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Effect of placement technique on the push-out bond strength of calcium-silicate based cements

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The purpose of this study was to evaluate the effects of ultrasonic and manual placement techniques on the push-out bond strength of Biodentine and MTA with and without calcium chloride. One hundred and twenty mid-root slices from forty freshly extracted single-rooted human mandibular premolar teeth were instrumented and randomly divided into six groups (n=20) according to the filling material and placement technique applied, as follows: G1: MTA-manual compaction, G2: Biodentine-manual compaction, G3: MTA+5% $CaCl_2$-manual compaction, G4: MTA-ultrasonic activation, G5: Biodentine-ultrasonic activation, G6: MTA+5% $CaCl_2$-ultrasonic activation. The push-out bond strengths were measured using an Instron testing machine. Data were analyzed using two-way analysis of variance (ANOVA) with Bonferroni correction. The ultrasonic activation significantly enhanced the bond strength values of the materials. Biodentine presented higher bond strength values than that of MTA groups. The addition of $CaCl_2$ to MTA did not improve the bond strength of the material.

Keywords: Biodentine, Bond strength, Calcium chloride, MTA, Ultrasonic activation

INTRODUCTION

Mineral trioxide aggregate (MTA), a calcium silicate-based cement (CSC) is widely used for perforation repair, root-end filling, pulpotomy, apexification and regenerative procedures[1]. MTA possesses several desirable properties such as superior sealing ability, biocompatibility, and the ability to set in the presence of fluids[1]. However, MTA presents some notable shortcomings such as difficult handling property and long setting time[1]. In recent studies, the addition of calcium chloride ($CaCl_2$) to MTA was proposed in order to improve its physicochemical properties and showed that the addition of $CaCl_2$ to MTA at the concentrations of 2 to 15% reduced the setting time of the material[2,3]. It was also shown that MTA mixed with 5% $CaCl_2$ had similar cell viability compared with MTA mixed with water[4]. Biodentine (Septodont, Saint Maur des Fosses, France) is a more recent CSC with improved physical properties and reduced setting time as compared to MTA[5]. Biodentine powder is mainly composed of tricalcium silicate, calcium carbonate (filler material) and zirconium oxide (radiopacifier), whilst the liquid supplied for mixing with the cement powder consists of $CaCl_2$ (used as a setting accelerator) and a hydrosoluble polymer (water-reducing/superplasticizing agent)[6]. This biomaterial is considered as a biocompatible and bioactive dentine substitute and has been indicated for coronal and radicular restorations[6].

The physical and chemical properties of CSCs can be affected by several factors such as the ratio of the constituent components, exposure to various clinical environments, storage conditions, mixing and placement technique[7,8]. Few studies have examined the effects of placement techniques on the properties of MTA-like materials[8-12]. Ultrasonic vibration, applied to an endodontic condenser is a placement technique to improve the flow, settling, and compaction of the material and is perceived to be a useful adjunct[9]. Matt *et al.* reported that apical barriers placed with ultrasonic activation demonstrated fewer voids than barriers placed without ultrasonic energy[10]. Furthermore, ultrasonic activation has been reported to enhance the surface microhardness[8], compressive strength[11], and sealing ability[12] of MTA. It was recently suggested that ultrasonic activation might also improve the adhesion of dental materials to the cavity walls[13].

Endodontic materials should be resistant to dislocating forces such as functional pressure or condensation. Different methods are used to evaluate the adhesion of dental materials to dentin including the push-out strength, tensile bond strength, and shear bond strength tests[14]. To date, no study has evaluated the effect of placement technique on the bond strength of MTA and Biodentine. Therefore, the purpose of this study was to determine the influence of ultrasonic and manual placement techniques on the push-out bond strength of Biodentine or MTA with and without calcium chloride.

