1). Some investigations have shown that WMTA has positive cell adherence properties (2), making it a unique material for root canal therapy. Despite such advantages, WMTA suffers from poor handling characteristics and long setting time, which may be attributed to particle size and shape (3-5).

The main constituents of mineral trioxide aggregate (MTA) include Portland cement (75wt%) with

bismuth oxide (20wt%) and gypsum (5wt%). Portland cement is a mixture of tricalcium silicate (C3S), dicalcium silicate (C2S), tricalcium aluminate (C3A), and tetracalcium aluminoferrite (C4AF) (6). WMTA has been reported to have lower concentrations of Al_2O_3 , MgO, and FeO compared with gray MTA (7). Furthermore, the initial setting time and the final setting time of WMTA are approximately 40 minutes and 3 hours, respectively (8, 9). This long setting time is undesirable when WMTA is used as a root-end filling material (1). Attempts have been made to enhance WMTA properties by incorporating some materials into its structure (10). However, these additions may

adversely affect the original material's physical and chemical properties (1). For example, if an additive is added to cement before hydration, it would impact the hydration process and may subsequently have a negative effect on the cement itself.

Studies have reported that particle size and surface area of Portland cement (11), calcium phosphate cements (12) and WMTA (13), play an important role in physicochemical properties, partly because of enhanced hydration with lower porosity (11). Decreasing particle size may offer new physical properties to dental materials (14), resulting in new horizons in dentistry (15). Instead of previous methods of adding chemicals to cement, novel methods in nanotechnology used to reduce particle size can be more effective. Future materials can have an improved initial reaction with water and have a subsequently shorter setting time. Some previous investigations have shown the suitability and practicality of the mechanical activation method by grinding materials to achieve this goal. Gbureck et al. (12) demonstrated that mechanical activation is a new approach for adjusting the reactivity of calcium phosphate cements. The sol-gel method is based on colloid chemistry in which solid raw materials are dissolved in selected solvents under specific conditions, including temperature and pressure, until a homogeneous solution is formed through hydration.

Saghiri et al. (16) introduce a new generation of WMTA named Nano WMTA by applying nano technology and adding different chemical components to the base formulation of WMTA cement. Studies have confirmed that different properties of WMTA, including different physicochemical and biological properties of the cement, had been improved with this nano-modification (17-21).

To date, neither the effect of mechanical activation nor the sol-gel method on particle size, specific surface area, and initial setting time of calcium silicate-based cement has been well documented. Therefore, this study aimed to explore the impact of mechanical activation and sol-gel synthesis on particle size analysis, specific surface area and initial setting time of calcium silicate-based cement.

MATERIALS AND METHODS

Per each test, the cements were divided into four groups of 5 specimens based on their production procedure. WMTA (Tooth-colored Formula, ProRoot MTA; Dentsply, Tulsa, OK) without bismuth oxide (WMTA-B) was ordered and randomly

divided into 3 groups. The first group received no further milling process and was considered as the control group. The second and third groups received 10 mins and 30 mins of milling in a process described below. The fourth group was a cement mixture prepared by the sol-gel process.

Grinding and specimens preparation (mechanically activated)

The specimens in the second and third groups were poured into a Mill MM 400 mixer (Retsch, Germany) with tungsten carbide balls in a 1:15 (w/w) ratio and a vibration frequency of 30 Hz together with absolute ethanol. Subsequently, they were mechanically activated for varying periods: 10 and 30 minutes, followed by drying in an Orbital shaker incubator (Lab-Line, Melrose Park, USA) overnight at 37°C. The remaining group did not receive any mechanical activation. Subsequently, all the specimens in each group were prepared for particle size analysis, specific surface area, and initial setting time measurements.

Chemical synthesis

The sol-gel method is used to synthesize fine particle cements, which have been described previously (22). Briefly, each component of the cement was prepared by using the materials and ratios shown in Table 1. Polyethylene glycol (PEG), calcium acetate ($\text{Ca}(\text{C}_2\text{H}_3\text{O}_2)_2$), SiO_2 , and aluminum oxide (Al_2O_3) were supplied by Sigma-Aldrich Co. (St., Louis, Missouri, USA). Ferrous oxide (Fe_2O_3) was supplied by Fisher Scientific Co. (Fairlawn, New Jersey, USA). Aside from SiO_2 , polydimethylsiloxane (PDMS) was also used in synthesizing the C3S component. This product was supplied by Acros Organics (New Jersey, USA) as a solution with a density of 0.98 g/mL; the molecular weight was specified as 90.200 g/mol. However, this figure may have been overestimated. Also, 1 mL of PDMS, 6.27 g of calcium acetate, and 15 mL of de-ionized water were used.

