

Push-out bond strength of MTA with antiwashout gel or resins

L. M. Formosa¹, B. Mallia¹ & J. Camilleri²

¹Department of Metallurgy and Materials Engineering, Faculty of Engineering, University of Malta, Msida; and ²Department of Restorative Dentistry, Faculty of Dental Surgery, University of Malta, Msida, Malta

Abstract

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Aim Assessment of the push-out bond strength of four MTA-based formulations for use as root-end filling materials.

Methodology MTA Plus mixed with (i) water ('MTA-W'); (ii) a proprietary water-based antiwashout gel ('MTA-AW'); (iii) Superbond C&B chemically curing resin ('MTA-Chem'); and (iv) Heliobond light-curing resin ('MTA-Light') was tested. Root slices 3 mm thick human had a 1.5 mm diameter hole drilled centrally and were treated with 17% EDTA for 60s. Forty specimens divided into groups 1–4 were prepared and filled with MTA-W, MTA-AW, MTA-Chem and MTA-Light, respectively. Groups 3 and 4 were etched with 37% phosphoric acid for 60s, and bonding agent was applied to the dentine surface. Specimens were stored for 28 days in Hanks' Balanced Salt Solution at 37 °C. Push-out strength was tested with a punch and die (punch diameter

1.3 mm, die diameter 2.0 mm, punch speed 1 mm min⁻¹). Stereomicroscopy was used to classify failure mode (adhesive, cohesive or mixed type).

Results The resulting push-out strengths were 5.1 MPa (MTA-W), 4.3 MPa (MTA-AW), 4.7 MPa (MTA-Chem) and 11.0 MPa (MTA-Light). MTA-W had higher push-out strength than MTA-AW ($P = 0.022$). The same was noted for MTA-Light relative to the other materials ($P < 0.05$). All materials exhibited adequate push-out strengths compared with MTA-W. Failure was predominantly mixed, except for MTA-Chem (predominantly adhesive).

Conclusions All materials exhibited adequate push-out strength. Previous studies have shown the new formulations have additional advantages including increased washout resistance and faster setting time, making them promising for future dental applications.

Keywords: antiwashout, bond strength, composite resins, mineral trioxide aggregate, push out, root-end filling materials.

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Introduction

Mineral trioxide aggregate (MTA) is a dental cement with numerous applications including pulp capping, apexification, repair of root perforations, root-end filling and others (Torabinejad & Chivian 1999, Bogen

& Kuttler 2009, Parirokh & Torabinejad 2010). The main clinical limitations of this material are its long setting time (Gomes-Filho *et al.* 2009, Porter *et al.* 2010) and consequent risk of washout (Bortoluzzi *et al.* 2006), which refers to the tendency of freshly prepared cement paste to disintegrate upon contact with blood or other fluids (Wang *et al.* 2007). Washout can occur when rinsing an osteotomy site (Porter *et al.* 2010) resulting in a compromised root-end seal.

A number of approaches can be taken to minimize this problem, such as the use of washout-resistant MTA. MTA Plus (Avalon Biomed Inc. Bradenton, FL,

Correspondence: Prof. Josette Camilleri Ph.D., Department of Restorative Dentistry, Faculty of Dental Surgery, University of Malta, Medical School, Mater Dei Hospital, Msida MSD 2090, Malta (Tel.: 00356 2340 1174; e-mail: josette.camilleri@um.edu.mt).

USA) is supplied with either water or an antiwashout gel. MTA Plus powder is similar to ProRoot MTA, but it is ground finer (Camilleri *et al.* 2013). The antiwashout gel was shown to be a water-based solution containing minor quantities of silicon, potassium, calcium and chlorine. The presence of an organic additive could be inferred from the FT-IR plots. No crystalline phases were detected in the dried gel (Formosa *et al.* 2013b). The gel significantly reduced washout when compared to the same MTA mixed with water (Formosa *et al.* 2013a). MTA Plus mixed with antiwashout gel exhibited lower levels of calcium ions in solution and reduced fluid uptake in the early stages of reaction. The antiwashout gel reduced the setting time of the cement and enhanced the compressive strength (Formosa *et al.* 2013b).

