Original Article

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# Early and 24-hour shear bond strength to dentine of three calcium silicate based pulp capping materials

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Objective: The aim of this study was to evaluate shear bond strength (SBS) at early and 24 h after setting of three calcium silicate based capping materials.

Materials and Methods: Eighty permanent maxillary premolars were sectioned to expose coronal dentine. Specimens were randomly divided into four groups, n=20 each. Biodentine, RetroMTA, Theracal, and Dycal (control) were filled in 3-mm diameter tubes and placed on dentine. In each group, specimens were further divided into two subgroups for SBS testing at 60 min and 24 h (n=10 each). Failure modes were evaluated using a stereomicroscope.

Results: At 60 min, all tested materials showed very low mean SBS (0.38-0.42 MPa), that did not significantly differ ($p$ >0.05), and all specimens showed 100% adhesive failure. At 24 h, means SBS of Biodentine (1.01 MPa) and RetroMTA (1.15 MPa) significantly increased and were significantly higher than those of Dycal (0.37 MPa) and Theracal (0.44 MPa) ($p$ <0.05). However, 24-h SBS of Biodentine and RetroMTA were not significantly different ($p$ >0.05). In addition, 24-h bond strength of Theracal was not significantly different from that of Dycal ($p$ >0.05). Dycal and Theracal showed 100% adhesive failure, while mixed failure was observed in Biodentine and RetroMTA groups.

Conclusions: SBS of Biodentine, Theracal and RetroMTA were very low at 60 min and did not significantly differ from Dycal. SBS of Biodentine and RetroMTA significantly increased at 24 h and were higher than Theracal and Dycal.

Keywords: Biodentine, bond strength, calcium silicate, dentine, pulp capping, RetroMTA

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## Introduction

Direct pulp capping is a therapy to maintain vitality of dental pulp. A pulp capping material is placed over the exposed pulp to promote pulp healing and generate reparative dentine [1]. An ideal capping material should stimulate reparative dentine formation, provide a bacterial seal, adhere to dentine and restorative material [2]. Calcium hydroxide (CH) has been considered as a material of choice for direct pulp capping with a long-term record of clinical success [1, 2]. However, CH possesses drawbacks such as high solubility, tunnel defects in reparative dentine, and no adhesive properties [1, 2].

Mineral trioxide aggregate (MTA; *ProRoot MTA, Dentsply, PA, USA*) was introduced as a dental calcium silicate cement (CSC) and has been promoted in use for direct pulp capping [3, 4] with high clinical success rate [5]. Nevertheless, major disadvantages of MTA are long setting time, poor handling properties, and induction of tooth

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discoloration [6]. In addition, MTA has relatively low adhesion to dentine [7]. In direct pulp capping, MTA tends to be displaced and needs a protective lining over the material.

Biodentine (*Septodont, Saint Maur des Fossés, France*), a fast-setting CSC, has been introduced. The powder consists of dicalcium silicate, calcium carbonate, calcium oxide and zirconium oxide as a radiopacifier. The liquid consists of water, calcium chloride and hydrosoluble polymer [8]. Biodentine has initial setting time at 12 min [9], and its physical properties are improved [8]. Biodentine adheres to dentine, which the bond strength increases over periods [7, 10]. It seems that Biodentine might be suitable as a direct pulp capping material [11].

Theracal (*Bisco Inc., Schaumburg, IL, USA*) is a resin-modified MTA [12], designated for pulp capping or as a lining. It consists of 45% Portland cement, 45% methacrylate resin and other ingredients [13]. This CSC can be set immediately by light curing for 20 s. Low solubility [14], adhesion to dentine [15] and ability to induce dentine bridge formation in primate [16] have been reported for Theracal.

RetroMTA (*BioMTA Co, Ltd, Seoul, Korea*) is a fast-setting CSC that initial setting is about two min and 30 s after mixing [17] . RetroMTA consists of calcium carbonate, silicon dioxide, aluminum oxide and calcium zirconia complex as a radiopacifier [18]. RetroMTA is as biocompatible as ProRoot MTA [19]. This CSC does not induce tooth discoloration [20] and provides good marginal adaptation [21]. RetroMTA might be used as a direct pulp capping material [22].

