



Chemical and Physical Properties of Local made Tricalcium Silicate Cement for Regenerative Endodontics

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Abstract

This study aimed to investigate chemical compositions and physical properties of local-made laboratory-synthesized fast setting tricalcium silicate cement for dental application. The laboratory-synthesized tricalcium silicate (TCS) powder was prepared by sintering silica (SiO_2) and calcium carbonate (CaCO_3) at 1450°C for 2 hours. The chemical composition of the TCS powder was analyzed by X-ray diffraction analysis (XRD). To develop fast setting TCS cement, zircon (ZrSiO_4) at the amount of 10%, 20%, and 30% was added to TCS as a radiopacifier. Calcium chloride (CaCl_2) at the concentration of 20% was used as an accelerator. Physical properties of the prepared materials were investigated accordingly to ISO 6876:2012 and 9917-1:2007 including initial and final setting time, compressive strength, radiopacity, and pH. XRD showed that tricalcium silicate was the main component of the TCS powder and Biodentine. TCS cement with a higher concentration of zircon exhibited a higher setting time. At the concentration of 20% zircon, the setting time of TCS cement was not significantly different from Biodentine. Adding zircon at a concentration of more than 10% resulted in higher compressive strength similar to Biodentine. Varying the amount of zircon to TCS did not affect pH values. At the duration of 3 hours to 3 weeks, TCS cement showed higher pH values in comparison to Biodentine (in the range of 12.1 to 12.8). TCS with 20% and 30% zircon showed higher radiopacity than Biodentine (5.64 and 6.47 mm of aluminum). In comparison to Biodentine, the local-made laboratory-synthesized TCS cement with 20% zircon showed suitable physical properties for dental application.

Keywords: tricalcium silicate, Biodentine, dental material, physical properties, endodontic

1. Introduction

The development of a suitable material is the key to the success of regenerative endodontic treatment. Calcium silicate-based cement is widely used for pulpal regeneration and hard tissue repair, including pulp capping, pulpotomy, apexification, apexogenesis, retrograde filling, and perforation repair (Parirokh & Torabinejad, 2010). The first generation of calcium silicate-based cement is Mineral trioxide aggregate (MTA; Pro Root MTA, Dentsply Tulsa Dental, Tulsa, OK, USA) (Torabinejad et al., 1995). MTA has suitable biocompatibility, sealing ability, and physiochemical (Ma, Shen, Stojicic, & Haapasalo, 2011; Sarkar, Caicedo, Ritwik, Moiseyeva, & Kawashima, 2005; Wang, Chang, & Hu, 2012). However, the main disadvantage of MTA is too long setting time to complete treatment in one visit. MTA can induce tooth discoloration by its iron content (Camilleri et al., 2005). The Portland cement component in MTA may contain some heavy metal contamination such as arsenic, chromium, and lead (Camilleri & Pitt Ford, 2006). MTA compose of purified Portland cement with bismuth oxide (Bi_2O_3) as a radiopacifier (Camilleri, 2008b). The main component of MTA is tricalcium silicate, which is the main constituent of Portland cement (Camilleri, 2008a).

Instead of using Portland cement, bioactive cement based on pure tricalcium silicate has been developed to avoid impurities and heavy metal in raw materials. Pure tricalcium silicate can be a replacement for Portland cement in MTA due to similar bioactivity (Chen, Ho, David Chen, & Ding, 2009). Tricalcium silicate can induce the proliferation and differentiation of human dental pulp cells in vitro (Peng et al., 2011). Calcium chloride is added into tricalcium silicate cement to accelerate setting time. Tricalcium silicate cement with 5-15% calcium chloride showed good setting properties and biocompatibility (Wang, Sun, & Chang, 2008). Zirconium oxide is added into tricalcium silicate to achieve preferable radiopacity. Tricalcium silicate



cement adding up to 50 wt% zirconium oxide revealed non-cytotoxic to dental pulp fibroblasts (Li et al., 2017) and induced reparative dentinogenesis in minipig pulps (Li et al., 2018). Tricalcium silicate cement with 20% zirconium oxide had preferable radiopacity and allowed the deposition of hydroxyapatite on the cement surface (Camilleri, Sorrentino, & Damidot, 2013).

