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Research Article

Setting Time of an Experimental Calcium Silicate Based Cement Versus Angelus White MTA and Sinai White Portland Cement

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Abstract

Aim: This study was conducted to evaluate and compare initial and final setting time of an experimental material (laboratory prepared) versus white MTA and white Portland cement.

Materials and Methods: The experimental highly purified calcium silicate based material was synthesized *de novo* in the lab from pure oxides. Specimens of the newly formulated calcium silicate based experimental material with and without hydroxyapatite nanoparticles were prepared by mixing the prepared powder and prepared liquid then measured and compared to the control commercial materials (Sinai white Portland cement and Angelus white MTA). Data were collected, tabulated and statistically analyzed. software.

Results: Within the limitations of this study it was found that there was significant decrease in setting time when calcium chloride solution was used.

Conclusion: Experimental calcium silicate cement with calcium chloride solution is an applicable material regarding setting time.

Keywords: *Calcium Silicate; Angelus White MTA; Sinai White Portland Cement*

Introduction

Calcium silicate based materials are interesting bioceramic products widely used in dentistry. These materials are broadly used in the field of conservative dentistry for regeneration, repair and reconstruction. These are available in different forms and compositions that act directly on vital tissue inducing its healing and repair [1].

Calcium silicate materials derived from the basic building material Portland cement were the first bioactive materials to appear for use in dentistry [2]. Portland cement is a common cement used in civil engineering, the major constituents of ordinary Portland cement are similar to those of MTA, it was reported that their pH, antimicrobial activity, biocompatibility and low resistance to compression are similar [3]. Mineral trioxide aggregate is a biomaterial that is widely used in various conservative treatments due to its excellent biocompatibility, superior sealing and ability to set in the presence of blood [4]. Recently many studies compared MTA with Portland cement and indicated that they have similar chemical composition and biocompatibility; however MTA is quite an expensive material [5-7]. Therefore, Portland cement may be possible substitute for MTA. From the disadvantages of MTA its long setting time and the presence of bismuth oxide which alters its physicochemical properties, so zirconium oxide promotes adequate radiopacity and biocompatibility when associated with Portland cement [8].

Nano-hydroxyapatite (n-HAp) containing products with different formulations have been developed, and early data have suggested remineralizing properties [9]. Calcium silicate cements have a very long setting time which is unaccepted in dental practice, so addition of calcium chloride to the liquid component decreases the setting time eliminating the major drawback of MTA [10].

Materials and Methods

Preparation of experimental calcium silicate based cement

Highly purified calcium silicate based cement was prepared *de novo* in the Physical Properties of Building Materials and Refractories Lab, department of Physics Faculty of Science Al-Azhar University from pure oxides with the same ratio as Sinai white Portland cement (Table 1) by sintering quenching technique and milling procedures.

Raw material	Weight percent
Calcium oxide (CaO)	69.74
Silicon oxide (SiO_2)	25.21
Aluminum oxide (Al_2O_3)	2.58
Iron oxide (Fe_2O_3)	0.21
Magnesium oxide (MgO)	0.24
Sulphate (SO_3)	0.90
Fluoride (F^-)	0.30

Table 1: *Raw material proportioning for Sinai white Portland cement.*

Preparation of experimental calcium silicate white clinker was done from pure oxides powder which was weighted on an analytical balance and mixed in a porcelain mortar then ball milled until uniform mixture was obtained. The mixture was then sintered in an electric melting furnace at 1450^0C for 2 hours in a platinum crucible After discharging from the furnace, the resulted clinker is then quenched by water spray and prevented from coming into contact with air by placing it in a desiccator, crushed in the mortar, then the granules were ball milled in a ball milling machine for 6 hours and sieved in a 0.045 micron mesh sieve in order to be defined as calcium silicate cement powder.