One of the required properties of a direct pulp capping material is adhesion to dentine [2]. For clinical relevance, bond strength to dentine of a pulp capping material should be tested after setting as early as possible. Currently, adhesion to dentine of these new fast-setting calcium silicate based cements remains unclear. Therefore, this study aims to determine shear bond strength to dentine, at early setting and 24 h, of Biodentine, Theracal and RetroMTA, in comparison with hard-setting calcium hydroxide cement, Dycal (*Dentsply Caulk, Milford, DE, USA*).

## Material and Methods

This research was approved by the Institutional Review Board, Faculty of Dentistry/Faculty of Pharmacy, Mahidol University with COE. No. MU-DT/PY-IRB 2016/002.0102. Eighty permanent maxillary premolars extracted for orthodontic reason from 16-40 years old patients were collected. Teeth with caries, restoration or crack were excluded. All teeth were cleaned, stored in 0.1% thymol solution, and used within three months. To estimate the level of pulp horns, a digital radiograph was taken in the mesio-distal view (*Kodak2200, Eastman Kodak Company, Rochester, NY and Kodak RVG 6500 sensor*). Each tooth was horizontally sectioned at the level of 1 mm in approximate above the pulp horns (Fig.1) with an Isomer diamond saw (Buehler, Lake Bluff, NY, USA) to expose coronal dentine. The sectioned tooth was embedded in a polyvinyl ring with self-curing acrylic resin (*Prothyl repair EVO, Zhermack, Badia Polesine, Italy*). The dentine surface was polished with 600-grit silicon carbide paper and then cleaned in an ultrasonic cleanser for 10 min. The specimens were randomly divided into four groups, 20 of each, according to tested capping materials- Biodentine, RetroMTA, Theracal, and Dycal (control).

Biodentine, RetroMTA, and Dycal were mixed according to the manufacturers' instructions. Mixed material was filled in a plastic tube, 3 mm in diameter and height, which was then placed on dentine surface. For Theracal, the tube was firstly placed on dentine, filled with one-mm increment of Theracal and then light-cured for 20 s (*Elipar Highlight; 3M ESPE, Seefeld, Germany*). Twenty specimens of each capping material were further divided into two subgroups (n = 10 each) for shear bond strength testing at 60 min and 24 h after setting. From a pilot study, the materials could not be tested before 60 min due to pre-testing failure of bonded specimens. All specimens were stored in an incubator (*Memmert, Schwabach, Germany*) at 37°C and 100% humidity.

Shear bond strength testing was performed using a universal testing machine (*EZ-S, Shimadzu, Kyoto, Japan*). A knife-edge plunger for shear test was placed close to the bonded surface and loaded with a speed of 0.5 mm/min until failure (Fig.1). Shear bond strength in MPa was calculated using a software program (*Trapezium 2, Shimadzu, Kyoto, Japan*). Failure modes were evaluated using a stereomicroscope (*EMZ-5TR SZTP, MEIJI, Japan*) at 20x magnification. Failure modes were classified into three categories according to Price *et al*. [23] as follows: 1) *adhesive failure*- failure at the material-dentine interface ≥70% of de-bonded area, 2) *cohesive failure*- failure within material ≥70% of de-bonded area and 3) *mixed failure*- either adhesive or cohesive failure at less than 70% of de-bonded area.

Non-parametric statistical analyses were used because the data were not normally distributed. Shear bond strengths were statistically analyzed using Kruskal-Wallis test and Mann-Whitney U test with a significant level at *p-va*lue of .05.

## Results

### Shear bond strength

At 60 min, all tested materials showed very low shear bond strength. Shear bond strength of Dycal (0.42 MPa), Biodentine (0.38 MPa), Theracal (0.42 MPa) and RetroMTA (0.41 MPa) did not significantly differ ($p > .05$) (Table 1). After 24 h, shear bond strength of Biodentine (1.01 MPa) and RetroMTA (1.15 MPa) significantly increased from those at 60 min ($p < .05$), and were significantly higher than those of Dycal (0.37 MPa) and Theracal (0.44 MPa) ($p < .05$) (Table 1). However, the 24-h bond strengths of Biodentine and RetroMTA were not significantly different ($p > .05$). In addition, the 24-h bond strength of Theracal was not significantly different from that of Dycal ($p > .05$).

