The effect of smear layer on the push-out bond strength of root canal calcium silicate cements

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A R T I C L E   I N F O

Article history:
Received 20 February 2013
Received in revised form 26 April 2013
Accepted 26 April 2013

Keywords:
Calcium silicate cements
MTA
Mineral trioxide aggregate
Endodontics
Obturation
Push-out bond strength

A B S T R A C T

Introduction. The aim of this study was to evaluate the effect of smear layer removal on the push-out bond strength between radicular dentin and three calcium silicate cements (CSC) in comparison with gutta percha and sealer.

Methods. Eighty human anterior extracted teeth were decoronated, cleaned and shaped to size 50/0.05 apically and randomly divided into 2 major groups: (A) smear layer preserved, and (B) smear layer removed using irrigation with 17% EDTA. Roots within each major group were further divided into 4 subgroups according to the obturation material used: (1) ProRoot MTA, (2) Biodentine, (3) Harvard MTA, (4) Gutta percha and AH-plus sealer. Obturated roots were stored in synthetic tissue fluid for 7 days to allow maximum setting of the root filling materials. Three 2-mm-thick slices were obtained from each root at different section levels (coronal, middle, apical). The canal diameters and slice thickness were measured, and the adhesion surface area for each slice was calculated. Push-out bond strength test was carried out using a universal testing machine. The bond failure mode was assessed under an optical microscope at 40×.

Results. The mean push-out bond strength in groups 1A, 2A and 3A were 7.54 (±1.11), 7.64 (±1.08) and 8.79 (±1.55) MPa respectively, while those for groups 1B, 2B and 3B were 6.58 (±1.13), 6.47 (±1.08), 7.71 (±1.81) MPa, respectively. In the gutta percha and sealer groups the push-out bond strength means were: 1.98 (±0.48) and 2.09 (±0.51) MPa in the preserved and removed smear layer groups respectively. The push-out strength values were significantly reduced when the smear layer was removed in the CSC groups (P<0.05) while no significant difference was detected in the gutta percha and sealer groups.

Conclusions. Based on the conditions of this ex vivo study, it can be concluded that smear layer removal is detrimental to the bond strength between calcium silicate cements and dentin.

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http://dx.doi.org/10.1016/j.dental.2013.04.020
1. Introduction

The aim of obturation of the root canal space is to provide a tight seal against the ingress of microbes and fluids into the disinfected root canal space [1]. Ideally, obturation materials should form a strong bond with the canal wall and resist dislodgement during function.

Mineral trioxide aggregate (MTA) was first developed at Loma Linda University in 1993 [2]. It was initially proposed as a perforation repair and retrograde filling material in surgical endodontics. Since then, other products with similar chemical constituents have been developed and are available commercially under different brand names. It has been proposed that a generic name be used for this class of materials [3]. “Hydraulic silicate cements” [3] and “calcium silicate cements” [4] are the most common of those proposed. Calcium silicate cements (CSC) possess several desirable properties such as superior sealing ability, bioactivity, and the ability to set in the presence of fluids [5]. Clinical studies demonstrated satisfactory outcomes of different clinical applications of CSCs [6], and recently they were used for obturation of the entire root canal space [7].

ProRoot MTA (Dentsply Maillefer, Ballaigues, Switzerland) is composed of a hydrophilic powder made of calcium silicates, which reacts with water and sets into a hard structure through a hydration reaction forming calcium hydroxide and calcium silicate hydrates [8]. Biodentine (Septodont, Saint Maur des Fosses, France) is a calcium silicate-based cement composed of a powder (in a capsule) and liquid (in a pipette). The powder consists of tricalcium and dicalcium silicate, calcium carbonate, and zirconium oxide, while the liquid contains calcium chloride as an accelerator and a water reducing agent [9]. Biodentine sets in 10 min [9] and it is promoted as a good dentin substitute in direct and indirect pulp capping procedures. Harvard MTA (Harvard Dental International GmbH, Hoppegarten, Germany) is a new encapsulated CSC with a predetermined powder: liquid ratio. The capsule is activated, mixed in an amalgamator, and the content is ejected with its corresponding gun. Harvard MTA has a working time of 2 min and sets in 40 min (manufacturer's material safety sheet).

Theoretically, CSC could be used for obturation, but details regarding the optimum conditions are not known. It has not been clearly demonstrated, for example, whether smear layer should be removed prior to obturation with these cements. The aim of this study was to determine the effect of smear layer removal on the push-out bond strength between different CSCs and dentin in comparison with that between gutta percha and sealer and dentin. The null hypotheses are: (a) there is no difference in the push-out bond strength between dentin and different obturation materials. (b) Smear layer removal does not affect the push-out bond strength between the obturation material and dentin.