X-ray fluorescence elemental analysis was used to confirm the presence of oxides in the final compound of the calcium silicate prepared powder. Phase composition and microstructure was then characterized with an X-ray diffractometer (XRD) by placing the powder specimen into the sample holder for detection, then to prepare the experimental material 20 weight percent radiopacifing materials (10 weight percent zirconium oxide powder with the hypothesis that zirconium oxide is a radiopaque material at creamy consistency that possess biocompatible characteristics and is indicated as a bioinert material with favorable mechanical properties [11] and 10 weight percent titanium oxide powder with the hypothesis that titanium oxide is a radiopaque material that has antimicrobial effects and increase the biocompatibility of the material [12] respectively) were added to 80 weight percent experimental calcium silicate powder.

Other group (Experimental material + nHAp) was prepared by adding 10 weight percent hydroxyapatite nanoparticles to a sample from the previously prepared experimental material [13]. To prepare aqueous solution of calcium chloride ten percent calcium chloride powder is added to distilled water [14] to decrease the setting time of the cement.

A total of 120 specimens were divided into two main groups (sixty specimens each) according to test procedure done, sixty for initial setting time and sixty for the final setting time. Specimens of the newly formulated calcium silicate based cements were prepared by mixing the prepared powder and liquid [the cement paste was mixed twice (one scoop of powder + one drop of distilled water) and (one scoop of powder + one drop of the prepared CaCl2 solution)] then measured and compared to the control commercial materials (Sinai white Portland cement and MTA Angelus white) regarding setting time. Initial and final setting time of the test materials was determined by using the method recommended by the ISO 9917-1 [15] using the designed Gillmore apparatus (fabricated manually) (Figure 1).

Figure 1: *Gillmore apparatus.*
a- Flat base plate,b- Cylindrical shaft with a horizontal arm, c- Circular mold, d- Gillmore needle for initial setting time
e- Gillmore needle for final setting time, f- Stop watch

The apparatus consists of:

1- A flat plate (base) and a cylindrical shaft which is perpendicular to the long axis of the base through which a horizontal arm with a screw is fixed to adjust the Gillmore needles [16].

2- Gillmore needles for initial and final setting times, these are two vertical arms with different weights with cylindrical flat end needles with different diameters, and one of Gillmore needles with which the initial setting time is measured is 2 mm in diameter and weighting 100 grams load. The second needle is 1 mm in diameter and weighting 400 grams load [17].

3- Stop watch.

Test procedures

Setting time

Freshly mixed cement pastes from each commercial material (Sinai white Portland cement and Angelus white MTA) were formed by mixing one scoop from the ground mineral powder and one drop of distilled water, while in case of (Experimental material and Experimental material + nHAp) the cement paste was mixed twice (one scoop of powder + one drop of distilled water) and (one scoop of powder + one drop of the prepared $CaCl_2$ solution) on a glass plate with stainless steel spatula [18] which then placed in a stainless steel mold measuring 10 mm in diameter and 2 mm in thickness [19]. Ninety seconds after the start of mixing the needle intender was lowered vertically on to the surface of tested cement and allowed to remain there for 5 seconds. After determining the initial setting time measurements continued until the time of the final setting. This procedure was repeated every 5 minutes until the needle failed to make a complete circular indentation (mark) in the tested material. The setting time was calculated by measuring the time elapsed between the start of mixing and the time when no indentation was visible on the cement surface. The test was repeated 10 times for each tested material. Then results were tabulated in excel sheets for statistical analysis and comparison of variables.

Results

Initial setting time

The highest mean value was recorded in (Sinai white Portland cement) group (101.02 ± 13.43), whereas the lowest value was recorded in (Experimental material + $CaCl_2$ solution) group (11.89 ± 1.49). One way analysis of variance (ANOVA) test revealed that the difference was extremely statistically significant ($p < 0.0001$). Tukey's post hoc test revealed no significant difference between (Angelus white MTA) and (Experimental material + distilled water) groups. Moreover, there was no significant difference between (Angelus white MTA) and (Experimental material + nHAp+ distilled water) groups. The difference between (Experimental material + $CaCl_2$ solution) and (Experimental material + nHAp + $CaCl_2$ solution) groups was also non-significant (Table 2 and Figure 2).