Each component was synthesized by dissolving the solid particles in water and mixing the solution by hand by using a metal spatula to form a homogenous sol. The sol was then heated on an electrical plate to 100°C for approximately thirty to forty minutes until a paste was formed. Next, the specimens were placed in crucibles and annealed at 1000°C for thirty minutes and then cooled to room temperature enabling the particles to crystallize and form the desired nano-cement components. Finally, the specimens were ground to a fine powder by using a mortar grinder, then placed in glass tubes and sealed with Para Film™ for protection from moisture.

TABLE 1. Raw materials used for sol-gel process

| Material | Molecular weight | C3S Tricalcium silicate | C2A Dicalcium aluminate | C3A Tricalcium aluminate | C4AF Tetracalcium aluminoferrite |
|---|------------------|----------------------------|----------------------------|-----------------------------|-------------------------------------|
| PEG | 3400 g/mol | 4g | 4g | 4g | 4g |
| $\text{Ca}(\text{C}_2\text{H}_3\text{O}_2)_2$ | 158 g/mol | 0.03 mol | 0.02 mol | 0.03 mol | 0.04 mol |
| SiO_2 | variable | 0.01 mol | 0.01 mol | N/A | N/A |
| Al_2O_3 | 101.96 g/mol | N/A | N/A | 0.01 mol | 0.01 mol |
| Fe_2O_3 | 159.7 g/mol | N/A | N/A | N/A | 0.01 mol |
| H_2O | De-ionized | 15 mL | 15 mL | 15 mL | 15 mL |

C3S: Tricalcium silicate, C3A: Tricalcium aluminate, C4AF: Tetracalcium aluminoferrite, PEG: Polyethylene glycol, $\text{Ca}(\text{C}_2\text{H}_3\text{O}_2)_2$: Calcium acetate, Al_2O_3 : Aluminum oxide, Fe_2O_3 : Ferrous oxide

SEM particle size analysis

The powder cement specimens (0.3g) were dispersed in 1 mL of absolute ethanol at 25°C. The specimens were then sprayed on copper tape and set to dry for one hour. Nanoparticle powders were coated with 10 nanometers of gold by using a Bio-Rad gold sputter coating apparatus stage (1 min). Scanning electron microscope (SEM) (model S2500, Hitachi High Technologies America, Pleasanton, CA) photomicrographs were taken and analyzed at $\times 1500$ magnifications. All analyses were carried out at 25 kV. Digital images were recorded by using Microsoft picture manager (Redmond, WA) to standardize each picture at 480×666 pixels. The surface area of powder was calculated with the ImageJ program (Rasband WS, ImageJ; US National Institute of Health, Bethesda, MD) (Fig. 1). Each figure was imported and made binary (Fig. 1a) with this program and the brightness was adjusted based on histograms for all specimens (Fig. 1b); image binary was made for considering the edge detection of particles by the outline in each micrograph (Fig. 1e and 1f). The micrograph was analyzed, and the surface area covered by powder size was measured.

Setting time measurement

This part of the study was similar to that reported by Saghiri et al. (18) and according to ISO Specification #6876:2001. In brief, after mixing each specimen with distilled water in a

3:1 powder/liquid ratio, the specimens were placed in cylindrical molds (height, 2 mm; depth, 10 mm), covered with water-moistened gauze (23), and incubated at 37°C. The specimens' tests started just before their anticipated initial setting time and at 1- or 5-minute intervals until the initial set was achieved. A Vicat needle (Shanghai Cement Test, Shanghai, China) test was carried out by using a 2.0 mm flat-ended needle indenter with a 100 g mass applied at a right angle to the surface of the specimens for 5 seconds. The initial setting time was defined as the amount of time during which the indenter failed to leave a definite mark on the surface of the specimen.

Statistical analysis

Once the data were analyzed and noted to be normally distributed, a One-way ANOVA followed by the post hoc Tukey test was used for statistical analysis at a significance level of $P < 0.05$.