Alternatively, incorporation of resin into MTA has been proposed as a way of reducing setting time, which may extend the material's clinical use (Gandolfi *et al.* 2011a). In this application, combinations of MTA with light-curing resin (Gomes-Filho *et al.* 2010, Gandolfi *et al.* 2011a,b) and chemical curing resin (Chung *et al.* 2011) have shown promising results, promoting remineralization, releasing calcium ions and producing an alkaline pH in physiological solution. Composites based on MTA Plus exhibited calcium ion release, alkalinizing pH and formation of apatite. These composites are recommended for applications where bioactivity is desirable but not critical, and only they have a significant advantage over MTA in some other aspect (Formosa *et al.* 2013c). The setting time of MTA with light-curing resin was analogous to the light exposure time, that is, 30–100 s (Gandolfi *et al.* 2011a), whilst for MTA mixed with chemical curing resin, the actual setting time was 11.2 min (Chung *et al.* 2011). The resin-modified materials are indicated for use as dressings over pulpotomies and as pulp-capping agents where the tooth can be etched and the reduction in setting time would be beneficial as the extended setting time could result in increased number of appointments and the risk of coronal leakage (Hong *et al.* 2010) and also reaction of unset MTA with materials used to temporize the tooth being treated (Camilleri 2011).

As leakage from the apical or coronal direction is a possible cause of root treatment failure (Madison & Wilcox 1988), an important factor for the success of various endodontic procedures is the marginal adaptation (Reyes-Carmona *et al.* 2010) and bond strength (Wennberg & Ørstavik 1990) of the material with dentine. A frequently used test for measuring

dislocation resistance and bond strength of a restorative material to dentine is the push-out test.

Push-out tests have been successfully conducted in the past on numerous brands of MTA under various environmental conditions (Frankenberger *et al.* 2000a, Gancedo-Caravia & Garcia-Barbero 2006, Reyes-Carmona *et al.* 2010, Saghiri *et al.* 2010, Shokouhinejad *et al.* 2010, Gunesser *et al.* 2013), giving push-out strength values in the region of 2.5–11.4 MPa. Resin-based sealers have also been studied. For instance, AH Plus has been shown to have push-out strength comparable to an MTA-based sealer (Assmann *et al.* 2012). However, no studies to date have evaluated the bond strength of antiwashout-type MTA or MTA-based resin composites to dentine.

The purpose of this study was to determine whether the novel MTA Plus-based formulations (i.e. MTA mixed with antiwashout gel, chemically curing resin or light-curing resin) bond to dentine as strongly as ordinary MTA Plus (mixed with water). The push-out strength of MTA root-end restorations using each of the four test materials after storage in physiological conditions for 28 days was used to assess the bond strength.

Materials and methods

The materials used in this study were based on MTA Plus (compounded by Prevest Denpro, Jammu, India for Avalon Biomed Inc. Bradenton, FL, USA) lot #2011022801, mixed with different liquids to obtain the following formulations:

- MTA-W: distilled water with a water-to-powder ratio of 0.35 by mass;
- MTA-AW: antiwashout gel (compounded by Prevest Denpro, Jammu, India for Avalon Biomed Inc. Bradenton, FL, USA) dosed by weight – 0.350 g gel per gram MTA Plus powder;
- MTA-Chem: chemical curing resin (Superbond C&B; Sun Medical, Shiga, Japan). The polymer powder and the MTA were dosed volumetrically using the scoops supplied in the Superbond kit. The proportions used are 0.4 mL (one scoop) clear L-type polymer, 0.136 g (8 drops) monomer and 0.01 g (2 drops) catalyst-V per 0.4 mL (one scoop) of MTA powder;
- MTA-Light: light-curing resin (Heliobond, Ivoclar-Vivadent, Schaan, Liechtenstein) filled with MTA Plus. The filler loading used was 1 g MTA per 0.3 g Heliobond.