Biodentine (Septodont, Saint Maur des Fosses, France) is the later calcium silicate-based cement commercial product for dentin substitution. Biodentine powder mainly contains tricalcium silicate with zirconium oxide serves as a radiopacifier and calcium chloride as an accelerator (Camilleri et al., 2013). Biodentine was claimed that setting time was 12 minutes and handling was easier than MTA (Dawood, Parashos, Wong, Reynolds, & Manton, 2017; Septodont, 2010). Biodentine has acceptable physicochemical and biological properties (Dawood et al., 2017). It can be applied for indirect pulp capping, direct pulp capping, cementum repair, and external root resorption (De Rossi et al., 2014; Strassler & Levin, 2012). However, the radiopacity of Biodentine is similar to dentin so the cement is not visible in the radiograph (Kaup, Schafer, & Dammaschke, 2015).

Tricalcium silicate can be synthesized by several methods. The solid-state reaction is one of the methods that is less complicate and requires cheaper raw materials. Briefly, tricalcium silicate can be produced by sintering silica (SiO_2) and calcium carbonate (CaCO_3) at 1450°C . After sintering, the product is processed with rapid cooling, and grinding into powder (Xiaohai Liu, 2020).

2. Objectives

- 1) To synthesize tricalcium silicate cement using calcium carbonate and silica as raw materials with calcined zircon (zirconium silicate) and calcium chloride as additives
- 2) To characterize the chemical composition of the product powder X-ray diffraction analysis (XRD) and compared with Biodentine
- 3) To investigate the physical properties including setting time, compressive strength, radiopacity, and pH and compared with Biodentine

3. Materials and Methods

3.1 Tricalcium silicate cement preparation

Tricalcium silicate cement was prepared from calcium carbonate (99.5%, Ajax Finechem, New Zealand) and silica (99.8%, Ajax Finechem, New Zealand) as shown in Figure 1. Calcium carbonate and silica were mixed with a stoichiometric ratio of tricalcium silicate using a zirconia planetary ball mill (Fritsch, Pulverisette 6) with a rotation speed of 500 rpm for 30 min. Distilled water was used as the milling medium. The mixture was dried using a rotary evaporator. The prepared mixed raw material powder was uniaxial hydraulic pressed at 10 MPa to be 35 mm diameter with 10 mm thick pellet. Next, Pellet samples were placed in an alumina crucible and fired at 1450°C for 2 hours with a heating rate of $10^\circ\text{C}/\text{min}$ using a high-temperature furnace (Heraeus, k1700). After firing, the samples were rapidly cooled down by a blower fan. Fired samples were crushed in an alumina mortar. The powder of tricalcium silicate powder was mixed with 10, 20, 30 wt% calcined zircon powder as a radiopacifier. TCS cement powder was milled using a zirconia planetary mill with a grinding speed of 500 rpm for 60 minutes. Ethanol was used as a grinding medium. The slurries were dried in an oven at 80°C overnight and were then ground with an alumina mortar.