2. Materials and methods

Eighty extracted human teeth with single canals and curvatures less than 5° [10] were used in this study. The crowns were removed using a water-cooled diamond wheel saw leaving 13 ± 1 mm long roots. After determining the working length and establishing a glide path, root canals were cleaned and shaped with a series of ProTaper files (S1–F3) (Dentsply, Maillefer, Ballaigues, Switzerland) to size 50/05 apically. Irrigation between each file was carried out with copious irrigation with 1% NaOCl using a 27-gauge monojet needle with a notched tip (Monoject, Kendall, Covidien, Mansfield, Massachusetts, USA) inserted to 1 mm short of the working length. Prepared roots were randomly divided into two major groups (n = 40). In group A, no attempt at removal of the smear layer was carried out. In group B, the smear layer was removed by irrigation with 1 ml of 17% EDTA (PULPDENT, Watertown, MA, USA) for a minute as recommended by Teixeira et al. [11] using the same irrigation needle as for NaOCl. To eliminate the EDTA action, irrigation with 2 ml of NaOCl was carried out followed by a final flush with 5 ml of sterile water. Within each major group, roots were further divided into four subgroups (n = 10) according to the obturation material used (Table 1). ProRoot MTA (subgroups 1A and 1B), Harvard MTA (subgroups 2A and 2B) and Biodentine (subgroups 3A and 3B) were used to obturate the root canals in their respective groups using a manual compaction technique with hand pluggers as described by EL-Ma’aita et al. [12]. In groups 4A and 4B (control groups), gutta percha and AH-plus sealer were used as the control groups. The gutta percha was applied into the canals in a thermoplastic injection technique (Obtura Spartan, Algonquin, IL, USA) to allow for the standardization of the technique between the three thirds of the canals. The coronal 2 mm of each root were sealed with a glass ionomer filling (AquaCem, DENTSPLY, Surrey, UK). Obturated roots were radiographed in 2 directions; bucco-lingual and mesio-distal, to ensure the canals were densely obturated. The samples were stored at 37° C in synthetic tissue fluid (STF) for 7 days to allow for maximum setting of the materials.

Following the storage period, each root was sectioned horizontally at three different levels (namely: coronal, middle and apical) to obtain three slices 2 ± 0.1 mm in thickness (Fig. 1). The greater and lesser root canal diameters and the thickness of each slice were recorded to the nearest 0.01 mm using a digital caliper. The adhesion surface area was calculated by the following equation:

\[
\text{Adhesion surface area (mm}^2) = \left( \frac{D_1 + D_2}{2} \right) \times \pi \times h
\]

where \(D_1\) and \(D_2\) are the greater and lesser canal diameters respectively, \(\pi\) is the constant 3.14 and \(h\) is the thickness of the obturated root slice.

The force required to dislodge the obturation material from the root slice was measured using a Universal Testing Machine (Roell Z200, Zwick GmbH & Co. KG, Germany). Each sample was attached to a metal jig with the coronal side facing downwards. The metal jig had an adjustable central hole which was made slightly bigger than the greater canal diameter to provide support for the root slice and to allow for unrestricted movement of the dislodged obturation material (Fig. 2). Compressive force was applied to the obturation material through a flat metal rod (0.5, 0.7 and 1.0 mm in diameter for the apical, middle and coronal slices respectively) attached to a load cell and moving downwards at a crosshead speed of 1 mm/min. The metal rod had a clearance of at least 0.2 mm from the
Table 1 – The materials used in this study and their manufacturers’ details.

<table>
<thead>
<tr>
<th>Group</th>
<th>Smear layer</th>
<th>Material</th>
<th>Manufacturer</th>
</tr>
</thead>
<tbody>
<tr>
<td>1A</td>
<td>Preserved</td>
<td>ProRoot MTA</td>
<td>Dentsply Maillefer, Ballaigues, Switzerland</td>
</tr>
<tr>
<td>1B</td>
<td>Removed</td>
<td></td>
<td></td>
</tr>
<tr>
<td>2A</td>
<td>Preserved</td>
<td>Harvard MTA</td>
<td>Harvard Dental International GmbH, Hoppegarten, Germany</td>
</tr>
<tr>
<td>2B</td>
<td>Removed</td>
<td></td>
<td></td>
</tr>
<tr>
<td>3A</td>
<td>Preserved</td>
<td>Biodentine</td>
<td>Septodont, Saint Maur des Fosses, France</td>
</tr>
<tr>
<td>3B</td>
<td>Removed</td>
<td></td>
<td></td>
</tr>
<tr>
<td>4A</td>
<td>Preserved</td>
<td>Gutta percha</td>
<td>Obtura Spartan, Fenton, MO, USA</td>
</tr>
<tr>
<td>4B</td>
<td>Removed</td>
<td>AH-plus sealer</td>
<td>Dentsply DeTrey, Konstanz, Germany</td>
</tr>
</tbody>
</table>

Fig. 1 – Each obturated root was sectioned at 3 different levels (a). The lesser and greater canal diameters and the height were measured for each root slice.

