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Evaluation of the dimensional changes of mineral trioxide aggregate sealer

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Abstract

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Aim To evaluate the setting time, early age restrained dimensional stability, fluid uptake, microstructure and porosity of a root canal sealer based on mineral trioxide aggregate (MTAS).

Methodology The MTAS, mineral trioxide aggregate (MTA) and a commercially available sealer pulp canal sealer (PCS) were investigated. The setting time of the materials was determined according to ISO 6876; 2002. The dimensional change in the vertical direction was measured over a period of 7 days from setting time using a linear variable differential transducer. The test samples were restrained in lateral directions by the metal mould. The fluid uptake of the cements was evaluated in Hank's balanced salt solution (HBSS), and their porosity was investigated using light optical microscopy.

Results The addition of a water-soluble polymer to MTA reduced its setting time but PCS displayed the shortest setting time ($P < 0.05$). The dimensional stability of the materials was not affected by the test environmental conditions ($P > 0.05$). PCS exhibited a much higher degree of shrinkage than MTA ($P = 0.997$, 0.640, 0.449, 0.191) and MTAS ($P = 0.952$, 0.523, 0.380, 0.149) at 3 h and 1, 3, 7 days, respectively, when allowed to set at 100% humidity. An increase in weight and expansion was recorded for MTA when immersed in HBSS. Microscope investigation of test specimens revealed the highest degree of porosity in MTA followed by MTAS and PCS.

Conclusions The novel sealer based on MTA demonstrated adequate setting time and was dimensionally stable. It has the potential to be used as root canal sealer cement in clinical practice.

Keywords: dimensional stability, fluid uptake, microscopy, mineral trioxide aggregate, porosity, pulp canal sealer, setting time.

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Introduction

Root canal sealers are used in combination with core materials such as Gutta-percha cones. The sealer binds the cones together and obliterates the irregularities between the canal wall and the core material. In combination with a bacteria-tight coronal restoration, such root fillings have been associated with success and a good prognosis (Ray & Trope 1995). In essence, the root canal is hermetically sealed from ingress of bacteria from the oral environment to the periapical tissues (Sundqvist & Figdor 1998). All the canal-filling techniques make use of a cement sealer to enhance the seal (El Deeb 1985, Hata *et al.* 1995). One of the requirements and characteristics of an ideal root canal sealer is dimensional stability and insolubility in oral and tissue fluids (Grossman *et al.* 1988). Dimensional changes (mainly shrinkage) of root canal sealers over time may introduce gaps along the sealer/dentine or sealer/Gutta-percha interface. In addition, sealers are in contact with periapical tissue fluid and if not dimensionally stable under these conditions, a lack of periapical seal can ensue.

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Sealers currently in use are subdivided into groups based on their constituents. These sealers include zinc oxide eugenol, calcium hydroxide, resin, silicone-based and glass–ionomer sealers. Zinc oxide eugenol sealers are based on Grossman's formula (Grossman 1958), which is a modification of the original Rickert's sealer (Rickert & Dixon 1931). More recently, sealers based on mineral trioxide aggregate (MTA) have been introduced. These sealers have been reported to have similar sealing properties to epoxy-resin-based sealers (Weller *et al.* 2008) and had a higher push-out strengths than AH Plus Jet (Dentsply DeTrey GmbH, Konstanz, Germany) or pulp canal sealer (SybronEndo Corporation, Orange, CA, USA) particularly after storage in simulated body fluid (Huffman *et al.* 2009) In addition, sealers based on MTA demonstrated apatite-like deposits in contact with physiological solutions (Weller *et al.* 2008, Gomes-Filho *et al.* 2009) and a biocompatibility similar to MTA (Gomes-Filho *et al.* 2009). A cement sealer could be developed by mixing MTA with adequate quantities of water-soluble polymer (Camilleri 2009).