MATERIALS AND METHODS

Specimen preparation

Forty freshly extracted human mandibular premolar teeth were selected following the University ethics committee approval (Ethics Board No: GO-15/702). The teeth were decoronated at the cementoenamel junction and the apical part of the roots were removed,

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leaving 5-mm root blocks. The lumen of the roots was instrumented using Gates Glidden burs (Dentsply Maillefer, Ballaigues, Switzerland) with the size of #2–5 to obtain a standardized cavity diameter of 1.3 mm. The roots were embedded in acrylic resin cylinders using self-cured acrylic resin (Melio Dent, Bayer Dental, UK). One hundred and twenty 1-mm-thick root sections were prepared (3 slices per specimen) using a water-cooled diamond blade on a cutting machine (Isomet, Buhler, Lake Bluff, NY, USA) under running water. The slices were then immersed in 17% ethylenediaminetetraacetic acid (EDTA) for 1 min, followed by the immersion in 1% sodium hypochlorite (NaOCl) for the same period of time. After that, they were immediately washed in distilled water and dried.

Experimental procedures

The root slices were randomly assigned into six groups (n=20) according to the filling material used and the placement technique applied as shown in Table 1. MTA and Biodentine were mixed according to the manufacturer's instructions. MTA+$CaCl_2$ was prepared in a 3:1 (powder:liquid) ratio by mixing MTA powder with 5% $CaCl_2$ (prepared as a solution by dissolving $CaCl_2$ in distilled water at a 5% concentration). Materials were placed into the lumen of the root canal in each slice with a MTA Endo gun (Dentsply Maillefer). In manual compaction groups, the specimens were obturated using a stainless steel endodontic plugger (Dentsply Maillefer) while for the ultrasonic activation groups specimens were obturated with a 2-s indirect ultrasonic activation against the endodontic plugger. A piezoelectric unit (Pmax, Satelec, Merignac, France) was used at the medium-power setting with an ultrasonic tip CPR-1 (Dentsply Tulsa Dental). To prevent extrusion of the material, the root slices were placed on glass slabs. Excess material was trimmed from the surface of the specimens using a scalpel. The obturation procedures were performed by a single operator in order to avoid inter-operator discrepancies. The specimens were stored in 100% humidity at 37°C for 96 h.

Push-out test

The bond strength of the materials was determined using a Universal Testing Machine (Instron, Model 1334, Instron, Canton, MA, USA). The specimens were

Table 1 Groups, materials used to fill the cavities and placement techniques

Groups	Material	Placement technique
G1	MTA[a]	Manual compaction
G2	Biodentine[b]	Manual compaction
G3	MTA[a]+$CaCl_2$[c]	Manual compaction
G4	MTA[a]	Ultrasonic activation
G5	Biodentine[b]	Ultrasonic activation
G6	MTA[a]+$CaCl_2$[c]	Ultrasonic activation

[a] Angelus, Londrina, PR, Brazil
[b] Septodont, Saint Maur des Fosses, France
[c] Sigma-Aldrich, St. Louis, MO, USA

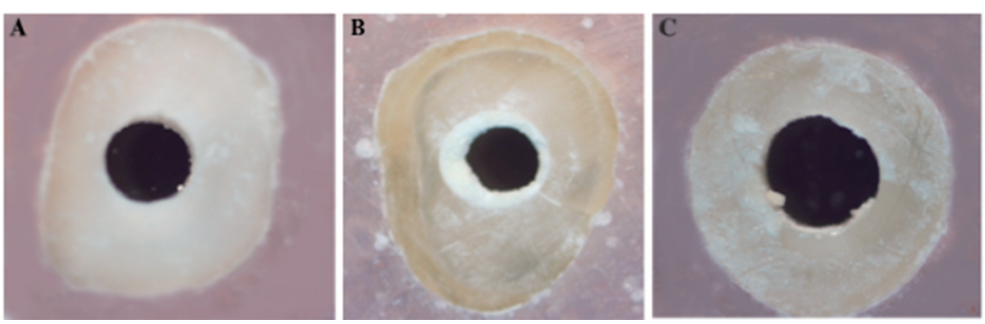

Fig. 1 Inspection of the samples under a stereomicroscope at ×10 magnification and various failure modes.
(A) Adhesive failure; note the clean canal wall. (B) Cohesive failure within the material. (C) Mixed failure; there are remnants of the material inside the canal.