Specimen preparation

Twenty single-rooted, extracted human teeth were selected and stored in sterile water until use. Each tooth was embedded in cold-curing resin (EpoFix, Struers, Ballerup, Denmark) and sectioned perpendicular to its long axis using a water-cooled diamond wafering disc (Buehler, Lake Buff, IL, USA) on a cutting machine (Struers Minitom, Ballerup, Denmark). Two 3 mm slices of mid-root dentin were obtained from each tooth. The root canal spaces were then drilled with a 1.5 mm hardened steel drill bit on a low-speed pillar drill (Fig. 1a) to obtain standardized cavities of 1.5 mm diameter. The sections were immersed in 17% EDTA (Glyde; Dentsply Maillefer,

Ballaigues, Switzerland) for 60 s and immediately rinsed out and dried with a pneumatic syringe. The specimens were then randomly divided into four groups ($n = 10$). In group 1 and group 2, MTA-W and MTA-AW, respectively, were mixed as described and placed directly inside the root canal space. Filling was carried out on a flat glass plate, and excess material was trimmed from the specimens with a plastic instrument.

Additional preparation steps were applied to groups 3 and 4 before filling. The specimens were etched with 37% phosphoric acid for 60 s and rinsed and dried. A thin layer of bonding agent (Heliobond; Ivoclar-Vivadent, Schaan, Liechtenstein) was applied to the internal surface of the cavity using a

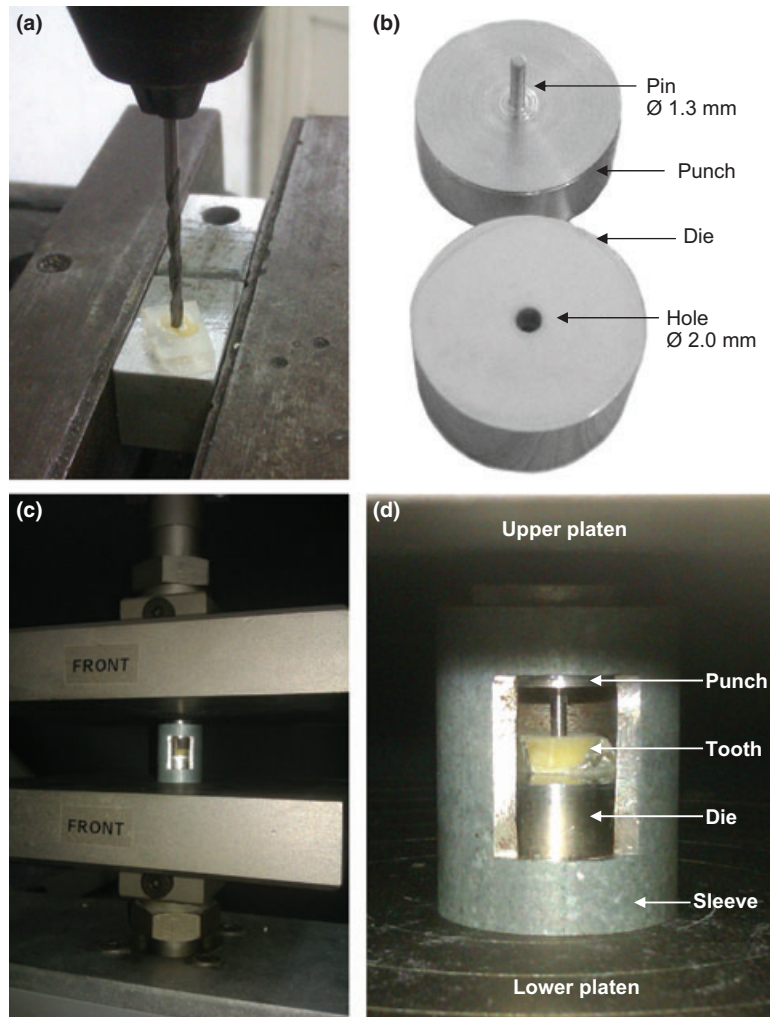


Figure 1 (a) Drilling tooth lumens to 1.5 mm diameter; (b) punch-and-die setup (sleeve not shown for clarity); (c) setup in universal testing machine; (d) close-up view of setup.

microbrush (Microbrush Intl., Waterford, Ireland) and light-cured for 30 s. MTA-Chem (group 3) or MTA-Light (group 4) was mixed as described and placed inside the cavity using the same method as for groups 1 and 2. No etch and bond was used in the control group (MTA-W) or in the MTA mixed with antiwashout gel because both these groups did not use resin-based systems.