The aim of this study was to evaluate the setting time, early age restrained dimensional stability, fluid uptake, microstructure and porosity of a root canal sealer based on mineral trioxide aggregate in simulated body fluid conditions.

Materials and methods

The materials used in this study include mineral trioxide aggregate (MTA White, Dentsply; Tulsa Dental Products, Tulsa, OK, USA), distilled water, water-soluble polymer (Degussa Construction Chemicals, Manchester, UK) and pulp canal sealer (PCS; Kerr-Hawe S.A., Bioggio, Switzerland). The various mixture proportions investigated are given in Table 1.

Determination of setting time

The setting time was determined in accordance with ISO 6876 (2002). A 100 g indenter with a needle attachment having a flat end of 2 mm diameter was used to determine the setting time. The cements were mixed, and the mixture was placed in an upright cylindrical metal mould measuring 10 mm internal diameter and 2 mm high. The surface was levelled with a metal spatula. After 120 s from the start of mixing, the mould was placed on a metal block measuring $8 \times 20 \times 10$ mm and the whole assembly was placed in an incubator at 37 °C and 100% humidity. The indenter was then lowered, and the resultant indentation on the cement surface was noted. This procedure was repeated at intervals. The setting time was established by measuring the time elapsed between the end of mixing up to when no indentation mark was visible to the naked eye after the indentation test. The test was repeated three times for each material tested, and the mean was determined.

Determination of vertical (horizontally restrained) dimensional change

The experimental set-up for the determination of dimensional change in the vertical direction is shown in Fig. 1. Preliminary investigations were carried out on the test rig to determine experimental errors. A test to determine the vertical dimensional change was conducted on a metal block. This was to ensure that no dimensional changes were recorded throughout the testing period at constant temperature. The experimental set-up (Fig. 1) included a metal base plate to which was affixed a metal mould 5 mm in diameter and 10 mm high. The moulds were coated with a thin layer of mould oil (Separol, Sika, Switzerland). The test materials were prepared and then compacted inside the mould in three incremental stages. The surface of the freshly overfilled mould was flattened with the flat end of a spatula. Five replicates were prepared for each material tested. Prior to testing, the set-up was left to acclimatize at 37 °C. The dimensional changes of the materials were investigated at 37 °C and both at 100% humidity and simulated body fluid (Hank's balanced salt solution; HBSS H6648; Sigma Aldrich, St. Louis, MO, USA) conditions. The composition of the HBSS was

Table 1 Mixture properties for the materials investigated in the study

Name	Mixture	Water/powder ratio	Polymer/cement
MTA	MTA and distilled water	0.3	–
MTAS	MTA, distilled water, water-soluble polymer	0.3	20 μL g^{-1}
PCS	ZnO powder and eugenol liquid	–	–

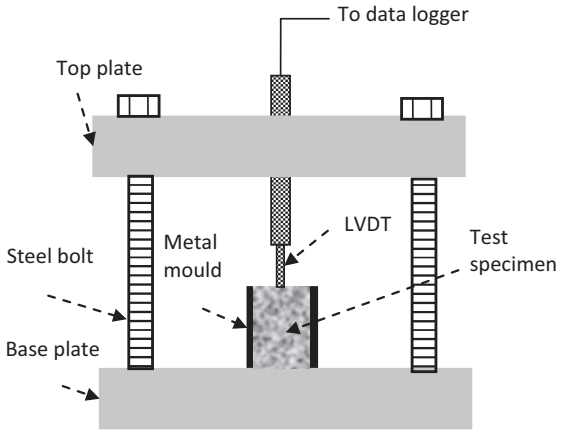

Figure 1 Experimental set-up for vertical dimensional change determination.

