Push-out strength of modified Portland cements and resins

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Abstract: Purpose: Modified calcium-silicate cements derived from white Portland cement (PC) were formulated to test their push-out strength from radicular dentin after immersion for 1 month. **Methods:** Slabs obtained from 42 single-rooted extracted teeth were prepared with 0.6 mm diameter holes, then enlarged with rotary instruments. After immersion in EDTA and NaOCl, the holes were filled with modified PCs or ProRoot MTA, Vitrebond and Clearfil SE. Different concentrations of phyllosilicate (montmorillonite-MMT) were added to experimental cements. ProRoot MTA was also included as reference material. Vitrebond and Clearfil SE were included as controls. Each group was tested after 1 month of immersion in water or PBS. A thin-slice push-out test on a universal testing machine served to test the push-out strength of materials. Results were statistically analyzed using the least squares means (LSM) method. **Results:** The modified PCs had push-out strengths of 3-9.5 MPa after 1 month of immersion in water, while ProRoot MTA had 4.8 MPa. The push-out strength of PC fell after incubation in PBS for 1 month, while the push-out strength of ProRoot MTA increased. There were no significant changes in Clearfil SE Bond or Vitrebond after water or PBS storage. (*Am J Dent* 2010;23:43-46).

Clinical significance: Incorporation of phyllosilicate in the experimental Portland cements did not improve the push-out strength compared to the commercially available ProRoot MTA. PBS immersion decreased the push-out strength of modified Portland cements while ProRoot MTA exhibited higher push-out strength after immersion in PBS.

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Introduction

Modified Portland cements (PCs) like mineral trioxide aggregate (MTA) have multiple uses in dentistry.[1-4] These materials contain tricalcium and dicalcium silicate, and consist of a powder of fine hydrophilic particles that sets in water. Several studies are available on the chemical and physical properties of these materials.[5-7] Many authors agree that a significant feature of these materials is their ability to create an adequate seal.[8-10] Evidence of the interaction of Portland cements with phosphate buffered saline (PBS) resulting in the formation of hydroxyapatite (HA) crystals[11,12] indicates that these cements can create HA in physiological tissue fluids.[13]

Although PC-based cements fulfill most of the requirements for an endodontic filling material, their working properties are less than ideal. When these cements are mixed with water, the resulting cement pastes are difficult to handle and the setting times are long. Calcium chloride has often been incorporated in PC-based cements as an accelerator to shorten the setting time with a minimal impact on their physical properties or leakage.[14-16] Calcium chloride and phyllosilicate[a] (montmorillonite)-containing materials based on Portland cement were recently developed for endodontics to improve handling and physical characteristics and extend the clinical applications. The new materials showed improved *in vitro* properties such as marginal adaptation and sealing ability[9,17] and biocompatibility.[18,19] These studies used phyllosilicate clay as a plasticizing agent to improve the handling characteristics and dimension stability of the PC-based cements.

The ability of endodontic materials to resist deformation of established seals *via* micromechanical retention or friction is essential to the survival of the material-dentin interface during intraoral tooth flexure.[20]

This study assessed the push-out strength of modified PCs. A dentin adhesive and a resin-modified glass-ionomer cement were used as control materials. All the materials were evaluated after 1 month of incubation in water or PBS. The null hypothesis was that the push-out strength does not differ in the modified PCs and ProRoot MTA.