Groups	Mean	SD
Sinai white Portland cement	101.02[a]	13.43
Angelus white MTA	31.73[b,c]	12.92
Experimental material + distilled water	41.69[b]	11.26
Experimental material + $CaCl_2$ solution	11.89[d]	1.49
Experimental material + nHAp + distilled water	26.08[c]	3.04
Experimental material + nHAp+ $CaCl_2$ solution	12.99[d]	1.47
P value	< 0.0001*	

Table 2: *Initial setting time (minutes) for all study groups.*

*Significance level p < 0.05, *significant*

Tukey's post hoc test: mean values sharing the same superscript letter are not significantly different

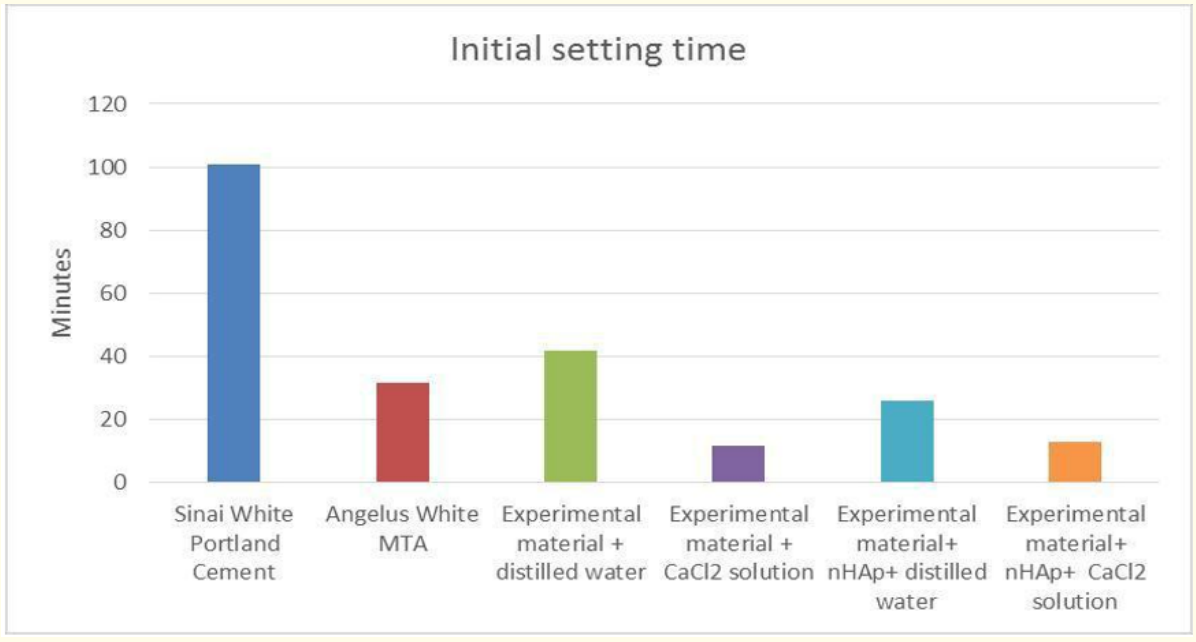

Figure 2: *Bar chart representing mean values of initial setting time (minutes) in different study groups.*

Final setting time

The highest mean value was recorded in (Sinai white Portland cement) group (185.41 ± 12.70), whereas the lowest value was recorded in (Experimental material + $CaCl_2$ solution) group (34.55 ± 4.16). One way analysis of variance (ANOVA) test revealed that the difference was extremely statistically significant (p < 0.0001). Tukey's post hoc test revealed no significant difference between (Experimental material + distilled water) and (Experimental material + nHAp+ distilled water) groups. Moreover, the difference between (Experimental material + $CaCl_2$ solution) and (Experimental material + nHAp + $CaCl_2$ solution) groups was also non-significant (Table 3 and Figure 3).