RESULTS

Particle size analysis

The means \pm standard deviations (μm) for four groups were 1.13 ± 0.24 (WMTA-B), 0.73 ± 0.13 (WMTA-B+10 min Milling), 0.55 ± 0.14 (WMTA-B+30 min Milling), 0.29 ± 0.07 (Sol-gel) respectively. The Levene's test showed that there was homo-

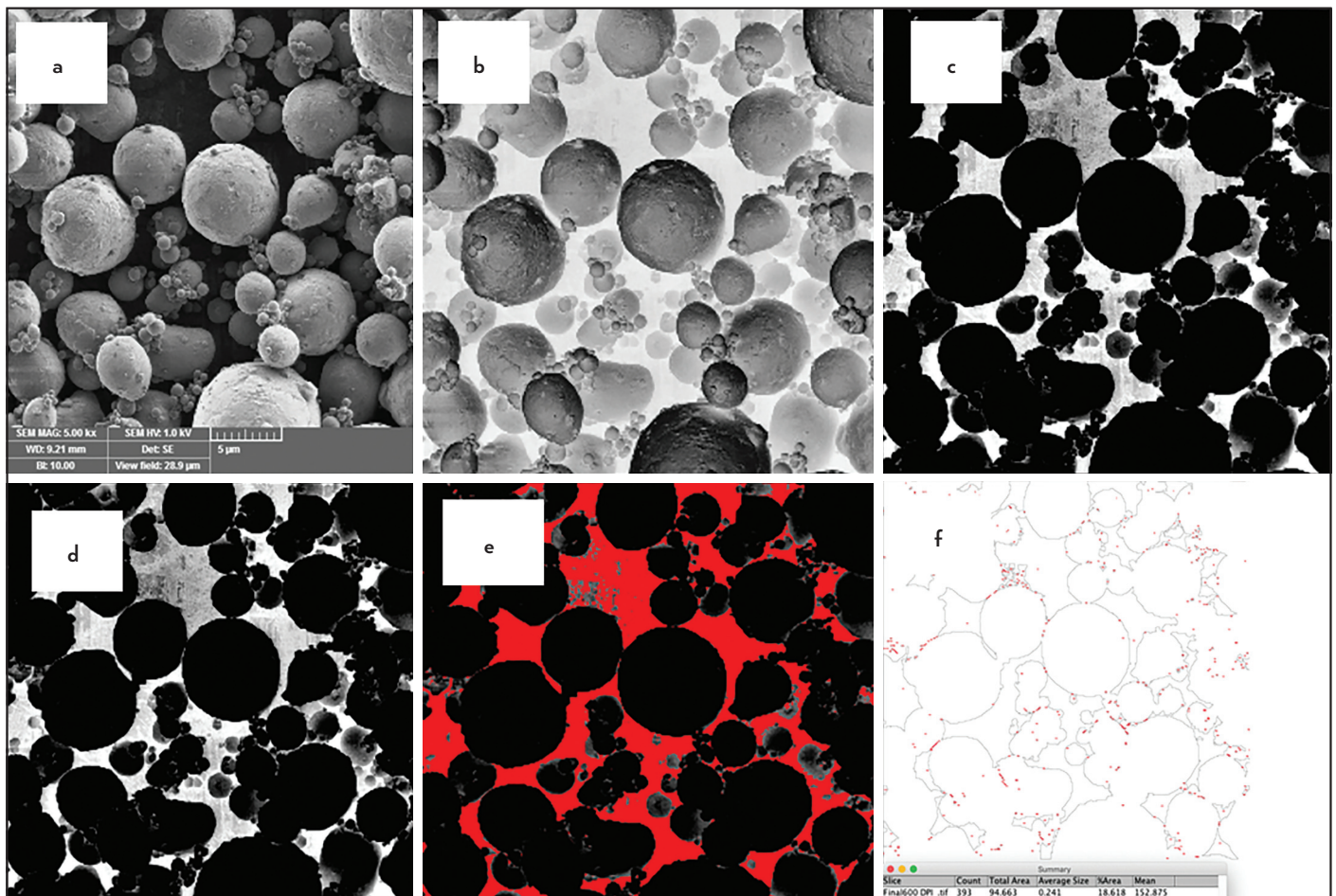


Figure 1. Sequence of image analysis by ImageJ. (a) Micrograph of SEM which is imported to ImageJ, (b) Make it binary and prepare for inversion (b) invert for feasible calculation of covered area (c) Contrast enhancement (d) Convert to 8-bit type for better calculation (e) Adjust threshold for better calculation (f) Calculate the amount of circle or ellipses on the micrograph in regard to the scale