placed on a metal slab with a 1.5-mm central hole. A cylindrical stainless steel plunger of 1-mm diameter and operating at a speed of 1 mm min^{-1} was used to apply force on materials inside root slices. The load applied to the material at the time of displacement was recorded in Newton. The recorded values were then converted to megapascals (MPa) using the following formula: Load/(2 prh), where p is the constant, r is the root canal radius, and h is the thickness of the root slice in millimeters. The nature of the bond failure was assessed under a stereomicroscope (Leica MZ16 A, Leica Microsystems, Wetzlar, Germany) at ×10 magnification. Each sample was categorized into 1 of the 3 failure modes: adhesive failure at test material and dentin interface, cohesive failure within test material, or mixed failure (Figs. 1A–C). Data were analyzed using a two-way analysis of variance (ANOVA) with Bonferroni correction using SPSS software version 21 (SPSS, Chicago, IL, USA). The level of significance was set at p=0.05.

SEM Analysis

One specimen from each group was used to characterize the microstructural surface morphology. The specimens were sputtered with gold and imaged using a scanning electron microscope (EVO 50, Carl Zeiss, Oberkochen, Germany) at ×10,000 magnification.

RESULTS

Table 2 shows the mean values and standard deviations of the push-out bond strength (MPa) and the distribution of failures of all groups. The bond strength values in the ultrasonic placement groups were significantly higher than the hand placement groups (p<0.05). Regardless of the placement technique used, Biodentine presented significantly the highest bond strength values (p<0.05) while no significant difference was found between MTA and MTA+$CaCl_2$ (p>0.05). Inspection of the samples revealed the bond failure to be predominantly adhesive for MTA groups and cohesive for Biodentine groups. The placement technique did not have a significant effect on the failure types of the tested materials (Table 2). In SEM examination, MTA groups showed variable sizes of globular structures and cubic crystals whereas needle-shaped crystals and honeycomb-shaped structures were mainly observed in Biodentine groups. Black areas were interpreted as pores and microchannels between particles (Fig. 2).

DISCUSSION

An ideal endodontic biomaterial should adhere to the cavity walls and resist dislodging forces to help maintain the integrity of the root filling–dentine interface either under static conditions or during function and operative procedures[15,16]. To assess this property *in vitro*, the push-out test has been shown to be efficient and reliable as the test conditions are comparable with the clinical situation in which the tested materials are placed directly into prepared canals with a natural canal shape and tubule arrangement[16]. Furthermore, the test's loading closely simulates clinical stresses as the applied load is perpendicular to the dentinal tubules[17]. Therefore, the push-out test allows accurate specimen standardization[18] and generates fewer stresses at the bonding interface during sample preparation than conventional tensile and shear bond testing[19]. On the other hand, it has been stated that the results of a push-out test can be affected by some variables such as specimen orientation, different root canal diameters and different plunger sizes[17]. To overcome these limitations, we sectioned the roots, then prepared the canal spaces with the same drills and did the filling procedure for the push-out test. This approach allowed to have straight sections with standardized canal diameter that were perpendicular to the applied load. In addition, the plunger used in the present study was selected according to the canal diameter, because a plunger size 70 to 90% of the canal diameter was reported not to affect the bond strength[17].

The effect of placement technique on the push-out bond strength of CSCs was evaluated in the present study. The results revealed that ultrasonically placed materials had higher push-out strength values than

Table 2 Summary statistics of push-out bond strength in the experimental groups (n=20)

Groups	Material	Placement technique	Mean (Mpa)±SD	Failure mode% (A/C/M)
G1	MTA	Manual compaction	6.82±2.13^{a}	53/22/25
G2	Biodentine	Manual compaction	9.40±3.04^{b}	10/60/30
G3	MTA+$CaCl_2$	Manual compaction	5.30±2.32^{a}	64/17/19
G4	MTA	Ultrasonic activation	8.50±2.30^{c}	50/23/27
G5	Biodentine	Ultrasonic activation	11.12±2.66^{d}	9/63/28
G6	MTA+$CaCl_2$	Ultrasonic activation	7.72±3.42^{c}	60/17/23

A, adhesive failure along the material-dentin interface; C, cohesive failure within the material; M, mixed failure. Different letters indicate statistically significant differences among groups (p<0.05).