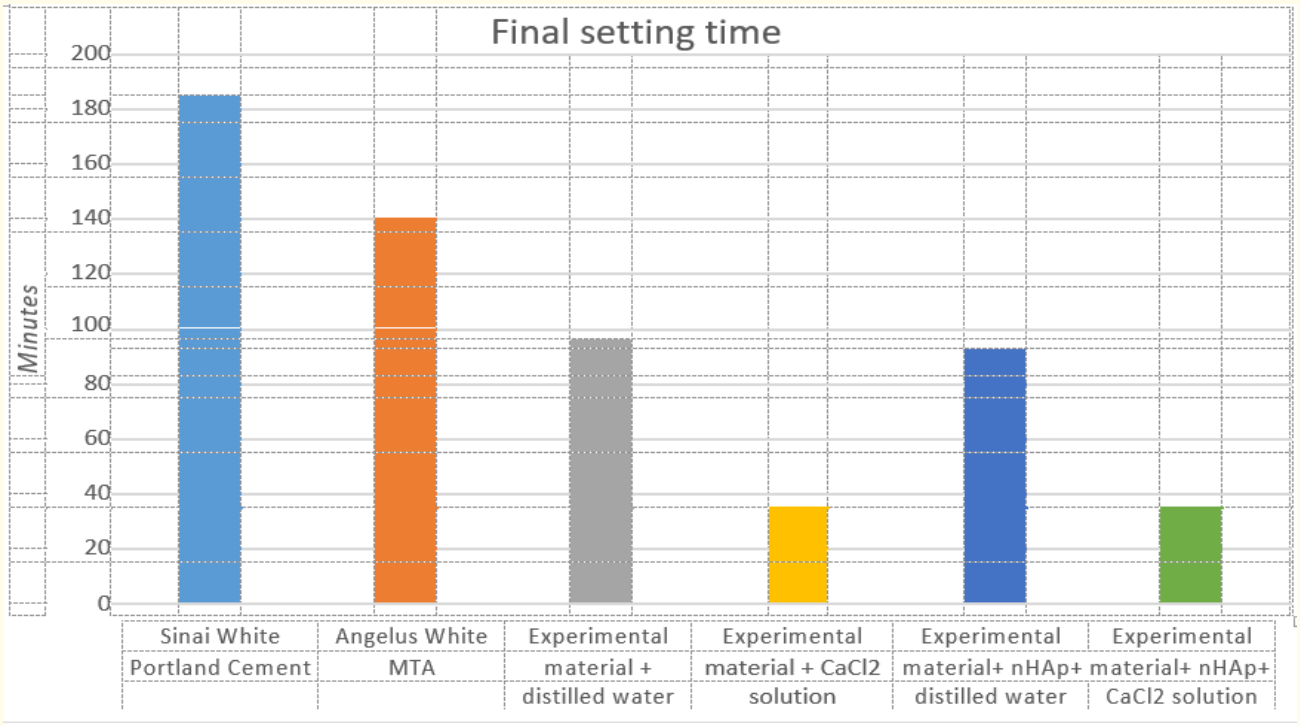

Figure 3: *Bar chart representing mean values of final setting time (minutes) in different study groups.*

Discussion

This study was conducted to evaluate initial and final setting time of an experimental material (laboratory manufactured) versus two bioactive materials (white MTA and white Portland cement). Portland cement which is used in industry as a binder for concrete was introduced as a root- end filling material in dentistry due to its hydraulic nature as it sets and develops its properties in a humid environment [17]. Calcium silicate cements derived from the basic building material Portland cement were the first type bioactive materials appeared in the dental field [2]. International Standardization Organization (ISO) and American Dental Association (ADA) have standardized some technological tests to investigate the physicochemical properties of water based cements. The setting reaction of calcium silicate cements requires water so that they are able to set in a wet and humid environment (in the presence of biological fluids) forming nano-porous calcium silicate hydrate gel, the characteristics of the aggregate depends on standardized conditions of the specimens as particle size, powder to liquid ratio and temperature. Sinai white Portland cement recorded the highest mean value for initial setting time (101.02 ± 13.43) in agreement with a previous study [20] and final setting time (185.41 ± 12.70) with a significant difference with that of Angelus white MTA, this may due to deposition of ettringite crystals resulted from reaction of tricalcium aluminate and gypsum which is responsible for the dormant period of the Portland cement hydration reaction [21]. However, a contrary study [22] reported that the shorted initial and final setting time was observed in PC followed by PC with 2% and 5% gypsum and then MTA in an increasing manner. If gypsum (calcium sulphate dihydrate) was not present, cement would harden immediately upon contact with water. The increase in setting time allows the components of the cement to reorganize to produce a more resistant and durable structure [23].