0.4 KCl, 0.06 KH_2PO_4 anhydrous, 0.35 $NaHCO_3$, 8.0 NaCl, 0.05 Na_2HPO_4 anhydrous and 1.0 D-glucose (g L^{-1}). The experimental set-up was placed inside a climatic chamber (Weiss-Gallenkamp, Loughborough, UK). At the end of setting time (calculated for each material according to ISO 6876; 2002), the contact probe of the linear variable differential transducer (LVDT Messotron; Messotron Hennig GmbH & Co, Seeheim-Jugenheim, Germany) was placed in contact with the cement surface. The LVDT was connected to a data logger (Peekel Instruments, Rotterdam, The Netherlands), and the probe position was recorded once every 15 min for 7 days.

Determination of fluid uptake

The fluid uptake was measured for cement specimens measuring 10 mm in diameter and 1.5 ± 0.5 mm high. Following setting at 37 °C and 100% humidity, the specimens were removed from their respective moulds. Twelve samples were prepared for each cement type. Six of these specimens following setting were stored in HBSS and the remaining six in air at 37 °C and 100% humidity. The weight of the specimens was recorded on a balance to the accuracy of 0.001 g after 3, 24, 72 and 168 h. The fluid uptake was determined using Equation 1.

$$\text{Fluid Uptake} = \frac{W_x - W_o}{W_o}$$

where W_x is the weight of the material after x hours. W_o is the original weight of the material after setting.

Characterization of the material microstructure in simulated body fluid

The material microstructure was observed using a light optical microscope on rectangular specimens measuring 8 × 10 mm and 5 mm high. The specimens were prepared and then stored for 24 h at 37 °C and 100% humidity. The specimens were then demoulded and stored in HBSS for 7 days after which they were sectioned in half along their cross section with a microtome cutter (Struers, Willich, Germany). They were subsequently polished using finer grit of silicon carbide paper. Both cross-sectioned surfaces were mounted in cold-curing resin. Once set, the surfaces were ground and polished with progressively finer grit of silicon carbide paper (MetaServ rotary grinder; Metallurgical Services, Betchworth, UK). The specimen surfaces were viewed under a stereo microscope (Remet SMZ-2T, Bologna, Italy) and a light optical microscope (Nikon Optihot -100, Tokyo, Japan) at higher magnification. Images of the cement surfaces were captured with a digital single lens reflex camera (Leica DFC 290; Leica Microsystems, Danaher Corporation, Washington DC, USA).

Statistical analysis

The data were evaluated using SPSS (Statistical Package for the Social Sciences) software (SPSS Inc., Chicago Illinois, USA). Parametric tests were performed as the data were normally distributed. Analysis of variance (ANOVA) with $P = 0.05$ and Tukey *post hoc* test were used to perform multiple comparison tests.

Results

Determination of setting time

The resultant setting time for the three materials under study is shown in Fig. 2. Pulp canal sealer had the shortest setting time of all the materials tested ($P < 0.05$). The addition of water-based polymer to MTA resulted in a shorter setting time ($P < 0.05$).

Determination of vertical (horizontally restrained) dimensional change

The percentage linear dimensional change with time of the materials in different curing environments (100% humidity or soaked in HBSS) is shown in Fig. 3. In 100% humidity conditions, the test materials exhibiting

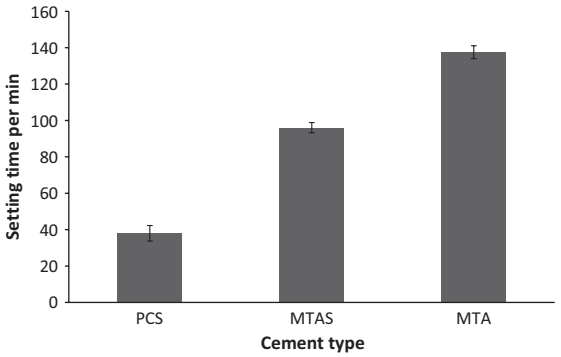

Figure 2 Means and standard deviation of the setting time of pulp canal sealer (PCS), MTA sealer (MTAS) and mineral trioxide aggregate (MTA) determined according to ISO 6876: 2002.

shrinkage with the pulp canal sealer displaying the most pronounced shrinkage ($P < 0.05$) for all assessment times. Specimen shrinkage increased gradually with time. There was no significant difference in dimensional stability of all materials tested when stored at either 100% humidity or soaked in HBSS ($P > 0.05$) over the 7-day period except for PCS at 3 h ($P = 0.378$).