Fig. 2 – Push-out bond strength test diagram.

Statistical analysis of the mean push-out bond strength using the one-way ANOVA test revealed no statistically significant differences at level of section (i.e.: coronal, middle and apical)
within each group. Therefore the values for all thirds within one group were combined to increase the power of the statistical test.

The mean and standard deviation values of the push-out bond strength of each group are presented in Table 2. Statistical analysis using the two-way ANOVA and Tukey post hoc tests revealed that the push-out bond strength is significantly influenced by the obturation material used and smear layer removal/preservation \( (P < 0.01) \). In the CSC groups (1A, 1B, 2A, 2B, 3A and 3B), the push-out bond strength was significantly reduced following removal of the smear layer (Fig. 3). In the gutta percha and sealer groups, no statistically significant difference was detected in the push-out bond strength between the removed and preserved smear layer subgroups (2.09 and 1.98 MPa respectively). Roots filled with Biodentine exhibited the highest push-out bond strength which was significantly higher than all other groups whether smear layer was removed or preserved (7.71 and 8.79 MPa, respectively). Gutta percha and sealer demonstrated the lowest adhesion with root canal dentin which was not significantly influenced by the removal of smear layer. There was no statistically significant difference in the push-out bond strength between the ProRoot MTA and Harvard MTA in both the preserved (ProRoot MTA: 7.54, Harvard MTA: 7.64 MPa) and removed (ProRoot MTA: 6.58, Harvard MTA: 6.47 MPa) smear layer groups \( (P = 1) \).

**Table 2 – The mean and standard deviation push-out strength values of each group.**

<table>
<thead>
<tr>
<th>Group (n = 30)</th>
<th>Mean (MPa)</th>
<th>St. deviation</th>
</tr>
</thead>
<tbody>
<tr>
<td>1A</td>
<td>7.54</td>
<td>1.11</td>
</tr>
<tr>
<td>1B</td>
<td>6.58</td>
<td>1.13</td>
</tr>
<tr>
<td>2A</td>
<td>7.64</td>
<td>1.08</td>
</tr>
<tr>
<td>2B</td>
<td>6.47</td>
<td>1.08</td>
</tr>
<tr>
<td>3A</td>
<td>8.79</td>
<td>1.55</td>
</tr>
<tr>
<td>3B</td>
<td>7.71</td>
<td>1.81</td>
</tr>
<tr>
<td>4A</td>
<td>1.98</td>
<td>0.48</td>
</tr>
<tr>
<td>4B</td>
<td>2.09</td>
<td>0.51</td>
</tr>
</tbody>
</table>

Examination of the roots slices under optical microscope (40×) revealed adhesive bond failure in the majority of the CSC groups except for 1 sample in the ProRoot MTA groups, 3 samples in the Harvard MTA groups and 3 samples in the Biodentine groups where cohesive and combined failure occurred. In the gutta percha and sealer groups, adhesive bond failure occurred in all the specimens. The null hypotheses were rejected according to these results (Fig. 4).

SEM images clearly demonstrated the presence/absence of smear layer in their respective groups (Fig. 5). Better adaptation was observed between the MTA and root canal dentin when the smear layer was preserved.

### 4. Discussion

The creation of a good seal is one of the major requirements of root canal obturation materials. The bond with radicular dentin depends in part on the type of material used. CSC release calcium hydroxide during their hydration setting reaction \([13]\). In the presence of tissue fluid, an interfacial layer resembling hydroxyapatite in structure and composition is formed between root canal dentin and the CSC \([13, 14]\). A recent study showed the formation of intra-tubular tags in conjunction with an interfacial mineral interaction layer referred to as the “mineral infiltration zone” \([15]\). The results from these studies suggest the formation of a chemical bond between the CSC and radicular dentin \([14]\).

Root canal instrumentation produces a layer of organic and inorganic material known as smear layer. It is a 1–5 μm-thick layer made of dentin shavings, necrotic pulp remnants, bacteria and their by-products \([16–18]\). Smear layer removal prior to obturation of the pulp space remains a controversial issue. On one hand, it is a loosely adherent layer that can provide a pathway for microbial micro-leakage \([19]\), it potentially harbors bacteria and can serve as a reservoir of irritants \([20]\), it can provide a substrate for any remaining bacteria following chemo-mechanical disinfection of the pulp space \([21]\), and can prevent the penetration of irrigation solutions and inter-appointment medication into the dentinal tubules, thus jeopardizing the effective disinfection during root canal treatment \([22]\). On the other hand, the smear layer can block the dentinal tubules and alter their permeability which can limit bacterial and toxin penetration \([23]\). Furthermore, bacteria surviving the disinfection protocol can be entombed within the dentinal tubules by the smear layer and the obturation material \([24]\).