Materials and Methods

Sample preparation - Forty-two single-rooted teeth extracted for orthodontic/periodontal reasons were collected under a protocol reviewed and approved by the Human Assurance Committee of the Medical College of Georgia. For each tooth, a 0.90 ± 0.10 mm thick longitudinal slab was prepared by making buccolingual cuts perpendicular to the longitudinal axis of the tooth using a slow-speed diamond saw[b] under water-cooling. A 0.6 mm drill bit was used to prepare pilot holes in the radicular dentin. Each pilot hole was carefully drilled so that it was equidistant from the cementum and the canal wall. Six pilot holes were prepared for each tooth. Each hole was then enlarged using a size 40, 25 mm long 0.04 taper Profile nickel titanium rotary instrument.[c] A miniature drill press was configured so that the Profile files penetrated to the D16 diameter of the rotary instrument along the surface of the tooth slab. This permitted preparation of 252 truncated holes that simulated standardized circular defects. The tooth slabs were immersed in 17% EDTA and ultrasonicated for 5 minutes to remove the smear layer created during the hole-shaping procedures. The slabs were then immersed in 6.15% sodium hypochlorite (NaOCl) and ultrasonicated for 5 minutes to dissolve organic debris.

The 42 root slabs containing 252 holes were divided into seven groups, each containing 36 holes: Group I was filled with white PC (CEM I[d]) mixed with anhydrous calcium sulphate and calcium chloride (PC1); Groups II, III and IV (PC2, PC3, PC4, Table 1) were filled with the same modified

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Table 1. Composition of tested materials..

Code	Composition
PC1	White Portland cement (thermally and mechanically treated), calcium sulphate, calcium chloride
PC2	Same as PC1 but with addition of 1% phyllosilicate
PC3	Same as PC1 but with addition of 2% phyllosilicate
PC4	Same as PC1 but with addition of 5% phyllosilicate
ProRoot MTA	Same as PC1 but with addition of bismuth oxide for radiopacity sterilization and sieving to narrow particle size.
Vitrebond	Polyacrylic acid with pendent vinyl goups and diphenyliodonium chloride to make it light curable, plaus acid-susceptable glass fillers.
Clearfil SE	*Primer:* hydroxyethyl methacrylate (HEMA), water, ethanol, 10-methacryloyloxydecamethylene phosphotic acid (MDP); *Adhesive:* HEMA, MDP, dimethacrylates.

Portland cement but mixed with 1, 2 and 5 wt% of phyllosilicate.[a] The experimental modified PCs are patented formulations (University Patent EP 07425074.7 and USA US60/900.467; extension PCT/EP2008/051583) designed and prepared at the Centre of Biomineralogy, Crystallography and Biomaterials.[e] Group V holes were filled with ProRoot MTA,[c] Group VI holes were filled with Vitrebond[f] and Group VII holes were filled with Clearfil SE Bond.[g] The PCs and ProRoot MTA were mixed with a powder/liquid ratio of 3/1. Vitrebond and Clearfil SE Bond were used according to the manufacturer's recommendations, and cured with a LED light-curing unit (Elipar FreeLight 2[f]) with an output intensity of 600 mW/cm^2. All cavities from one tooth slab were filled with one type of cement or adhesive. Each tooth slab was placed over a Mylar strip,[h] which in turn was placed over a microscope glass slide. The cement material was forced into the cavities with a small spatula so that each hole was filled to excess with the material. The surface of the tooth slab was then covered with another Mylar strip and a glass slide. The assembly was secured with binder clips so that excess material was expressed laterally from the surface and bottom Mylar strips. The assemblies were transferred to a humidity chamber to be stored under 100% relative humidity for 48 hours. The surfaces of each tooth slab were polished with 800-grit silicon paper under water to remove excess material.

Push-out strength - The push-out strength of the material was investigated after 1 month of incubation in water or in phosphate buffered saline (PBS). To prevent microbial growth, 0.02% sodium azide was included in the solutions.

The push-out strength of the set root canal sealers was evaluated using a thin-slice push-out test design according to the method of Chandra & Ghonem.[21] Prior to testing, the thickness of each tooth slab was measured using a pair of calipers. A 0.7 mm diameter carbon steel cylindrical plunger was used for the push-out test. The plunger was attached to a 100 N load cell connected to a universal testing machine (Vitrodyne, Model V1000 Universal Tester[i]). All specimens were loaded at a cross-head speed of 0.6 mm/minute.