geneity of variances across the groups ($P=0.058$). The One-way ANOVA was used to assess if the effect of the average particle size was significantly different across the 4 groups. The ANOVA results showed significant differences in the effect of average particle size on the groups ($P<0.001$). A Turkey HSD test was, therefore, used to make multiple comparisons of the 4 groups. Results showed that WMTA was statistically different from the other 3 groups ($P<0.05$) (Fig. 2).

Initial setting time

The means±standard deviations for WMTA-B, WMTA-B+10 min milling, WMTA-B+30 min milling, and Sol-gel for the results of the average initial setting time (mins) were 72.00 ± 12.61 , 34.71 ± 4.75 , 23.43 ± 5.71 , 20.00 ± 2.89 , respectively. The Levene’s test showed that no homogeneity of variances across the groups ($P=0.002$). The One-way ANOVA was used to assess if the average initial setting time effect was significantly different across the 4 groups. The ANOVA results revealed significant differences in the initial setting time of each group ($P<0.05$). A Turkey HSD test was, therefore, used to make multiple comparisons of the 4 groups. Results show that WMTA was statistically different from the other 3 groups ($P<0.05$), WMTA+10 min milling was also different from WMTA+30 min milling and Sol-gel ($P<0.05$). However, there was no significant difference between WMTA+30 min milling and Sol-gel ($P=0.825$) (Fig. 2)

Correlation tests (particle size and initial setting time)

The correlation test gave a correlation coefficient ($r=0.808$), indicating a significant correlation between the average particle size and average initial setting time. The result shows that there is a statistically significant relationship between the two variables ($P<0.001$).

DISCUSSION

Mechanical activation with a ball mill, and chemical synthesis by using sol-gel, are introduced as two distinct methods applied for nano modification of materials. While many of the processes have their specific advantages, they also showed deficiencies in some aspects. Thus, in the present study, we aimed to compare the effect of the modification process applied to properties of calcium silicate-based cements.

The effect of bismuth oxide particle size on the physical properties of WMTA has been reported (24), and data regarding the bismuth oxide particle size used in ProRoot MTA was not available. Thus, to eliminate the possible effect of the radio pacifier particle size on measured properties, in the current study, WMTA without bismuth oxide was ordered, and the tests were performed on powders without bismuth oxide.

There is a widely accepted belief among powder and cement researchers that there is an inverse relationship between surface area and initial setting time. In other words, as the surface area of the powder increases, the tendency of the particles to react with water increases, which in turn results in a faster hydration process and reduction of initial setting time. As it was mentioned before, one of the problems of WMTA is its long setting time (9). Taking a glance at Fig. 1, this inverse relationship and faster hydration of powders with finer particle size could be seen. Thus, the use of finer particle size in WMTA shortened its initial setting time and eliminated one of

the properties that were cited to be the main disadvantage, consistent with a previous publication (25).

By comparing data obtained from milling for 10 and 30 mins, 10 mins might be considered the optimal duration for the milling process of the powder. Based on the SEM particle size test, further milling resulted in a significant increase in the surface area. Additionally, there was a slight decrease in the setting time.

ProRoot MTA generally was found to induce fibroblasts to express genes associated with cementum formation of an osteogenic phenotype (alkaline phosphatase, osteonidogen, osteonectin, and osteopontin) (26), which are highly influenced by surface characterization of the powder. Also, the compressive strength of cement increases with fineness, or specific surface area, and that for equal surface area, cements with a narrow particle size distribution have a higher strength than those with a wide size distribution (27, 28). Thus, particle size analysis was used in the present study (Fig.