Determination of fluid uptake

Fluid uptake values by specimens immersed in HBSS measured using a gravimetric method are shown in Fig. 4. All the materials tested displayed a reduction in weight when cured at 100% humidity over the 7-day period ($P > 0.05$). The reduction in weight increased with time. When immersed in HBSS, PCS demonstrated a reduction in weight ($P < 0.001$) at all time-points. This trend in weight reduction was similar to the PCS cured at 100% humidity at all time periods tested ($P > 0.05$). Both MTA and MTAS showed an incremental increase in weight when immersed in HBSS for 7 days. This increase in weight was consistent at all time periods monitored. MTA displayed a statistically significant higher fluid absorbance than MTAS ($P < 0.001$).

Characterization of the material microstructure in simulated body fluid

The materials microstructure after immersion for 7 days in Hank's balanced salt solution was viewed using a stereo and a light microscope. The micrographs are shown in Figs 5 and 6. The pulp canal sealer had a uniform surface speckled with homogenous distribution of shiny particles approximately 5 μm in diameter (Fig. 5a). It exhibited superficial cracks that were extended from one pore to another (Fig. 5b). The pores were generally round, up to 100 μm in size, and were evenly distributed in the microstructure. The MTAS contained porosity, which was evident at low and high magnifications (Fig. 5c,d). The bismuth oxide particles were interspersed within the cement matrix (Fig. 5c), and the individual cement particles were resolved at higher magnifications (Fig. 5d). The MTA displayed a higher volume of micropores evident at both low (Fig. 5e) and high magnifications (Fig. 5f). The stereo microscopy of the materials displayed differences in microstructure between the edges and the centre of the

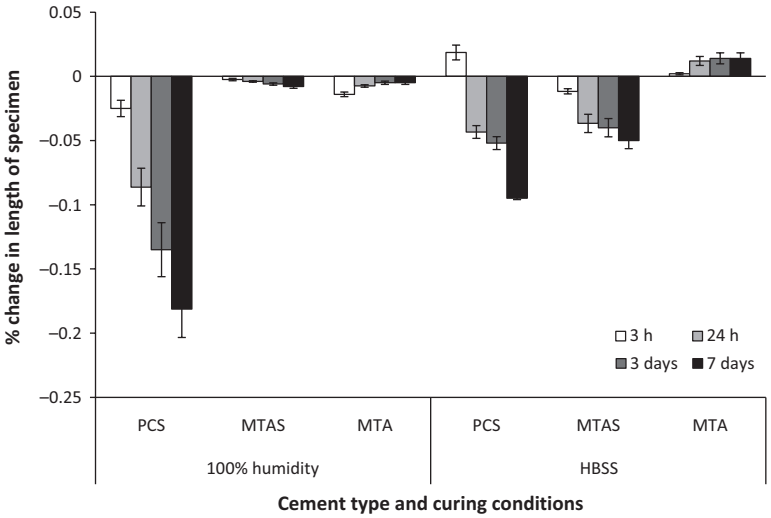

Figure 3 Mean percentage change in length (±SD) of pulp canal sealer (PCS), MTA sealer (MTAS) and mineral trioxide aggregate (MTA) stored at 37 °C and either at 100% humidity or in Hank's balanced salt solution for 7 days.