The push-out device consisted of a clear Plexiglas platform with a vertical cylindrical channel, which served as the support for the tooth slab and provided space for the vertical movement of the plunger through the truncated hole (Fig. 1). To ensure optimal alignment of the plunger with the sealer-filled hole, a horizontal channel was drilled through the Plexiglas platform into the vertical channel (Fig. 1). A fiber optic light guide was inserted into the horizontal channel to provide high intensity illumination of the restored truncated hole during the alignment procedure.

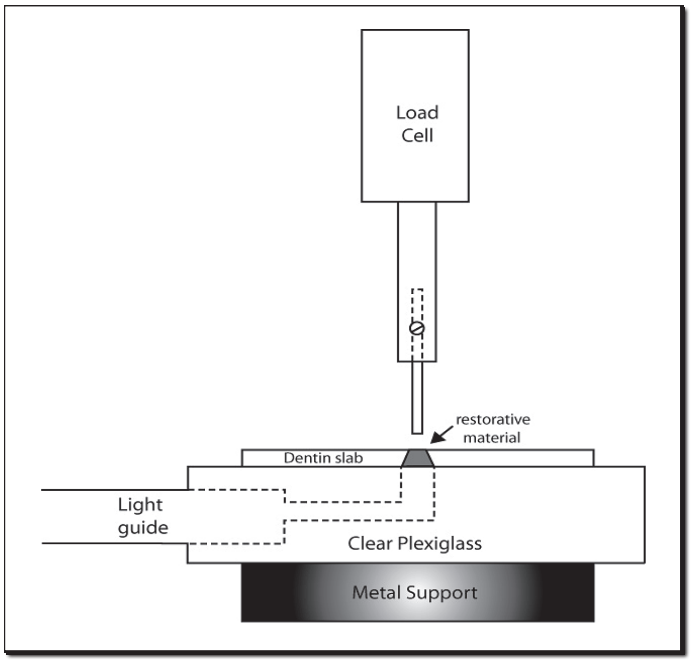

Fig. 1. Diagram of the clear plastic push-out platform mounted below the 0.7 mm diameter steel plunger that in turn was mounted on a 100 N load cell. Note the hole in the platform is directly beneath the plunger. A fiber-optic light guide inserted into a horizontal channel in the plastic plate provides high intensity illumination of the restored truncated hole during alignment procedures.

Statistical analysis of strength tests - Push-out strength of the materials was computed by dividing the maximum load (N) derived from the load displacement curve by the material-dentin interfacial area (mm^2) and expressed in megaPascals (MPa). Initial attempts to analyze the data with a two-way ANOVA (material *vs*. storage media) revealed that the data were not distributed normally, had unequal variances and had significant interactions. Therefore, the data were analyzed using the least square means (LSM) method. Least square means are the expected value of group means that one expects for a balanced design involving the group variable, with all covariates held at their mean value. The variance in the LSM value are given in standard error of the mean (SEM) instead of standard deviation (SD). Multiple comparisons of the LSM were performed by the Holm-Sidak method. Statistical significance was set in advance at $\alpha = 0.05$. The power of the LSM test was 1.0 for material, 0.9 for storage media and 0.85 for material *vs*. storage media.

Results

The push-out strength results are shown in Fig. 2. The mean 1-month push-out strength of PC1 was 10 MPa regardless of the storage solution (water or PBS). By contrast, PC2 had a very low push-out strength after 1 month of storage. When PC3 specimens were tested, although their mean values were lower than those of PC1 due to their relatively high variance, they were not significantly different from PC1, and they did not change in water or PBS. Portland cement 4 (PC4) 1-month push-out strengths in water or PBS did not significantly differ from those of PC1-2. ProRoot MTA

Table 2. Push-out strength of test materials in PBS or water.