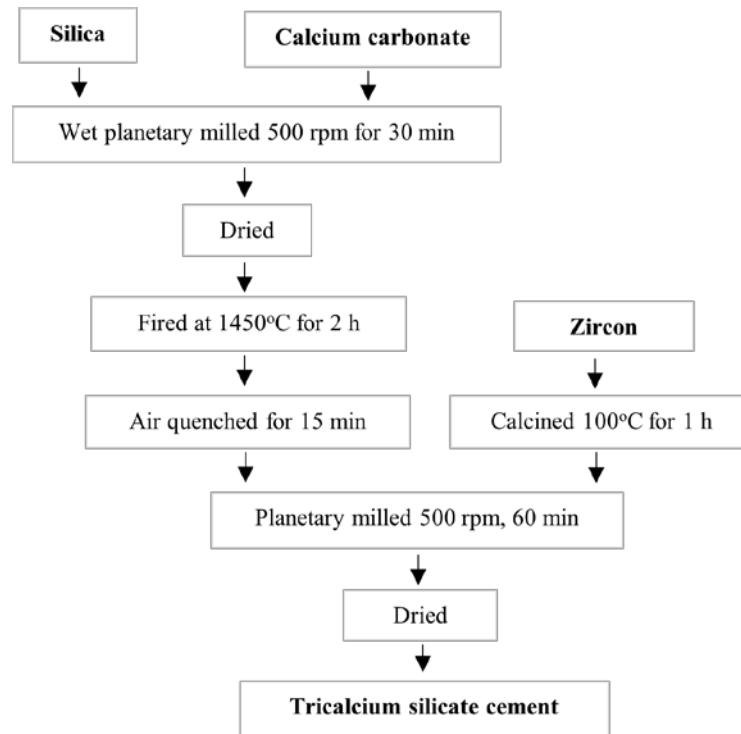


Figure 1 Flow chart of TCS cement preparation

3.2 Chemical composition and particle size distribution

After the preparation of TCS cement powder, the chemical composition of powders of tricalcium silicate cement was characterized. Chemical phase analysis was analyzed by XRD (X-ray diffractometer, Bruker AXS Model D8 Discover, Karlsruhe, Germany). The diffractometer used the radiation at 40 mA and 40 kV. The detector rotated between the angle of 5°-70°, 0.02 degree/step, and 240.5 sec/step was used. Particle size distribution was characterized by dynamic laser scattering particle size analyzer (Mastersizer 2000, Malvern, UK)

3.3 Physical properties testing

For physical properties testing, tricalcium silicate powder was mixed with 20 wt% calcium chloride solution (power: solution weight ratio of 1: 0.3) by capsule mixer for 30 seconds. Biodentine (Septodont, SaintMaur-des-FossésCedex, France) was mixed according to the manufacturer's instructions.

3.3.1 Setting time

Setting time was evaluated based on ISO 6876:2012 and 9917-1:2007. Three test specimens for each group were mixed and placed into a metal block with internal dimensions of 5 x 8 x 10 mm. A Gilmore needle with a tip of diameter 2±0.1 mm having the weight of 100±0.5 g and 400±0.5 g was used to determine the initial setting time and final setting time respectively. Gilmore needle was lowered vertically onto the surface of the specimen for 5 seconds. The process was repeated at a 30-second interval. Recorded the time elapsing from the end of mixing and the time when the needle failed to make a complete circular indentation on the specimens. The materials were kept in a cabinet at 37±1°C and 100% humidity throughout the analysis.

3.3.2 Compressive strength

A compressive strength test was performed based on ISO 9917-1:2007. The metal mold with an internal dimension of diameter 6±0.1 mm high and diameter of 4±0.1 mm was used to fabricate 20 specimens of each material. The specimens were kept at 37±1°C and 100% humidity for 24 hours. Each specimen was



placed with the flat end between the platens of the Universal testing machine (LLOYD, Ametek, Pennsylvania, USA). The compressive load was applied along the long axis of the specimen. The maximum force applied when the specimen fracture was recorded. The compressive strength was calculated using the following equation:

Compressive strength = Force at failure / Cross-sectional area of the specimen

3.3.3 Radiopacity

Radiopacity evaluation was performed using ISO 6876 recommendations. For sample preparation, metal mold with an internal diameter of 10 ± 0.1 mm and a height of 1 ± 0.1 mm was used. Three specimens per group were produced and kept at $37\pm 1^\circ\text{C}$ and 100% humidity for 24 hours. Each sample was placed together with an aluminum step wedge on a size 2 CS7600 digital imaging plate (Carestream Dental, NY, USA). Radiographs were taken by X-Mind DC (Acteon Group, Norwich, United Kingdom) with 70 kV, 8 mA, and target film distance of 40 mm. The average gray pixel value of the specimens and the aluminum step wedge were determined using the Dental Imaging Software CS (Acteon Group, Norwich, United Kingdom). Equivalent radiopacity expressed in mm of aluminum (mm Al) of each specimen was then calculated.

3.3.4 pH

The pH values of each specimen were evaluated following the method of Camilleri, 2011. Seven specimens per material were produced with polysiloxane mold with a dimension of 10 ± 0.1 mm diameter and 1 ± 0.1 mm high. The individual specimen was immersed upright in 10 ml. of distilled water. Each container was sealed with plastic wrap throughout the experiment. The pH of each container was measured at 3 hours, 6 hours, 24 hours, 48 hours, 1 week, 2 weeks, 3 weeks, 4 weeks, 6 weeks, and 8 weeks. The pH meter (CLEAN L'EAU, Kunling Instruments and Equipment Co., Beijing, China) was used following the manufacturer's instructions.

3.4 Data analysis

The results obtained for all the tests were submitted to the normality test. After proving the normality of the sample data distribution, the data were analyzed using one way ANOVA statistical test and Tukey's multiple comparisons test by statistical software (IBM SPSS Statistics, version 22) at a significance level of 5%.