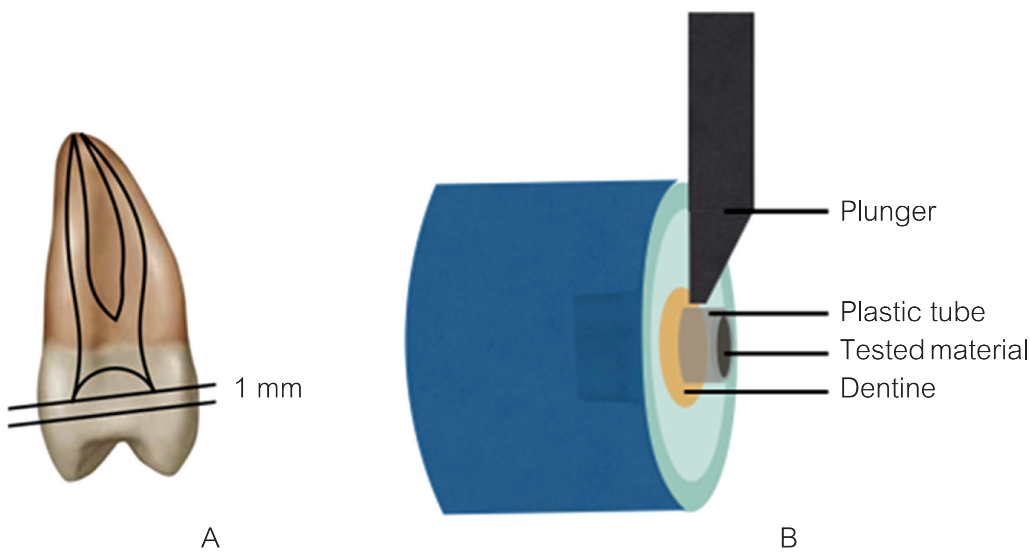


Fig. 1 (A) Each tooth was horizontally sectioned at the level of 1 mm in approximate above the pulp horns with an Isomer diamond saw (Buehler, Lake Bluff, NY, USA) to expose coronal dentine. (B) Shear bond strength testing was performed using a universal testing machine. A knife-edge plunger for shear test was placed close to the bonded surface and loaded with a speed of 0.5 mm/min until failure.

Table 1 Shear bond strength to dentine (MPa, mean ± SD) of pulp capping materials at 60 min and 24 h after setting.

| Material | Shear bond strength (mean ± SD) | | *p* - value* |
|---|---|---|---|
| | 60 min | 24 h | |
| Dycal | 0.42 ± 0.18 | 0.37 ± 0.11 | 0.63 |
| Biodentine | 0.38 ± 0.13 | 1.01 ± 0.36 | < 0.01 |
| Theracal | 0.42 ± 0.14 | 0.44 ± 0.20 | 0.91 |
| RetroMTA | 0.41 ± 0.15 | 1.15 ± 0.32 | < 0.01 |
| *p* - value ** | 0.91 | < 0.01 | |

* Mann-Whitney U test to compare SBS between setting periods ($p < 0.05$).

** Kruskal-Wallis test to compare SBS among pulp capping materials ($p < 0.05$).

## Mode of Failure

At 60 min, all specimens showed adhesive failure (Fig.2). At 24 h, all specimens of Dycal and Theracal showed adhesive failure, whereas mixed failure was observed in Biodentine (10%) and RetroMTA (20%) (Fig.3).