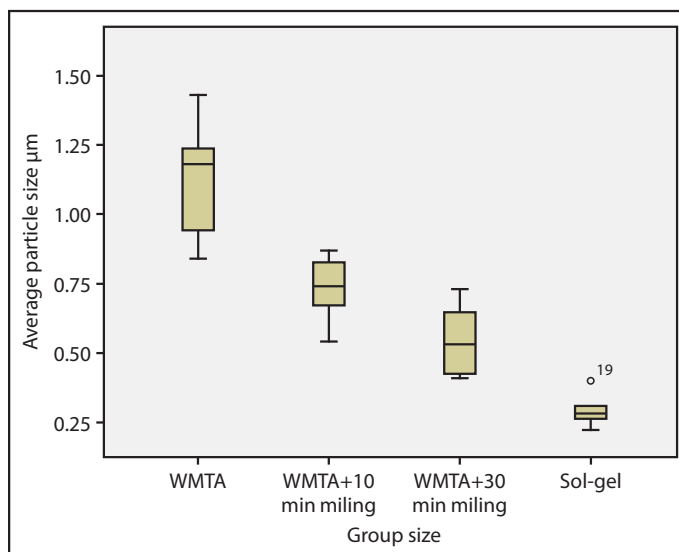


Figure 2. Average initial setting time with SD

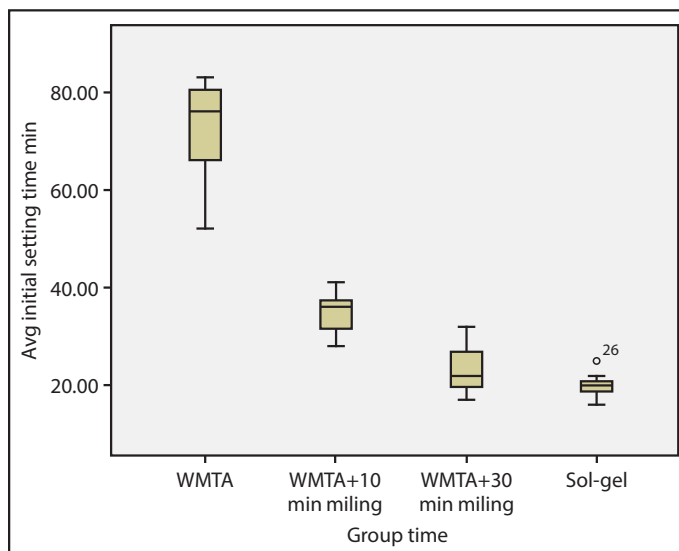


Figure 3. Average particle size area with SD

3). As mentioned, the differences in the particle size of four tested materials are of great importance to the bound cement's mechanical and biological characteristics. The results indicated that the sol-gel method resulted in finer powder with a narrower range of particles and normal powder particle distribution compared with milling and control groups, which will naturally result in higher compressive strength, higher pH, and calcium release rate of the powder as previously claimed (29).

Both modification processes had a significant effect on powder properties; however, based on statistical analysis, the effect of the sol-gel process was significantly higher than milling. The product of the sol-gel method was found to be finer, narrower in range of distribution, and greater in uniform particles than in other groups (Fig. 3).

The handling characteristics of Portland cement is heavily dependent on particle size and shape (5). Additionally, particle modification in ceramics and polymers improves handling characteristics (30, 31). Thus, fine powder with a small range of distribution can be achieved through the sol-gel method. Particle size analysis or measurement is technique sensitive, and most of the time, it needs solvent to measure (32). For measuring dental cement particles, it is difficult to find a solution that dental cement does not react or hydrate with cement. Brunauer-Emmett-Teller (BET) and SEM are routine techniques to measure the particle size (16, 32). BET surface area analysis was used in the U.S. granted patent (16), but it is a costly technique. In the current research, SEM was used to understand the size of particles in 2 dimensions and generally look for a potential agglomeration, and the practicability was confirmed in the previous studies (33, 34).

Porosity can have a significant effect on the powder's physicochemical properties, which is influenced by the manufacturing process (35-37) and will affect final products (38). The sol-gel method results in a purer homogenous powder when compared with the mechanical activation method. Some tungsten impurities will be mixed with the powder to provide a wider range of particle-size distribution, as presented in Fig. 3. Thus, the powder's porosity obtained from the sol-gel method will be lower than the milled powder. Further studies will need to be done to suggest a relationship between porosity and mechanical properties.

CONCLUSION

Within the limitations of this study, both methods tested increased surface area. However, mechanical activation was not as successful in reducing the initial setting time, as the sol-gel process. Moreover, the sol-gel method resulted in finer particles with a narrower range of size distribution.

Disclosures

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