4. Results and Discussion

4.1 Results

4.1.1 Chemical composition and particle size

After the preparation of tricalcium silicate cement, phase analysis element compositions of the TCS cement powder and Biodentine powder were characterized using XRD as shown in Figure 2. The results of XRD showed that tricalcium silicate was the main phase of Biodentine and TCS cement. Zirconia phase appeared in Biodentine while zircon was presented in tricalcium silicate cement.

The particle size of tricalcium silicate cement was characterized by a dynamic laser scattering particle size analyzer. The results revealed that particle size distribution (d50) of tricalcium silicate cement with 10%, 20%, and 30% zircon were 3.26, 4.25, and 3.15 μm respectively. Biodentine showed a larger particle size of 6.26 μm .

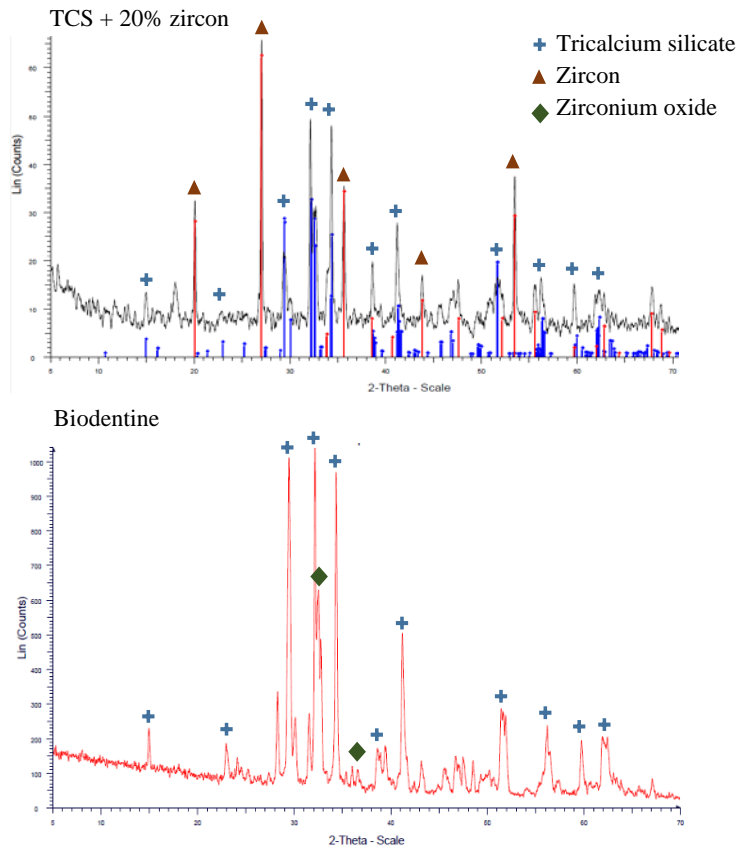


Figure 2 Phase analysis from XRD of TCS cement with 20% zircon and Biodentine

4.1.2 Setting time

The initial and final setting times of all test materials were shown in Table 1. TCS cement with a higher concentration of zircon exhibited a longer initial and setting time. At the concentration of 20% zircon, the initial and final setting time of TCS cement (14:10 and 20:20 min) was not significantly different from Biodentine (14:30 and 19:00 min).

Table 1 Initial and setting time of Biodentine, TCS cement with 10%, 20%, and 30% zircon

Group	Initial setting time (min)			Final setting time (min)		
	mean	SD	p	mean	SD	p
Biodentine	14:30 ^b	0:42	0.022	19:00 ^e	1:24	0.017
TCS 10% zircon	06:45 ^a	1:45		10:30 ^d	2:49	
TCS 20% zircon	14:10 ^b	0:14		20:20 ^e	0:28	
TCS 30% zircon	18:35 ^c	0:35		25:25 ^f	1:18	

Values with the different superscript letter are significantly different (Tukey multiple comparisons test, $p < 0.05$)



4.1.3 Compressive strength

The compressive strength testing of all cement groups displayed comparative stress at maximum load as shown in Table 2. TCS cements with the concentration of zircon higher than 10% resulted in higher compressive strength. The compressive strength of TCS cement adding 20% zircon (53.07 MPa) and 30% zircon (56.23 MPa) were similar to Biodentine (53.98 MPa).