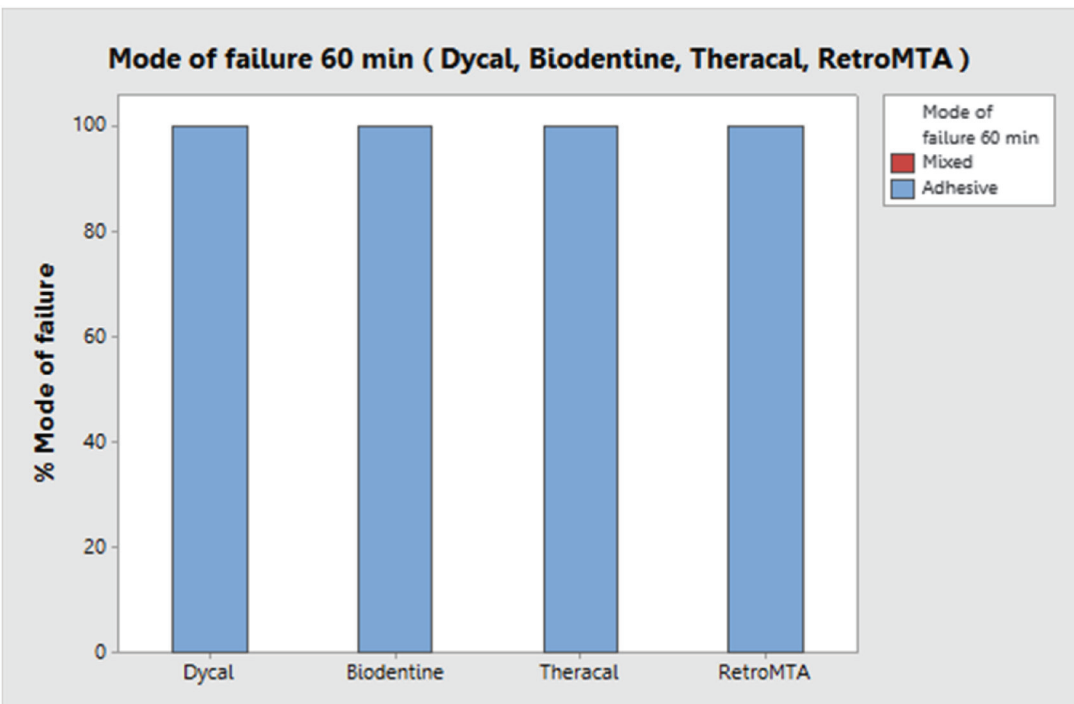


Fig. 2 Mode of failure from the 60-min shear-bond testing. All specimens showed adhesive failure.

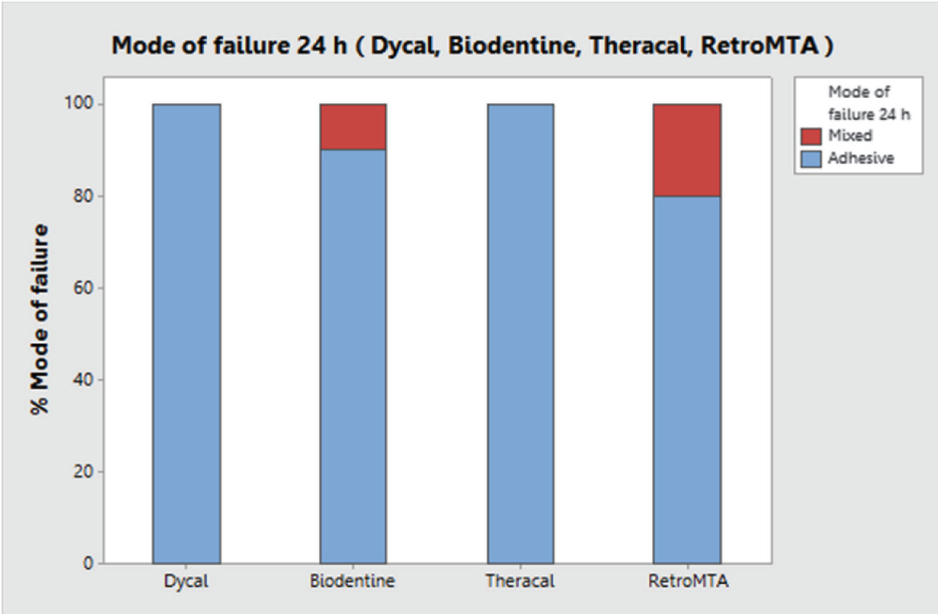


Fig. 3 Mode of failure from the 24-h shear-bond testing. All specimens of Dycal and Theracal showed adhesive failure. Mixed failure was observed in Biodentine (10%) and RetroMTA (20%).

## Discussion

Since a tooth is immediately restored after direct pulp capping, adhesion to dentine of a capping material should be tested as early as possible. However, at our preliminary test, the materials could not be tested earlier than 60 min because pre-testing failure was very high. Thus, early bond strength of these materials was tested at 60 min. The capping materials were also tested at 24 h since the bond strength might increase over period.