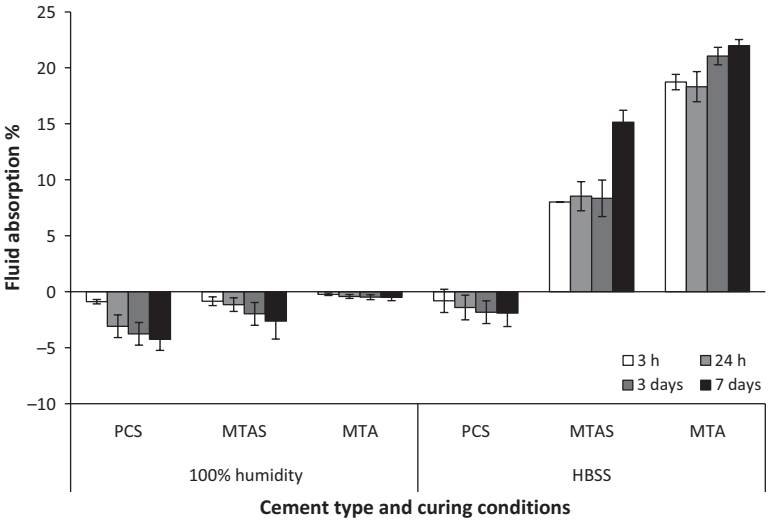

Figure 4 Mean percentage change in weight (±SD) of pulp canal sealer (PCS), MTA sealer (MTAS) and mineral trioxide aggregate (MTA) stored at 37 °C and either 100% humidity or in Hank's balanced salt solution for 7 days.

sectioned MTA sealer specimen (Fig. 6c,d, respectively). These differences were not evident in PCS (Fig. 6a,b) and MTA (Fig. 6e,f). The cement matrix was clearly distinguishable in the centre of the sectioned surface of MTAS. The morphology of the border was different exhibiting porosity. The radiopacifier was segregated along the outer skin of the MTAS.

Discussion

Dimensional stability is an important property for all dental materials. Shrinkage of cement sealers can lead to loss of marginal adaptation with resultant bacterial leakage. Expansion could result in fracture of fragile root-ends when the materials are used as root-end-filling materials.

The novel sealer was composed of MTA mixed with water and a water-soluble polymer. This material has already been investigated for flow and film thickness (Camilleri 2009) and hydration kinetics (Camilleri 2009), and also the sealing ability was assessed over a period of 28 days using the fluid filtration method (Camilleri *et al.* 2011). Pulp canal sealer was used as control in this study. This material was chosen because it had a powder and liquid formulation and has been used for sometime in clinical dentistry. In addition, pulp canal sealer has been used as a control in previous studies performed on this novel material (Camilleri 2010).

The dimensional stability of root-end-filling materials as suggested by ISO 6876 (2002) is measured on cast specimens (6 mm in diameter and 12 mm high) stored in water for a set period of time. The change in length of the specimens is recorded at different time intervals. Other specimen sizes have been used by different researchers (Chng *et al.* 2005, Islam *et al.* 2006, Wiltbank *et al.* 2007). The method for determining dimensional stability is simple but has several limitations. The main limitation is that dimensional changes are measured in only one direction. The instrument sensitivity (±1 μm) may not be enough to record the small changes occurring within the material. The use of a metal mould enables horizontal restraint against expansion of the specimen, thus dimensional changes are only permissible in the vertical direction. The use of a linear variable differential transducer (LVDT) allows accurate measurement in the vertical direction. This method has already been used to measure the hygroscopic linear setting expansions of MTA in a physiological solution (Storm *et al.* 2008, Gandolfi *et al.* 2009). In the current study, the materials were monitored over a period of 7 days rather than for 24 h as was reported in previous publications (Storm *et al.* 2008, Gandolfi *et al.* 2009).