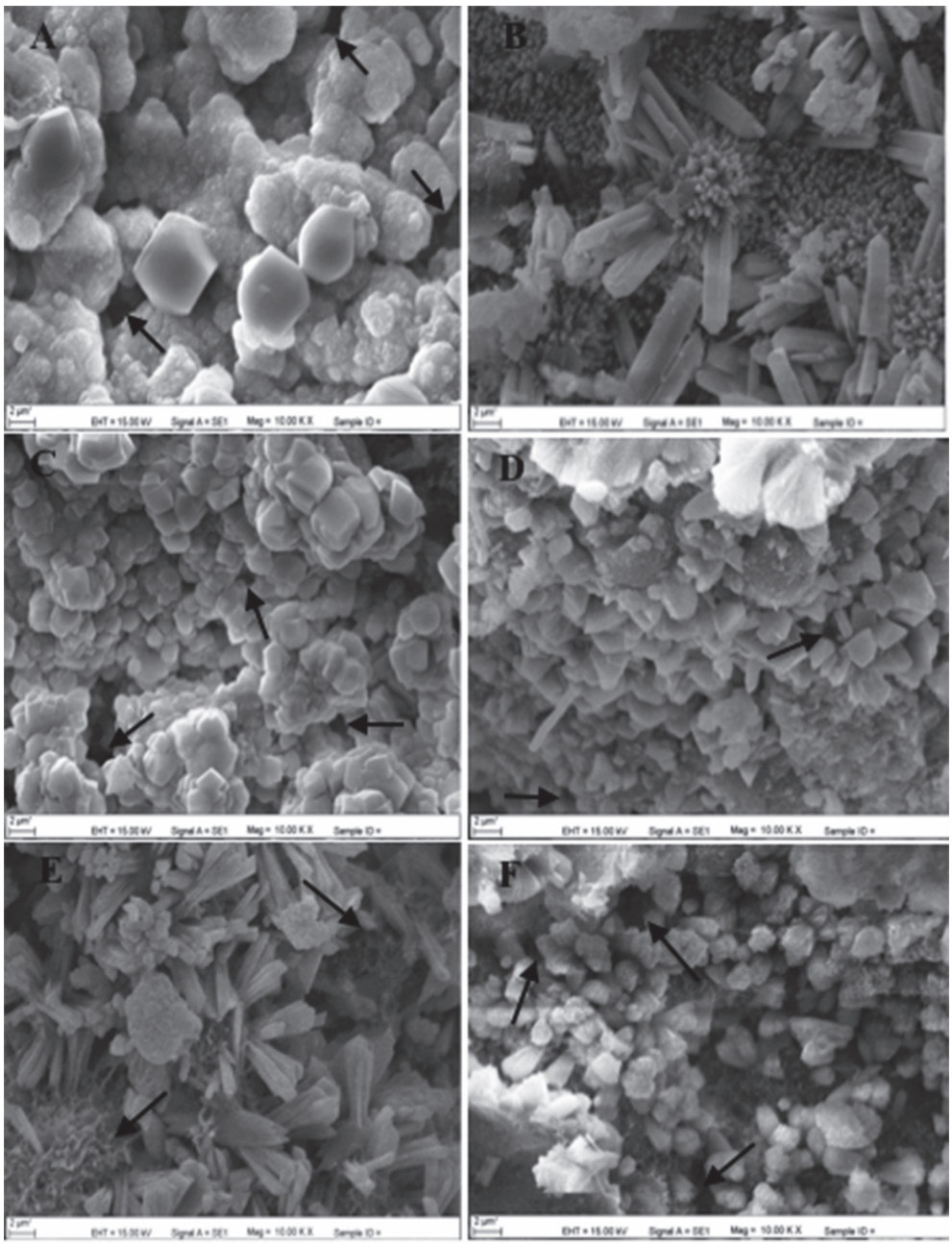

Fig. 2 Representative SEM images of each group (×10,000).
(A) View of globular structures and cubic crystals with microchannels (arrows) in G1. (B) Needle-shaped crystals that appear firmly attached to the underlying surface of the material in G2. (C) A cluster of cubic crystals with microchannels (arrows) in G3. (D) An amorphous surface containing globular and cubic crystals in G4. (E) Needle-shaped crystals with honeycomb-shaped structures (arrows) in G5. (F) Globular and cubic crystals with microchannels (arrows) in G6.