Very low early bond strength of all tested capping materials may be interpreted as a clinical concern. These materials might be displaced during restorative procedures. The capping material should be covered with lining, such as glass ionomer cement, before acid etching and rinsing. Biodentine and RetroMTA showed increased bond strength at 24 h with a presence of mixed failure. The results are similar to Kaup *et al*. [7] who compared bond strength to dentine of Biodentine at 2, 7 and 14 days after setting and reported that the bond strength significantly increased over the periods. However, the increased bond strength were very low and not enough to withstand any force during restoration placement.

Bond strength of Biodentine and RetroMTA may be explained by adhesion to dentine of CSC that chemical and/or micromechanical adhesion might be created [24-26]. CSC might bond chemically to dentine through the formation of hydroxyapatites [24]. Furthermore, additional retention of CSC to dentine would be expected through physical adaptation [25] and penetration in dentinal tubules. High pH of hydrated Biodentine could induce denaturing and increasing permeability of dentine [26], and flowable consistency of CSC may promote the penetration into dentinal tubules, resulting in micromechanical retention [26]. RetroMTA might adhere to dentine through the same mechanism of chemical reaction and micromechanical retention.

Theracal is partially composed of CSC, but shear bond strength did not differ from Dycal, a hard-setting calcium hydroxide, that did not adhere to dentine [2]. Low bond strength of Theracal may be attributed to methacrylate resin content that can cause shrinkage after light-curing polymerization. Polymerization shrinkage might result in early bond failure and low bond strength [8]. In addition, CSC content in Theracal was only 45%, and this reduces the chance to form a chemical or micromechanical adhesion to dentine.

## Conclusion

At 60 min after setting, Biodentine, Theracal, and RetroMTA had very low shear bond strength that did not significantly differ from that of Dycal. At 24 h, bond strength of Biodentine and RetroMTA significantly increased and were higher than those of Theracal and Dycal.

### Conflict of interest, funding and ethic approval

The authors deny any conflicts of interest. This research was supported by a grant from the Endodontic Division, Department of Operative Dentistry and Endodontics, Faculty of Dentistry, Mahidol University. This research was approved by the Institutional Review Board, Faculty of Dentistry/Faculty of Pharmacy, Mahidol University with COE. No. MU-DT/PY-IRB 2016/002.0102.

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## References

1. Hilton TJ. Keys to clinical success with pulp capping: a review of the literature. ***Oper Dent*** 2009; 34: 615-625.
2. Qureshi A, E S, Nandakumar, Pratapkumar, Sambashivarao. Recent advances in pulp capping materials: an overview. ***J Clin Diagn Res*** 2014; 8: 316-321.
3. Torabinejad M, Parirokh M. Mineral trioxide aggregate: a comprehensive literature review--part II: leakage and biocompatibility investigations. ***J Endod*** 2010; 36: 190-202.
4. Gandolfi MG, Taddei P, Siboni F, Modena E, Ciapetti G, Prati C. Development of the foremost light-curable calcium-silicate MTA cement as root-end in oral surgery. Chemical-physical properties, bioactivity and biological behavior. ***Dent Mater*** 2011; 27: e134-157.
5. Mente J, Hufnagel S, Leo M, Michel A, Gehrig H, Panagidis D, et al. Treatment outcome of mineral trioxide aggregate or calcium hydroxide direct pulp capping: long-term results. ***J Endod*** 2014; 40: 1746-1751.
6. Parirokh M, Torabinejad M. Mineral trioxide aggregate: a comprehensive literature review--Part III: Clinical applications, drawbacks, and mechanism of action. ***J Endod*** 2010; 36: 400-413.
7. Kaup M, Dammann CH, Schafer E, Dammaschke T. Shear bond strength of Biodentine, ProRoot MTA, glass ionomer cement and composite resin on human dentine ex vivo. ***Head Face Med*** 2015; 11: 14-21.
8. Dawood AE, Parashos P, Wong RH, Reynolds EC, Manton DJ. Calcium silicate-based cements: composition, properties, and clinical applications. ***J Investig Clin Dent*** 2015; DOI: 10.1111/jicd.12195.
9. Camilleri J, Sorrentino F, Damidot D. Investigation of the hydration and bioactivity of radiopacified tricalcium silicate cement, Biodentine and MTA Angelus. ***Dent Mater*** 2013; 29: 580-593.
10. Raju VG, Venumbaka NR, Mungara J, Vijayakumar P, Rajendran S, Elangovan A. Comparative evaluation of shear bond strength and microleakage of tricalcium silicate-based restorative material and radioopaque posterior glass ionomer restorative cement in primary and permanent teeth: an in vitro study. ***J Indian Soc Pedod Prev Dent*** 2014; 32: 304-310.