Other researchers have used a combination of two LVDTs placing the materials in polytetrafluoroethylene ring moulds (Kanchanavasita *et al.* 1995) and also used LVDTs in combination with the ISO 6876 (2002) method (Ørstavik *et al.* 2001). The horizontal displacement with the ISO method was not accounted for, and the materials were unrestrained. In addition, the materials' lack of bulk strength posed limitations on the test (Ørstavik *et al.* 2001). Other methods reported for the evaluation of dimensional stability for root canal sealers include a volumetric method where the dimensional stability of sealers is determined by weighing

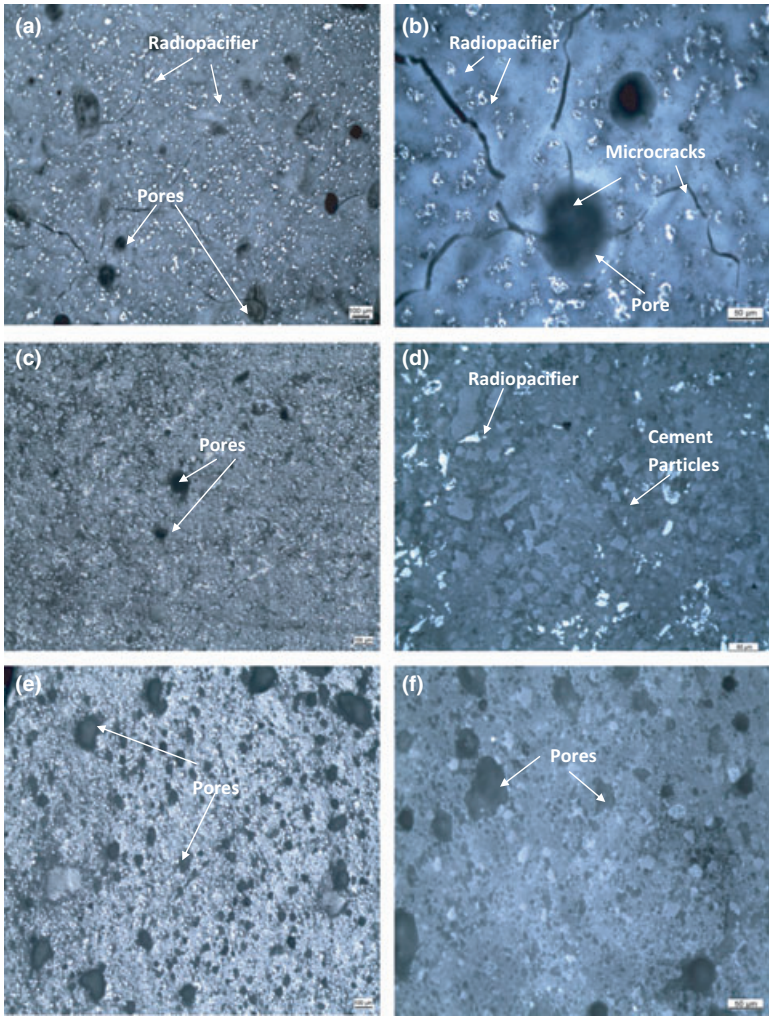

Figure 5 (a,b) Pulp canal sealer, (c,d) MTA sealer and (e,f) mineral trioxide aggregate polished sections viewed under the light microscope under (a, c, e) ×50 magnification and (b, d, f) ×200 magnification.

after injection in the internal walls of a glass pipette filled with water (Kazemi *et al.* 1993).

The experiments in this study were performed on specimens subjected to 100% humidity or immersed in a simulated body fluid. In other studies evaluating the dimensional stability of root canal sealers, water was used to store the specimens and the materials were tested in saturated and surface dry condition (Ørstavik *et al.* 2001). Research on the dimensional changes of MTA and related materials has utilized water and HBSS (Storm *et al.* 2008) and water, phosphate-buffered saline (PBS), 80% PBS and 20% foetal bovine serum and hexadecane (Gandolfi *et al.* 2009) for evaluation of linear restrained dimensional change. In the present study, the dimensional changes recorded in the vertical direction are related to fluid uptake and the measurement of porosity. The white MTA sustained a greater expansion in HBSS than in water (Storm *et al.* 2008). These findings were similar to the use of PBS where the expansion of MTA increased marginally on changing the soaking solution from water to PBS. The use of hexadecane resulted in material shrinkage (Gandolfi *et al.* 2009).