All specimens were then immersed in Hanks' Balanced Salt Solution (HBSS; H6648, Sigma Aldrich, St. Louis, MO, USA) and stored for 28 days in an incubator at 37 °C.

Push-out test

The push-out bond strength of the various test materials was measured using a cylindrical punch-and-die set-up manufactured from an austenitic stainless steel rod. The punch has a 1.30 ± 0.05 mm diameter, and the die has a through hole of 2.00 ± 0.01 mm diameter (Fig. 1b). An external sleeve was used to keep the hold and die aligned (Fig. 1c,d). The filled tooth slice under test was placed on the die, and the centre of the test material filled cavity was visually aligned with the centre of the punch. The punch was pushed against the test specimen at a speed of 1.0 mm min^{-1} using a Testometric M350-10CT universal materials testing machine (Testometric Co. Ltd., Rochdale, UK), extruding the filling test material into the die. The maximum push-out force during the test was recorded. The shear strength of the bond (in MPa) was calculated according to the following formula: $F/(\pi \times d \times t)$, where F is the peak force in Newtons, d is the hole diameter in mm (nominally 1.5 mm), and t is the thickness of the tooth slice (nominally 3 mm, but measured accurately with a micrometre prior to loading).

The slices were examined under a stereomicroscope (Nikon, Tokyo, Japan) at variable magnification to determine the mode of the bond failure. Each sample was classified into one of four possible failure modes (Fig. 2). These were as follows:

1. adhesive failure occurring at the material-to-dentine interface, characterized by a clean intact extruded cylinder and no material deposits on the walls of the tooth after push-out testing;
2. cohesive failure occurring within the test material, characterized by heavy deposits of test material on the dentinal walls and a significantly eroded extruded cylinder following push out;
3. cohesive failure within the dentine, characterized by the extruded cylinder emerging with a pieces

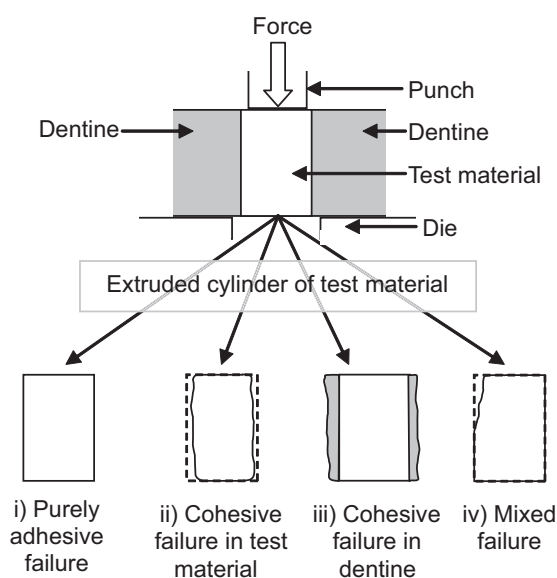


Figure 2 Characteristic features of the four possible failure modes. Dotted lines indicate boundary of original cement plug prior to push-out test. Shaded regions indicate dentine.

of dentine still attached, and a correspondingly enlarged and rough hole in the tooth following push-out testing; and

4. mixed-failure mode which is a combination of adhesive failure and cohesive failure within the test material, characterized by holes and extruded cylinders having a combination of regions with the characteristics described in (1) and (2).

Statistical analysis

The data were evaluated using SPSS (Statistical Package for the Social Sciences) software (PASW Statistics 18; SPSS Inc., Chicago Illinois, USA). Parametric tests were performed as K-S tests on the results indicated that the data were normally distributed. The data were analysed using one-way ANOVA and Tukey's post hoc tests with a 0.05 level of significance.