11. Nowicka A, Lipski M, Parafiniuk M, Sporniak-Tutak K, Lichota D, Kosierkiewicz A, et al. Response of human dental pulp capped with Biodentine and mineral trioxide aggregate. *J Endod* 2013; 39: 743-747.
12. Gomes-Filho JE, de Faria MD, Bernabe PF, Nery MJ, Otoboni-Filho JA, Dezan-Junior E, et al. Mineral trioxide aggregate but not light-cure mineral trioxide aggregate stimulated mineralization. *J Endod* 2008; 34: 62-65.
13. Shen Y, Peng B, Yang Y, Ma J, Haapasalo M. What do different tests tell about the mechanical and biological properties of bioceramic materials? *Endo Topics* 2015; 32: 47-85.
14. Gandolfi MG, Siboni F, Botero T, Bossu M, Riccitiello F, Prati C. Calcium silicate and calcium hydroxide materials for pulp capping: biointeractivity, porosity, solubility and bioactivity of current formulations. *J Appl Biomater Funct Mater* 2015; 13: 43-60.
15. Hebling J, Lessa FC, Nogueira I, Carvalho RM, Costa CA. Cytotoxicity of resin-based light-cured liners. *Am J Dent* 2009; 22: 137-142.
16. Cannon M, Gerodias N, Viera A, Percinoto C, Jurado R. Primate pulpal healing after exposure and TheraCal application. *J Clin Pediatr Dent* 2014; 38: 333-337.
17. Souza LC, Yadlapati M, Dorn SO, Silva R, Letra A. Analysis of radiopacity, pH and cytotoxicity of a new bioceramic material. *J Appl Oral Sci* 2015; 23: 383-389.
18. Kang CM, Kim SH, Shin Y, Lee HS, Lee JH, Kim GT, et al. A randomized controlled trial of ProRoot MTA, OrthoMTA and RetroMTA for pulpotomy in primary molars. *Oral Dis* 2015; 21: 785-791.
19. Chung C, Kim E, Song M, Park J-W, Shin S-J. Effects of two fast-setting calcium-silicate cements on cell viability and angiogenic factor release in human pulp-derived cells. *Odontology* 2016: 104:143-151.
20. Kang SH, Shin YS, Lee HS, Kim SO, Shin Y, Jung IY, et al. Color changes of teeth after treatment with various mineral trioxide aggregate-based materials: an ex vivo study. *J Endod* 2015; 41: 737-741.
21. Ghorbanzadeh A, Shokouhinejad N, Fathi B, Raoof M, Khoshkhounejad M. An In Vitro Comparison of Marginal Adaptation of MTA and MTA-Like Materials in the Presence of PBS at One-Week and Two-Month Intervals. *J Dent (Tehran)* 2014; 11: 560-568.
22. Lee H, Shin Y, Kim SO, Lee HS, Choi HJ, Song JS. Comparative Study of Pulpal Responses to Pulpotomy with ProRoot MTA, RetroMTA, and TheraCal in Dogs' Teeth. *J Endod* 2015; 41: 1317-1324.
23. Price RB, Doyle G, Murphy D. Effects of composite thickness on the shear bond strength to dentine. *J Can Dent Assoc* 2000; 66: 35-39.
24. Sarkar NK, Caicedo R, Ritwik P, Moiseyeva R, Kawashima I. Physicochemical basis of the biologic properties of mineral trioxide aggregate. *J Endod* 2005; 31: 97-100.
25. Reyes-Carmona JF, Felippe MS, Felippe WT. The biomineralization ability of mineral trioxide aggregate and Portland cement on dentine enhances the push-out strength. *J Endod* 2010; 36: 286-291.
26. Atmeh AR, Chong EZ, Richard G, Festy F, Watson TF. Dentine-cement interfacial interaction: calcium silicates and polyalkenoates. *J Dent Res* 2012; 91: 454-459.