Table 2 Compressive strength value of Biodentine, TCS cement with 10%, 20% and 30% zircon

Group	Mean (MPa)	SD	p
Biodentine	53.98 ^b	5.80	<0.000
TCS 10% zircon	46.15 ^a	3.84	
TCS 20% zircon	53.07 ^b	3.97	
TCS 30% zircon	56.23 ^b	5.32	

Values with a different superscript letter are significantly different (Tukey multiple comparisons test, $p < 0.05$)

4.1.4 Radiopacity

The results of radiopacity evaluation were shown in Table 3. TCS cement with 10% zircon (3.66 mm Al) had similar radiopacity to Biodentine (3.92 mm Al). The radiopacities of TCS with 20% zircon (5.64 mm Al) and 30% zircon (6.47 mm Al) were significantly higher than Biodentine and TCS cement with 10% cement.

Table 3 Radiopacity of Biodentine, TCS cement with 10%, 20% and 30% zircon

group	Mean (mm Al)	SD	p
Biodentine	3.92 ^a	0.52	<0.000
Chem 10%	3.66 ^a	0.30	
Chem 20%	5.64 ^b	0.15	
Chem 30%	6.47 ^b	0.11	

Values with a different superscript letter are significantly different (Tukey multiple comparisons test, $p < 0.05$)

4.1.5 pH

The pH values of Biodentine and TCS cement after immersed in distilled water for the duration of 3 hours to 8 weeks were illustrated in Figure 3. Varying the amount of Zircon did not affect the pH values of TCS cement. TCS cement showed significantly higher pH compared to Biodentine at the duration of 3 hours to 3 weeks. At three hours, TCS cement exhibited a pH of 11.9-12 while Biodentine showed lower pH of 10.2. At two weeks, the peak pH values of Biodentine and TCS cement were 12.2 and 12.7-12.8 respectively. Then, the pH values of all tested cement reduced and were stable after 4 weeks at approximately 10.2.

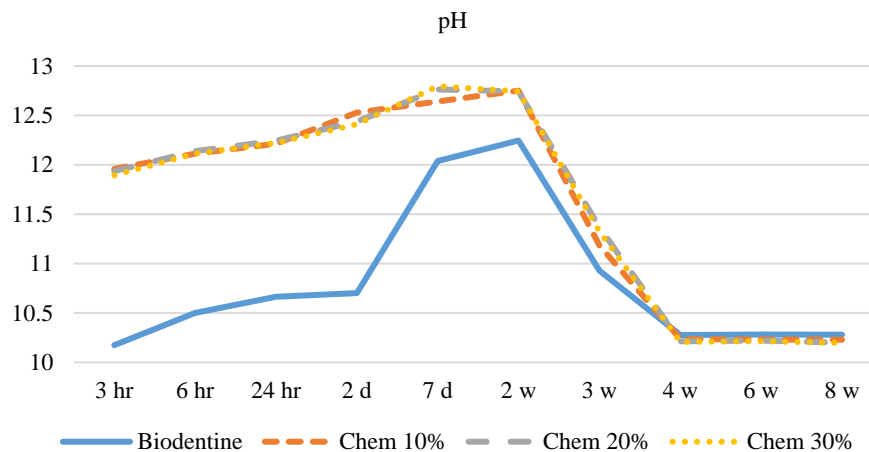
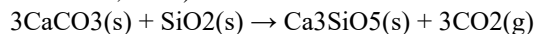


Figure 3 The pH values of Biodentine and TCS cement with 10%, 20%, and 30% zircon after immersed in distilled water for 3 hours to 8 weeks

4.2 Discussion

The chemical analysis of TCS cement showed similar compositions to Biodentine, which showed the main composition of tricalcium silicate without other impurities such as heavy metal similar to the study by (Camilleri et al., 2013). The reaction of tricalcium silicate cement was displayed by the equation:



The critical point to maintain the TCS phase is a rapid temperature reduction to below 1250°C. If the cooling down step of quenching is not enough, a small amount of TCS could be dissociated to dicalcium silicate and calcium oxide in the quenching step.