This study revealed that all the materials sustained shrinkage on curing at 100% humidity. This dimensional change was measured with the set-up shown in Fig. 1. Other researchers also reported a decrease in weight of MTA and related materials when immersed in hexadecane (Gandolfi *et al.* 2009). Light microscopy of the cements demonstrated porosity with varying size and morphology. MTA and the novel sealer exhibited porosity, which was different to that of pulp canal sealer. The system of pores in MTA and related materials was very likely interconnected. Although

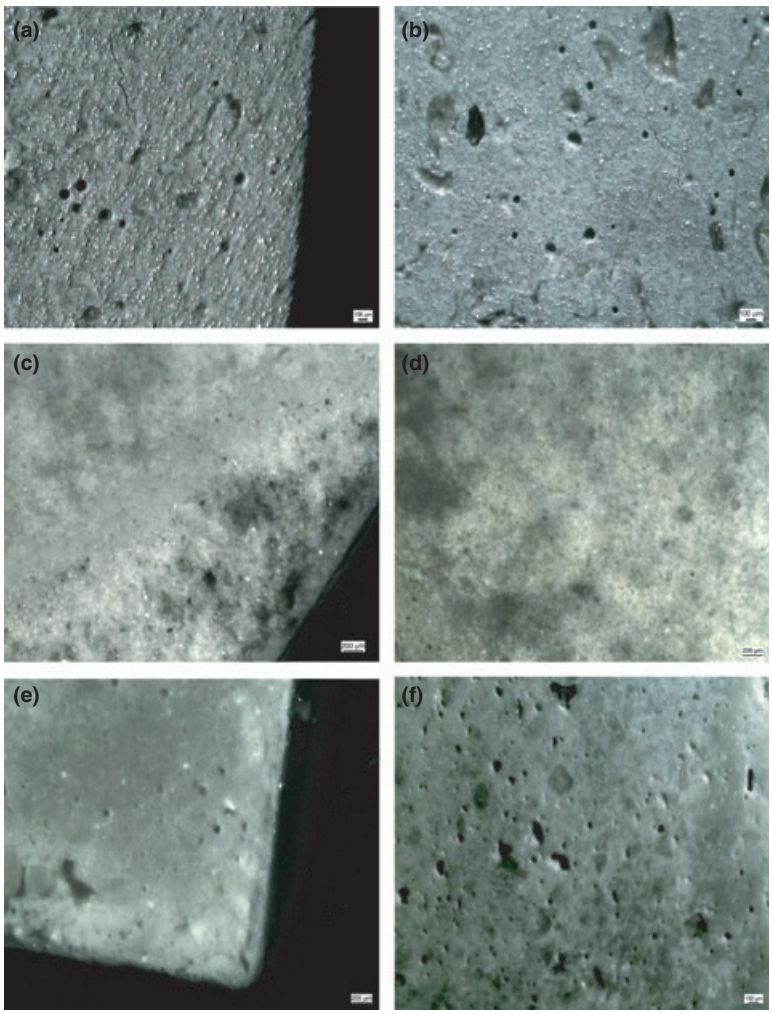

Figure 6 (a,b) Pulp canal sealer, (c,d) MTA sealer and (e,f) mineral trioxide aggregate polished sections viewed under the stereo microscope. (a, c, e) showing the materials edge and (b, d, f) the centre of the section (×18 magnification).

this could not be resolved using the light microscope, this feature is evident from the results of fluid uptake. The material could absorb fluids from the environment. It is postulated that this resulted in swelling, which was directly dependent on the amount of pores present. The PCS porosity was not interconnected and thus absorption was less. This porosity was also demonstrated under the scanning electron microscope (Camilleri *et al.* 2011).