those placed manually. One explanation for this result may be the different fill density of materials after the placement procedure. Although the fill density of the materials was not analyzed in the present study, a previous study showed that one second of indirect ultrasonic activation resulted in an MTA fill that was significantly heavier and denser than hand condensation[20]. However, it has also been suggested that excessive ultrasonic activation of MTA may show poorer physical characteristics because ultrasonication may incorporate air into the MTA and produce a fill less dense and less uniform than that produced by hand

compaction[20]. Similarly, in another study the effects of ultrasonication time on MTA were analyzed showing that a time of two seconds of ultrasonication per increment provided improvement in material properties[21]. Based on this finding, in the present study two seconds of indirect ultrasonic activation was applied which may have contributed to higher bond strength results by providing a denser mass of tested materials. Ultrasonic activation may also improve the penetration of the material to dentine tubules by allowing the material to flow. Recently, the technique of MTA placement using ultrasonic vibration in association with smear layer removal was found to improve the marginal adaptation of MTA[22]. The irrigation protocol of the present study allowed the smear layer removal and with the ultrasonic vibration, the materials may have penetrated to open dentine tubules more efficiently, leading to improved bond strength.

In the present study, regardless of the placement technique used, Biodentine presented significantly higher bond strength values when compared with both MTA groups. This result is in agreement with previous studies[23,24]. The higher bond strength values of Biodentine may result from its smaller particle size, which has potential to enhance the penetration of the cement into the open dentinal tubules, leading to improved bond strength[24]. This effect might be further reinforced with more prominent biomineralization ability of Biodentine than MTA, which leads to increased micromechanical retention through formation of dentinal bridges as a result of crystal growth within the dentinal tubules[25]. Although the results of the present study did not reveal significant differences between MTA groups, there is a tendency of reduction on bond strength values presented by MTA when mixed with 5% $CaCl_2$. Similarly, a previous study found that the addition of $CaCl_2$ to MTA negatively influenced the bond strength[26]. The lower bond strength can be explained by the less expansion of the material due to acceleration of the setting time[26,27]. Furthermore, the immediate contact of MTA+$CaCl_2$ with moisture may have altered the powder-liquid ratio and reduced the cohesive strength between the cement particles, negatively influencing the bond strength to dentine[26,28]. However, addition of $CaCl_2$ may improve the biomineralization ability of MTA, leading to improvement in the bond strength after a period of time[29]. Further research is necessary to establish the long-term effects of addition of $CaCl_2$ on MTA.

The bond failures observed in MTA groups were predominantly at the MTA-dentin interface (adhesive type). This finding is in agreement with previous studies[14,23] that showed the MTA-dentin bond failures were usually adhesive. The adhesive mode of failure may be related to the short storage time of the tested materials before the evaluation of bond strength, which was 4 days in the present study. It was shown that an adherent interfacial hydroxyapatite-like layer at the dentin wall is produced when teeth filled with MTA stored in synthetic tissue fluid for 2 months[30]. Hachmeister *et al.* suggested that the formation of this layer leads to enhanced attachment of dentin to MTA over time[12]. In contrast to MTA groups, Biodentine samples presented predominantly cohesive mode of failure in the present study. Similar findings were also reported in a recent study[23]. The different failure types of MTA and Biodentine may be explained by the particle size of these materials, which affects the penetration of material into dentinal tubules. A smaller particle size and uniform components of Biodentine might have resulted in a better penetration to dentin, which finally caused cohesive failure inside the cement. According to our SEM examinations, Biodentine had particles that appear firmly attached to the underlying surface of the material while MTA presented many pores and microchannels between its particles. These findings may also explain why Biodentine and MTA showed different failure types.

Based on this study, the ultrasonic placement of CSCs can be considered the preferred method to increase the bond strength against displacement forces caused by functional stresses or by the condensation of restorative and root-filling materials. Biodentine showed more resistance to dislodgement forces as compared to MTA. Despite the lower bond strength values, the addition of $CaCl_2$ did not statistically affect the push-out bond strength of MTA.

CONFLICT OF INTEREST

The authors deny any conflicts of interest related to this study.

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