Gypsum controls the effect of tricalcium aluminate (which starts the setting reaction quickly leading to immediate setting), through the rapid dissolution of gypsum in mixing water leading to dissociation of aluminates from cement powder forming a gel-like layer around the powder particles preventing quick reaction of aluminates and flush setting (instantaneous setting) [14].

On the other hand Angelus MTA material is composed of 80% Portland cement and 20% bismuth oxide with no evidence of calcium sulphate dihydrate (Gypsum) [24]. The lowest initial and final setting time mean values were recorded in (Experimental material + $CaCl_2$ solution) group which are (11.89 ± 1.49) and (34.55 ± 4.16) respectively. This may be due to the effect of zircomiun oxide in comparison to pure experimental Portland cement during final setting time [8], but it was contraindicated with the results of the initial setting time.

In MTA, bismuth replaced silicon in the cement mixture forming a complex microstructure known as calcium silicate bismuth hydrate thus altering the hydration reaction, also bismuth oxide particles may impair the extension of crystallites during the hydration phase and act as a barrier between the calcium silicate cement particles and water during the initial hydration stage increasing setting time [25]. Moreover, zirconium oxide is an inert filler and did not participate in the hydration reaction of the Portland cement [26]. It was reported that incorporation of titanium oxide nanoparticles to white Portland cement results in decrease in its setting time [27] which could explain the shortened setting time of the experimental materials in this study.

Also it was reported that addition of 10% nano-hydroxyapatite particles did not affect the initial setting time but decrease final setting time [13], this fact could be explained by the ability of the added hydroxyapatite nanoparticles to penetrate in the cement porosities accelerating the hydration of silicates and reducing its setting time.

Results of the present study showed that 10% $CaCl_2$ solution accelerated the setting time of the experimental material, this could be due to the penetration of $CaCl_2$ into the pores of the cement leading acceleration of the reaction due to the hydration of silicates which reduces their crystallization time [28]. The accelerative power of $CaCl_2$ may come from its ability to flocculate hydrophilic colloids, such as calcium silicate hydrate facilitating diffusion of ions and water through the initial calcium silicate hydrate layer due to an increased mean pore diameter, and thus allowing a higher rate of hydration and decreasing setting time [29].

Conclusion

Experimental calcium silicate cement with calcium chloride solution is an applicable material regarding setting time.

Bibliography

1. Sonarkar S and Purba R. "Bioactive materials in conservative dentistry". *International Journal of Contemporary Dental and Medical Reviews* (2015).

2. Jefferies S. "Bioactive and biomimetic restorative materials: a comprehensive review part I". *Journal of Esthetic and Restorative Dentistry* 26.1 (2014): 14-26.

3. Silva Neto DJ., *et al*. "Portland cement with additives in the repair of furcation perforations in dogs". *Acta Cirúrgica Brasileira* 27.11 (2012): 809-814.

4. Suzuki Y., *et al*. "Effect of novel fluorapatite-forming calcium phosphate cement with calcium silicate on osteoblasts in comparison with mineral trioxide aggregate". *Journal of Oral Science* 57.1 (2015): 25-30.

5. Hwang Y., *et al*. "Chemical composition, radiopacity, and biocompatibility of Portland cement with bismuth oxide". *Oral Surgery, Oral Medicine, Oral Pathology, Oral Radiology, and Endodontics* 107.3 (2009): e96-e102.

6. Said J., *et al*. "Cell and tissue reactions to mineral trioxide aggregate and Portland cement". *Oral Surgery, Oral Medicine, Oral Pathology, Oral Radiology, and Endodontics* 95 (2003): 483-489.

7. Yoshino P., *et al*. "In vitro cytotoxicity of white MTA, MTA Fillapex and Portland cement on human periodontal ligament fibroblasts". *Brazilian Dental Journal* 24.2 (2013): 111-116.

8. Tanomaru-Filho M., *et al*. "Influence of addition of calcium oxide on physicochemical properties of Portland cement with zirconium or niobium oxide". *Journal of Conservative Dentistry* 18.2 (2015): 105-108.