The adhesion of obturation materials to root canal dentin can be influenced by the presence/absence of smear layer \([25]\). Zinc oxide eugenol sealers have been shown to fail to penetrate the dentinal tubules when smear layer was preserved \([16]\). Plastic filling materials (pHEMA and silicone) \([26]\) and sealers penetrated the dentinal tubules to depths of 40–60 μm after removal of smear layer \([27]\). Micro-leakage studies demonstrated improved sealing ability of gutta percha and sealer when smear layer was removed \([28–30]\). This was explained by the improved penetration of sealers into the dentinal tubules in the absence of smear layer.

The apical sealing ability of CSC, on the other hand, was significantly reduced after smear layer removal \([31–33]\). This

![Fig. 3 – The mean and standard deviation push-out bond strength values for each group. The P values show the difference in push-out strength between each two groups of the same obturation material.](image-url)
was attributed mainly to the inability of the CSC particles to penetrate the dentinal tubules due to their larger particle size (2.44–3.05 μm) [34] compared with dentinal tubules (0.9–2.5 μm) [35]. A recent in vitro study used a scanning electron microscope demonstrated that ProRoot MTA failed to penetrate the dentinal tubules at any level [36]. Therefore, removal of smear layer results in poor adaptation of the CSC to dentin walls, and consequently, reduced bond strength to dentin.

The effect of smear layer on the bond strength between CSC and dentin has not been sufficiently investigated. The push-out test has been demonstrated to be a reliable method of assessing bond strength to root canal dentin [37]. In our study, three CSCs were investigated in addition to gutta percha and AH-plus sealer. The samples were stored for one week before the mechanical testing to allow for the hydration of the dicalcium silicate constituent in the silicate cements, which is responsible for the increase in compressive strength over the subsequent week following mixing [38]. The effect of smear layer removal on the push-out bond strength was investigated in the four material groups. The push-out bond strength values in our study are within the range of other recently published papers [39–41]. White ProRoot MTA was reported to have a push-out bond strength that ranged from 4.8 MPa, 5.68–9.46 MPa [40] and 5.95–7.88 MPa [41]. The push-out values for Angelus MTA (Angelus Dental Industry Products, Londrina, Brazil) were erroneously higher and ranged from 99.60–118.95 MPa [42]. Therefore, comparison of the push-out bond strength of different CSC was not possible due to the different methodologies used in previous studies.

Analysis of the push-out bond strengths revealed no statistically significant difference between the different thirds within each group as the dislodgement forces were directly proportional to the adhesion surface areas. Our results showed a consistent decrease in the push-out bond strength of the three CSC groups when the smear layer was removed. It seems that the smear layer is important in the formation of the interfacial layer and possibly gets actively involved in the

Fig. 4 – Modes of bond failure in the calcium silicate groups: adhesive (a and b), cohesive (c) and combined (d). In the gutta percha and sealer groups adhesive bond failure was detected (e) with sealer tags remained attached to the dentin walls (f).

Fig. 5 – Scanning electron microscopy images of root slices filled with ProRoot MTA where the smear layer was preserved (a) and removed (b). The images suggest better adaptation between the MTA and dentin when the smear layer is preserved.
mineral interaction between the CSC and radicular dentin. In the gutta percha and AH-plus sealer groups, sealer remnants/tags were detected on the dentin walls in most of the samples following the push-out test. While this observation needs to be confirmed by SEM analysis, it suggests that the bond failure occurred at the gutta percha/sealer interface. Therefore, whether smear layer removal improved the bond strength between the sealer and the radicular dentin could not be detected by this test.

The results of this study could have a clinical significance as root filling materials should resist displacement when they are indirectly subjected to occlusal forces, which could reflect on their sealing ability. It is essential to reiterate that there are certain drawbacks for obturating the root canal space with CSCs as they are difficult to handle, extremely challenging to retrieve and while their placement is possible in straight wide root canals, it could be exceedingly demanding in curved narrow canals. Nevertheless, owing to their bioactivity and excellent sealing abilities, CSCs have a definite place in dentistry and their use as obturation materials merits further research.

5. Conclusion

Within the limitations of this ex vivo study, it can be concluded that smear layer removal is detrimental to the bond strength between calcium silicate cements and root canal dentin.

References