Mineral trioxide aggregate soaked in HBSS had a linear expansion of 0.002% after 3 h, which increased over the 7-day period to 0.01%. These findings are similar to previous research which demonstrated an expansion when MTA is in contact with HBSS (Storm *et al.* 2008). The expansions recorded in the current study were lower than in the previous experiment. In the previous experiment, the cements were cured until they hardened at room temperature (Storm *et al.* 2008). The time taken for complete hardening was not recorded. In addition, the time of test was limited to 24 h while in the present study, the testing was concluded after 7 days and the experiment was commenced at the end of setting time for each material. A 0.7% change in length in cylinders 3 mm diameter and 6 mm high stored in water for 30 days was reported by others evaluating the dimensional stability of MTA (Wiltbank *et al.* 2007). Islam *et al.* (2006), Chng *et al.* (2005) described a 0.3% change in length upon setting using a similar method. More studies and an increase in conformity of testing are required to verify the changes occurring within the materials.

The novel sealer exhibited shrinkage both in humid and in soaked conditions. In HBSS, fluid uptake

resulting in increase in weight was recorded. However, the materials still exhibited shrinkage which increased over time. The stereo microscopy demonstrated evidence of material segregation in MTAS which was not evident in MTA. The borders of the cement block viewed under the stereo microscope had high porosity, and radiopacifier had settled at the borders rather than being evenly distributed in the cement matrix as in PCS. The MTA increased by 20% of its original weight after 7 days in HBSS compared to MTAS which had a lower increase in weight. The light microscopy revealed MTA to be more porous than MTAS. The high water absorption and porosity of MTA are in accordance with previous research (Fridland & Rosado 2003). The pulp canal sealer exhibited shrinkage and also a loss in weight over the 7-day period both at 100% humidity and in HBSS. This is in accordance with other research where zinc oxide eugenol sealers were reported to sustain shrinkage within the first few hours after setting (Kazemi *et al.* 1993). The shrinkage over a 4-week period testing using the ISO 6876 (2002) and LVDT was reported to be between 0.3% and 1%. This was similar to the shrinkage reported for PCS in the present study. The shrinkage was less when the material was soaked in HBSS. All the materials tested demonstrated a dimensional stability within the norms suggested by ISO 6876 (2002) for root canal sealer materials namely that the dimensional change in length should not exceed 1% in shrinkage and 0.1% in expansion. MTA sealer has been tested indirectly for dimensional changes by testing its dislocation resistance. The dislocation resistance was higher in wet conditions (Gancedo-Caravia & Garcia-Barbero 2006, Huffman *et al.* 2009), which implies that the material expands on wetting and is similar to the change reported in this study. The dislocation resistance of ProRoot Endo sealer was higher than that of pulp canal sealer and AH Plus (Huffman *et al.* 2009) again in accordance with the observations in this study. Immersion in phosphate-buffered saline increased the push-out strength of MTA and Portland cement. The biomineralization activity of MTA increased its resistance to displacement (Reyes-Carmona *et al.* 2010) in accordance with findings in the current study.

The setting time of the materials was more than 30 min and less than 72 h as classified in EN 6876; 2002 Section 4.3.3. For these materials, the measured setting time has to comply within the range set by the manufacturer. The setting time of MTA was similar to that reported in other studies (Torabinejad *et al.* 1995, Islam *et al.* 2006, Ber *et al.* 2007, Bortoluzzi *et al.* 2009, Camilleri 2010). In this experiment, the setting time of MTA was determined using a 100 g indenter as is specified in ISO 6876 (2002). The 100 g indenter as opposed to the 400 g indenter used for restorative materials can give different results owing to its lower weight. This indenter was used in this study as the materials under study were for use as root canal sealers.

Conclusions

The novel sealer based on MTA demonstrated adequate setting time, and its dimensional stability was within the limits suggested by ISO 6876. The material has the potential to be used as root canal sealer cement in clinical practice.

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