9. Tschoppe P., *et al*. "Enamel and dentin remineralization by nanohydroxyapatite tooth pastes". *Journal of Dentistry* 39.6 (2011): 430-437.

10. Arora V., *et al*. "Bioactive dentin replacement". *IOSR Journal of Dental and Medical Sciences (IOSR-JDMS)* 12.4 (2013): 51-57.

11. Malkondu O., *et al*. "A review on biodentin, a contemporary dentin replacement and repair material". *BioMed Research International* (2014).

12. Hosseinzade M., *et al*. "Physicochemical properties of MTA, CEM, hydroxyapatite and nano-hydroxyapatite- chitosan cements". *Biomedical Research* 27.2 (2016): 442-448.

13. Guerreiro-Tanomaru JM., *et al*. "Effect of addition of nano-hydroxyapatite on physicochemical and antibiofilm properties of calcium silicate cements". *Journal of Applied Oral Science* 24.3 (2016).

14. Camilleri J., *et al*. "Investigation of the hydration and bioactivity of radiopacified tricalcium silicate cement, Biodentine and MTA Angelus". *Dental Materials* 29.5 (2013): 580-593.

15. International Standards Organization. ISO 9917-1 2007. Dentistry water based cements: powder /liquid based cements 1-22 (2007).

16. Altan H and Tosun G. "The setting mechanism of mineral trioxide aggregate". *Journal of Istanbul University Faculty of Dentistry* 50.1 (2016): 65-72.

17. Guo Y., *et al*. "Physical properties and hydration behavior of a fast-setting bioceramic endodontic material". *BMC Oral Health* 16 (2016): 23.

18. Gandolfi MG., *et al*. "Biomimetic calcium-silicate cement support differentiation of human orofacial mesenchymal stem cells". *Journal of Endodontics* 37.8 (2011): 1102-1108.

19. Gandolfi MG., *et al*. "In vitro screening of the apatite-forming ability, biointeractivity and physical properties of a tricalcium silicate material for endodontics and restorative dentistry". *Dentistry Journal* 1 (2013): 41-60.

20. Dhani R., *et al*. "Comparative study of physicochemical properties of MTA, Portland cements and modified Portland cement as a new endodontic cement". *Journal of Pharmaceutical and Biomedical Sciences* 14.7 (2012): 1-9.

21. Camillieri J. "Characterization and chemical activity of Portland cement and two experimental cements with potential for use in dentistry". *International Endodontic Journal* 41.9 (2008): 791-799.

22. Chakraborty A. "Will Portland cement be a cheaper alternative to mineral trioxide aggregate in clinical use? A comprehensive review of literature". *International Journal of Contemporary Dental and Medical Review* (2015).

23. Goncalves JL., *et al*. "Evaluation of physicochemical properties of Portland cement and MTA". *Brazilian Oral Research* 24.3 (2010): 277-283.

24. Salehimehr G, *et al*. "Comparison of physical and chemical properties of Angelus MTA and new endodontic restorative material". *Journal of Applied Pharmaceutical Science* 4.7 (2014): 105-109.

25. Ha W., *et al*. "Methodologies of measuring the setting times of mineral trioxide aggregate and Portland cement products used in dentistry". *Acta Biomaterialia Odontologica Scandinavica* 2.1 (2016): 25-30.

26. Camillieri J., *et al*. "Hydration characteristics of zirconium oxide replaced Portland cement for use as a root-end filling material". *Dental Materials* 27.8 (2011): 845-854.

27. Samiei M., *et al*. "Effect of the TiO2 nanoparticles on the selected physical properties of mineral trioxide aggregate". *Journal of Clinical and Experimental Dentistry* 9.2 (2017): e191-e195.

28. Arunagiri D., *et al*. "A comparative evaluation of the effect of various additives on selected physical properties of white mineral trioxide aggregate". *Journal of Conservative Dentistry* 18.3 (2015): 237-241.

29. Saghiri M., *et al*. "Calcium silicate based cements and functional impacts of various constituents". *Dental Materials Journal* 36.1 (2017): 8-18.

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