According to ISO 6876:2012 (3) and 9917-1:2007 (4), dental cement should provide satisfactory physical properties, which are practical for clinical application. The physical properties tested including setting time, compressive strength, radiopacity, and pH evaluations were conducted according to ISO 6876:2012 (3) and 9917-1:2007 (4), which was similar to the previous studies (Camilleri, 2011; Camilleri et al., 2013; Formosa, Mallia, & Camilleri, 2012; Grech, Mallia, & Camilleri, 2013). The initial and final setting time of TCS cements with a higher concentration of zircon exhibited a higher setting time. As the concentration of zircon in TCS cement was increased, the concentration of TCS was decreased. The setting time of TCS was accelerated by the CaCl_2 solution. It is hypothesized that with the progress of hydration, the area of hydrated tricalcium silicate pastes gradually increases due to the formation of hydration products such as calcium silicate hydrate gel and calcium hydroxide, and the addition of calcium chloride would accelerate this increasing rate (Wang et al., 2008). Therefore, if the proportion of TCS was decreased by adding the bigger amount of zircon, the ratio of TCS to CaCl_2 solution was lower, which caused the shorter setting time. At the concentration of 20% zircon, the initial and final setting time of TCS cement (14:10 min and 20:20 min) was not significantly different from Biodentine (14:30 min and 19:00 min). Within the observed setting time, treatment of single visit regenerative endodontics was possible.

The compressive strength of TCS was influenced by the proportion of the added zircon. TCS cement with 10% to 30% zircon showed the compressive strength in the range of 46.15 to 56.23 MPa, which were higher than tricalcium silicate cement with 20% zirconium oxide (23.55 MPa) (Grech et al., 2013) and 20% bismuth oxide (27.16 MPa) (Formosa et al., 2012). TCS cement with more than 10% zircon exhibited a similar compressive strength to Biodentine. The ISO 9917-1:2007 recommends the minimum compressive strength of 50 MPa for dental cement used for base/lining of which was comparable to our results.



The added radiopacity to dental cement enhanced the visual differentiation of the materials from the tooth structure on the radiograph. ISO 6876:2012 recommends that dental cement should provide the radiopacity of more than 3 mm of aluminum. Literature showed that zirconium oxide (Camilleri et al., 2013; Grech et al., 2013) and bismuth oxide (Formosa et al., 2012; Torabinejad et al., 1995) has been widely used as radiopacifier. The drawback of using bismuth oxide was causing tooth discoloration (Marciano et al., 2014) and reduction of the compressive strength (Camilleri, 2008b; Coomaraswamy, Lumley, & Hofmann, 2007). The rationale for using zircon (zirconium silicate) as a radiopacifier were inexpensive, providing high radiopacity, biocompatible, bioinert, and favorable mechanical properties (Piconi C, 1999). Up to date, there is no dental cement that uses zircon as a radiopacifier. Our results showed that adding zircon at the concentration of 10% or more provided more than 3 mm radiopacity, which was higher than the recommended value of 3 of ISO 6876:2012. 20% and 30% zircon exhibited the radiopacity of 5.6 and 6.4, respectively, which was higher than Biodentine.

The observed pH values of TCS cement at the observation of 3 hours to 3 weeks were higher than Biodentine. After four weeks, pH values of TCS and Biodentine were stable at approximately 10.2. During hydration, TCS reacted with water to form calcium silicate hydrate gel and calcium hydroxide. The release of calcium hydroxide resulted in increasing the pH of the cement over time. A previous study found that TCS mixed with pure water exhibited a pH of 11.5 after immersed in water for 28 days (Camilleri, 2011). The high pH exhibits antimicrobial activities, triggering low-grade inflammation leading to tissue healing and promoting remineralization (Siqueira & Lopes, 1999). Several bacterial species commonly found in infected root canals including *E. faecalis* are eliminated at pH 12.5 (Bystrom, Claesson, & Sundqvist, 1985). The pH 8.6 to 10.3 activates alkaline phosphatase, which relates to the process of mineralization (Thompson, 1966).

5. Conclusion

The local-made laboratory synthesized TCS with 20% zircon and 20% calcium chloride solution posed a similar setting time (14:10 min) and compressive strength (53.07 MPa) to Biodentine. Further research should investigate the biocompatibility of TCS cement. Moreover, TCS cement should be clinically tested in both animals and humans prior to recommendation for clinical application.

6. Acknowledgements

This research was supported by the Chulalongkorn Academic Advancement into Its Second Century Project.

7. References

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