Results

The mean push-out bond strengths are shown in Table 1. MTA-AW gave significantly lower mean push-out strength than MTA-W ($P = 0.022$) and MTA-Light had significantly greater push-out strength than the other formulations tested ($P < 0.05$ in each case). All formulations except MTA-Chem exhibited

predominantly mixed-failure mode, whilst MTA-Chem exhibited predominantly adhesive failure (Fig. 3). The percentage occurrence of each failure mode is shown in Table 1. None of the specimens exhibited purely cohesive failure of either test material or dentine; moreover, the partial cohesive failures exhibited in the mixed-failure specimens were confined to cohesive failure of the test material and not of the dentine.

Discussion

Different formulations based on MTA were assessed in this study. The control MTA was mixed with water as

suggested by the manufacturer. This MTA has poor antiwashout characteristics (Formosa et al. 2013a,b). The antiwashout gel is a water-based solution containing minor quantities of chlorides and a water soluble polymer. These additives modify the behaviour of the freshly mixed material and also of the set MTA (Formosa et al. 2013b). The addition of antiwashout gel improved the washout resistance of the material (Formosa et al. 2013b). Such an improvement would enhance the properties of MTA used as root-end filling material. Two resin types were used to develop a resin-based MTA with the aim of reducing the setting time. These novel materials were aimed at

Table 1 Mean and median bond strength values (MPa) and Standard Deviation (SD) recorded for each material

Group	Formulation	Mean (MPa)	Median (MPa)	SD (MPa)	Failure Mode ^a	
					Adhesive (%)	Mixed ^b (%)
1	MTA-W	5.08	5.08	2.41	30.0	70.0
2	MTA-AW	4.34	4.02	1.16	30.0	70.0
3	MTA-Chem	4.68	4.84	1.26	55.6	44.4
4	MTA-Light	10.99	11.35	0.81	44.4	55.6

^aNo specimens failed by purely adhesive mode or by pure cohesive failure of dentine.

^bThis involved partial adhesive failure and partial cohesive failure within the test material.

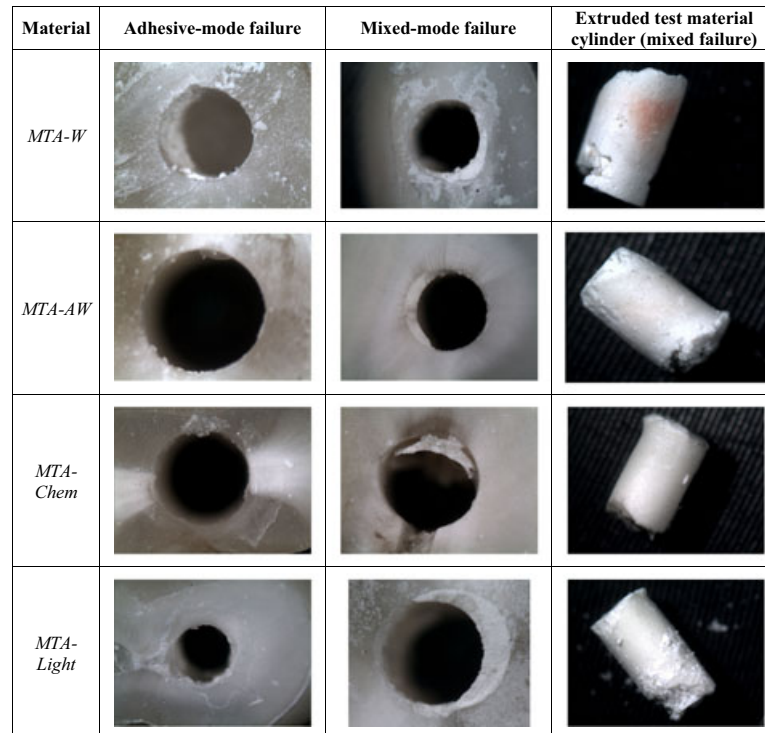


Figure 3 Stereomicrographs of typical samples from the four materials showing the cavity topography and extruded test materials following push out testing. The failure modes for the test materials investigated are indicated (all extruded cement fillings shown exhibit mixed-mode failure).

utilization of MTA for pulp capping, and as a dressing over pulpotomies where the setting time of the material is an important property as a long setting time would jeopardize the success of treatment. The light-curing variant (MTA-Light) was based on bisphenol-A-glycidylmethacrylate (Bis-GMA) and triethylene glycol dimethacrylate (TEGDMA) with MTA filler, whilst the MTA-Chem was composed of polymethyl methacrylate polymer, methyl-methacrylate monomer and partially pre-oxidized tributylborane in acetone as the catalyst and MTA filler (Formosa *et al.* 2013c).