Material	Time	Storage	Push-out strength (MPa)*
PC1 + 0% ps	1 month	PBS	12.3 ± 1.5 c
PC2 + 1% ps	1 month	PBS	1.3 ± 1.2 a
PC3 + 2% ps	1 month	PBS	7.0 ± 1.3 b
PC4 + 5% ps	1 month	PBS	8.4 ± 1.4 b
Pro Root MTA	1 month	PBS	11.6 ± 1.6 c
Vitrebond	1 month	PBS	24.2 ± 1.2 d
Clearfil SE Bond	1 month	PBS	21.2 ± 1.6 d
PC1 + 0% ps	1 month	Water	9.5 ± 1.4 c
PC2 + 1% ps	1 month	Water	3.1 ± 1.5 a
PC3 + 2% ps	1 month	Water	6.5 ± 1.2 b
PC4 + 5% ps	1 month	Water	5.1 ± 1.2 b
Pro Root MTA	1 month	Water	4.8 ± 1.4 b
Vitrebond	1 month	Water	19.7 ± 1.2 d
Clearfil SE Bond	1 month	Water	25.4 ± 1.1 d

*Values are least squares ± standard error of the mean. PC = Portland cement, ps = phyllosilicate.
Values identified by different letters are significantly different (P< 0.05).

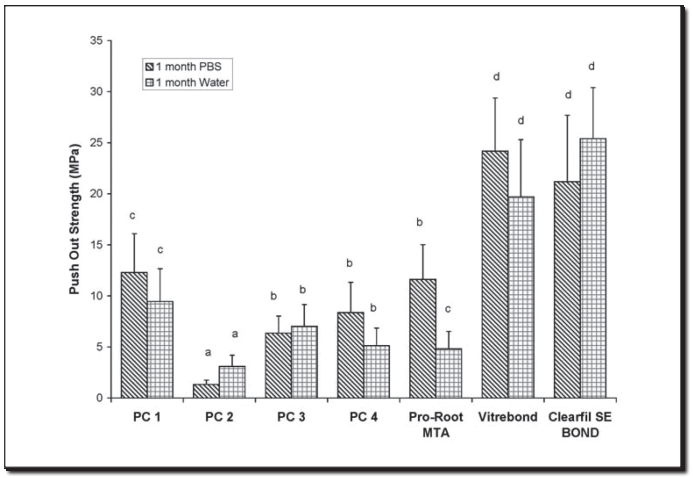

Fig. 2. Push-out strength (MPa) of modified Portland cements PC1-PC4, ProRoot MTA, Vitrebond and Clearfil SE Bond. The height of each bar represents the mean value of 10 specimens. Half brackets indicate plus one standard deviation. Different lower case letters indicate significant differences (P< 0.05) between groups tested after 1 month of immersion in water or phosphate buffered saline (PBS).

push-out strength was twice as high (P< 0.05) in PBS as in water (Table 2).

The two resin-based restoratives, Vitrebond and Clearfil SE Bond had significantly (P< 0.05) higher push-out strengths than those of the modified PCs, and their bond strengths were unaffected by time or storage solution (Fig. 2).

Discussion

The present study assessed the 1-month push-out strength of phyllosilicate-modified Portland-based cements formulated to improve their handling characteristics. Montmorillonite is a phyllosilicate mineral (deriving from deposits of weathered volcanic ash) formed by stacked silicate sheets (two silica-oxygen tetrahedral sheets sandwiching an aluminium or magnesium octahedral sheet) interposed by water and exchangeable interlayer cations (charge-balancing counterions). Montmorillonite is characterized by high cation exchange ability, swelling capacity and strong adsorption. Because of its hydrophilic nature the montmorillonite swells with the addition of water and may expand considerably due to water penetrating the interlayer molecular spaces and concomitant adsorption. Swelling produces an increase in the 001 interlayer d-spacing.[22] Crystalline swelling of 2:1 layer phyllosilicates is a thermodynamically irreversible process[23,24] and dehydration (removal of interlayer water) is an endothermic reaction starting below 150°C.[22] Previous studies included montmorillonite in the composition of glass-ionomer and bone substitute cements.[25,26]

Shrinkage is a detrimental problem affecting many cements and is responsible for gap formation and marginal sealing reduction. The irreversible swelling of montmorillonite may counteract the shrinkage and enhance dimensional stability over time. With the exception of PC2 in water and PBS, push-out strength did not differ among the modified PCs and ProRoot MTA. Thus, the null hypothesis is accepted, except for PC2.