Push-out tests have been shown to be effective and reliable as a means of assessing bond strength to dentine (Goracci *et al.* 2004). Micropushout testing was found to be superior to microtensile testing in the case of resin composites as microtensile testing involves the risk of premature failure (Cekic-Nagas *et al.* 2008). This study was designed to assess the resistance to dislodgement of MTA Plus, and three novel formulations based on it.

Mixing technique was shown to have an insignificant effect on push-out bond strength of white MTA (Shahi *et al.* 2012), and hence, conventional mixing was employed for all samples. However, a number of other factors have been shown to affect push-out strength. The presence of humidity (under conditions which result in an effective humidity level of 100%) has been shown to increase the push-out strength of MTA in root fillings (Gancedo-Caravia & Garcia-Barbero 2006). Irrigation of the root canal with NaOCl provided significantly higher push-out strength with MTA than samples treated with chlorhexidine gluconate (Hong *et al.* 2010). The presence of chlorhexidine gluconate reduced the MTA strength (Guneser *et al.* 2013). Conversely, for resin-based materials, post-etching treatment with NaOCl has been shown to decrease bond strength and marginal adaptation (Frankenberger *et al.* 2000a). To this effect, no NaOCl treatment was carried out on the teeth used in this study. For light-curing composite resins, push-out strength was found to increase whether pulse-delay or soft-start illumination methods were used (Cunha *et al.* 2008). However, a standard high-intensity illumination profile was adopted in this study to quantify the minimum (worst-case) bond strength that could be expected of the novel materials. Etching and application of bonding agent was only utilized for the resin-modified prototypes because resins bond by micromechanical retention. The dentine surface area available for bonding increases 156% after etching in the middle third of the root (Ferrari *et al.* 2000). The

increased surface area of the etched surface could have affected the bonding of MTA-W and MTA-AW. This factor is one of the limitations of the study. The mixing solution was not resin-based; thus, it was considered unnecessary to include etch and bond procedure for MTA-W and MTA-AW more so because these materials are indicated for use as root-end fillers. Furthermore, etching the root-end is a difficult procedure when performed during surgery. The bonding mechanism of MTA-based materials is different to that of resin-based materials. Whilst the etching enhances bonding by micromechanical retention, MTA-based materials bond by deposition of hydroxyapatite, which is deposited within collagen fibrils, triggering the formation of an interfacial layer with tag-like structures at the interface with the dentine (Reyes-Carmona *et al.* 2009).

Sections from the mid-root of single-rooted teeth were used. This was carried out to standardize the diameter of the cavity prepared in dentine as sections taken further apically would result in the need to overcut with the drill resulting in a specimen with a very small diameter, whilst preparing specimens taken cervically could result in a wider cavity. The orientation of the dentinal tubules and the effect of the bonding were not considered an issue. Although it has been demonstrated that the thickness of the hybrid layer depended on the density of tubules with a thinner hybrid layer formed where tubule density was low, research has shown that tubule density varies in different locations within the same tooth (Ferrari *et al.* 2000). Thus, location of the section within the tooth was not considered an important parameter affecting bonding thus affecting the results of the study.

Increased plunger diameter was shown to give higher apparent strengths (Nagas *et al.* 2011), although it did not affect the comparative rankings of the materials tested in that study. As no recognized standard for push-out testing exists, several plunger sizes have historically been used for push-out testing, from 0.7 mm (Huffman *et al.* 2009) up to 1.5 mm (Hong *et al.* 2010). In this study, a 1.5-mm drill bit was used to produce a cavity with parallel sides (as opposed to being tapered). This ensures that the force placed on the material/dentine interface is purely shear force and is also a requisite for the equation used for calculating bond strength to be valid as it is based on the assumption of constant diameter along the entire height of the cavity. The use of a pillar drill ensured that the axis of the drilled hole was exactly perpendicular to the bottom surface of the tooth.