The use of 1 mm thick root slabs perforated by standardized truncated cone holes made all holes for rinsing identical, rinsing with EDTA/NaOCl lasted exactly the same time, and all specimens were tested with the same-sized plunger. We previously found that there were no regional differences in the dislocation resistance of modified PCs among the coronal, middle and apical thirds of the radicular dentin, so that data from all regions, including the sclerotic dentin along the apical thirds of the root canal, could be pooled.[27]

Several studies have used thin slice push-out tests to evaluate the dislocation resistance of root filling materials.[28-35] Our study adopted a modified push-out protocol specifically designed to examine the retentive potential of pure sealer materials in radicular dentin.[27] Gancedo-Caravia & Garcia-Barbero[36] demonstrated that humidity increased the push-out strength of ProRoot MTA. Huffman *et al*[27] compared the push-out strength of an experimental calcium silicate-based root canal sealer, AH Plus Jet and Pulp Canal Sealer. They demonstrated a higher push-out strength of the calcium silicate-based cements, particularly after storage in PBS wherein carbonated apatites may be formed along the material-radicular dentin interface,[37] improving the frictional resistance[38] of the cement to dislocation.

Modified PCs resist displacement from dentin due to the intrinsic roughness of EDTA/NaOCl treated radicular dentin, the intrinsic roughness of the cements, and their intrinsic cohesive strength. There is some micromechanical retention due to interfacial friction and the cohesive shear strength of cement particles extending into microscopic undercuts in the dentin. As displacement force is applied to these cements vertically, it creates shear stress on the cement particles within dentin undercuts. When these shear stresses exceed the cohesive strength of the material, the bulk cement is vertically displaced slightly but may stop as another cement particle encounters another dentin undercut.

The effect of immersing calcium-silicate cements in PBS on a push-out test was first tested by Huffman *et al*[27] and then in the present study. Neither the resin-based material nor those of the PC-based materials Clearfil SE Bond and Vitrebond push-out strengths were significantly different when stored in water *vs*. PBS, except for Pro-Root MTA. Pro-Root MTA stored in water gave lower push-out strengths (P< 0.05) than those stored in PBS (Table 2, Fig. 2). The biocoating of apatite formed on the surface of Pro-Root MTA after immersion in PBS[12,27] may modify the retention and friction of cements on dentin walls. ProRoot MTA showed results not statistically different from white Portland cement

the active ingredient in white ProRoot MTA.[39] The incorporation of phyllosilicate (MMT) in white Portland cements (PCs) did not improve the push-out strength of these materials compared to commercially available ProRoot MTA. The push-out strength of ProRoot MTA was significantly higher ($P < 0.05$) after immersion in phosphate buffered solution, suggesting that simulated body fluids play an important role in increasing its mechanical properties. Further investigations are necessary to evaluate the chemical and mechanical transformation of white MTAs induced by PBS.

a. Montmorillonite-MMT, CNR, Italy.
b. Isomet, Buehler Ltd., Lake Bluff, IL, USA.
c. Dentsply Tulsa Dental Specialties, Tulsa, OK, USA.
d. CEM I, Aalborg White, Denmark.
e. Department of Earth Science, University of Bologna, Italy.
f. 3M ESPE, St. Paul, MN, USA.
g. Kuraray Medical Inc., Tokyo, Japan.
h. Angst & Pfister, Geneva, Switzerland.
i. Liverco, Inc., Burlington, VT, USA.

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