Other variations in methodology exist, for instance with regard to the aligning method (visual or aided by casting the tooth specimen in a cylindrical resin mould), hole profile (parallel-sided or tapered), thickness of root slices and the ratio between plunger diameter and the diameter of the hole drilled in the tooth. In this study, a method similar to that reported by other researchers (Reyes-Carmona *et al.* 2010, Saghiri *et al.* 2010) was adopted (with the exception of using a 1.5-mm parallel-sided drill bit and 3-mm section thickness). Regardless of the differences in methodology, the mean values found for MTA Plus in this study are consistent with the range of values reported in previous studies (Frankenberger *et al.* 2000a, Gancedo-Caravia & Garcia-Barbero 2006, Reyes-Carmona *et al.* 2010, Saghiri *et al.* 2010, Shokouhinejad *et al.* 2010) for ProRoot MTA and MTA Angelus. A novel nano-modified MTA claimed to have 'similar composition to white (Angelus) MTA, but with very low particle size and high specific surface area of powder which may produce a faster and better hydration process' (Saghiri *et al.* 2013) has been reported to exhibit a push-out strength of 138.48 MPa, which is an order of magnitude higher.

The use of antiwashout gel instead of water resulted in a decrease in push-out strength from 5.08 MPa to 4.34 MPa, which might be attributed to the increased viscosity the gel imparts to the cement paste, which may affect marginal adaptation. Although this decrease is statistically significant, it is not drastic enough to preclude the use of washout-resistant MTA in cases where traditional water-based MTA is indicated. MTA-Chem exhibited a push-out strength that was not statistically different to that of MTA-W, even though the teeth were etched and a bonding agent was applied only for the former. Care was taken to avoid application mistakes when preparing samples. In particular, one study (Frankenberger *et al.* 2000b) reports that prolonged etching and excessive drying after conditioning were found to significantly decrease the bond strength of resin composites to dentine. In this study, a non-self-etching resin (Superbond C&B) was used, and the etching was performed separately in a previous step. This was carried out to avoid the potential risk of self-adhesive sealers failing to self-etch and thus giving poor performance in the case of incomplete smear-layer removal (Babb *et al.* 2009).

As explained, the bonding mechanism of the water-based materials (MTA-W and MTA-AW) is different to that of the resin-based materials (MTA-Chem and

MTA-Light). The high polymerization shrinkage of the resin causes it to pull away from the cavity walls, bringing about loss of adhesion. This explains the low strength observed and is supported by the fact that the failure mode of this material was predominantly adhesive. Qualitatively, similar results were reported in one study (Assmann *et al.* 2012) comparing the bond strength of an MTA/water-based sealer (Endo-CPM sealer) with an MTA/resin-based sealer (MTA Fillapex). The MTA/water-based sealer exhibited significantly higher strength than both the MTA/resin-based sealer and a purely epoxy resin-based sealer (AH Plus sealer).

MTA-Light on the other hand had more than twice the push-out strength of the other materials tested in this study. The resin used in this formulation (Heliobond) contains a high content of Bis-GMA – a high molecular weight monomer that helps minimize polymerization shrinkage (Garg & Garg 2010). This lower polymerization shrinkage compared with the resin used in MTA-Chem is the likely reason for the results observed. The high strength of MTA-Light, coupled with the fact that it has the shortest setting time (as reported in previous studies), makes MTA-Light the most promising of the novel materials tested.

More studies of the materials' chemical properties, particularly tests of their ability to promote remineralization, need to be undertaken. In addition, as the bond strengths of resin-based composites have been shown to decrease significantly over time periods of several years (Frankenberger *et al.* 2004), long-term studies of the novel materials are recommended.

Conclusion

Through the results of this study, it can be concluded that from the aspect of push-out bond strength, each of the novel materials appears to be adequate replacements for traditional water-based MTA, whilst previous research has shown them to possess additional advantages with regard to setting time and washout resistance. MTA mixed with light-curing resin proved to have the strongest bond to dentine. Further studies should be conducted to quantify other physical and chemical properties of these promising new materials.

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