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The effect of condensation pressure on selected physical properties of mineral trioxide aggregate

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Abstract

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Aim To examine the effect of condensation pressure on surface hardness, microstructure and compressive strength of mineral trioxide aggregate (MTA).

Methodology White ProRoot MTA (Dentsply Tulsa Dental, Johnson City, TN, USA) was mixed and packed into cylindrical polycarbonate tubes. Six groups each of 10 specimens were subjected to pressures of 0.06, 0.44, 1.68, 3.22, 4.46 and 8.88 MPa respectively. The surface hardness of each specimen was measured using Vickers microhardness. Cylindrical specimens of 4 mm in diameter and 6 mm in height were prepared in polycarbonate cylindrical moulds for testing the compressive strength. Five groups of 10 specimens were prepared using pressures of 0.06, 0.44, 1.68, 3.22 or 4.46 MPa. Data were subjected to one-way ANOVA. The microstructure was analysed using a scanning electron microscope (SEM) after sectioning specimens with a scalpel.

Result A trend was observed for higher condensation pressures to produce lower surface hardness values. A condensation pressure of 8.88 MPa produced specimens with significantly lower values in terms of surface hardness than other groups ($P < 0.001$). A condensation pressure of 1.68 MPa conferred the maximum compressive strength; however, it was not statistically different. Higher condensation pressures resulted in fewer voids and microchannels when analysed with SEM. In specimens prepared with lower condensation pressures distinctive crystalline structures were observed. They tended to appear around microchannels.

Conclusion Condensation pressure may affect the strength and hardness of MTA. Use of controlled condensation pressure in sample preparation for future studies is suggested.

Keywords: compressive strength, condensation pressure, mineral trioxide aggregate, scanning electron microscopy, Vickers microhardness.

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Introduction

Mineral trioxide aggregate (MTA) was developed and introduced for use in endodontics for the repair of root perforations (Lee *et al.* 1993). Subsequently, it was recommended for use in surgical endodontics as a root-end filling material (Torabinejad *et al.* 1993, 1995a). It has also been used in vital pulp therapy (Eidelman *et al.* 2001, Holan *et al.* 2005) and as an apical barrier in treatment of immature teeth with nonvital pulps and open apices (Torabinejad & Chivian 1999, Shabahang & Torabinejad 2000). MTA also provides an effective seal against penetration of bacteria and their by-products (Tselnik *et al.* 2004), and has been recommended as a temporary filling material (Schmitt *et al.* 2001) and as a coronal plug after filling of the root canal system (Mah *et al.* 2003, Tselnik *et al.* 2004).

Several studies have shown that MTA is more biocompatible and less cytotoxic than other materials

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used for similar clinical applications (Torabinejad *et al.* 1995b, Mitchell *et al.* 1999, Moretton *et al.* 2000, Apaydin *et al.* 2004). However, the number of clinical trials undertaken using MTA is limited (Theodosopoulou & Niederman 2005).

MTA is a mixture of oxides and consists of fine hydrophilic particles that harden on contact with water (Torabinejad *et al.* 1995a, Fridland & Rosado 2003, Lee *et al.* 2004, Camilleri *et al.* 2005a). Hydration of the powder results in a colloidal gel that sets to a hard composition. The principle compounds involved are tricalcium silicate, dicalcium silicate, tricalcium aluminate and tetracalcium aluminoferrite (Islam *et al.* 2006). There are two types of MTA available: grey and white. Several authors have examined similarities between different types of MTA and Portland cement (Funteas *et al.* 2003, Menezes *et al.* 2004, Dammaschke *et al.* 2005). Camilleri *et al.* (2005b) showed that both white and grey MTA have a similar chemical constitution to Portland cement except for the addition of bismuth oxide to make it radiopaque (Camilleri *et al.* 2005a).

Although MTA has been recommended for a wide range of clinical applications, many properties of the material have not been investigated. For example, the effect of condensation pressure during placement on its physical properties is unknown. Characterization of MTA under standardized and controlled conditions should lead to an improved understanding of its behaviour and the optimization of its use in clinical practice.

In most previous studies (Torabinejad *et al.* 1993, 1995c, Hachmeister *et al.* 2002, Fridland & Rosado 2003, Lawley *et al.* 2004, Lee *et al.* 2004, Fridland & Rosado 2005, Walker *et al.* 2006) condensation pressure when placing MTA was an uncontrolled variable that could have affected its properties and performance. For instance, when used as a root-end filling it is likely that some forms of pressure will be applied during placement, both as it is packed into various carrying devices and also when aliquots of MTA are manipulated *in situ* in the root-end cavity. A similar situation would presumably exist for surgical repair of perforations. However, when MTA is used for nonsurgical repair of perforations, as an apical barrier in treatment of immature teeth with open apices or as a pulp-capping/pulpotomy material the condensation pressure is likely to be reduced considerably to prevent the material being forced into the periodontal ligament or pulp tissue.

Torabinejad *et al.* (1993) reported that the characteristics of hardened MTA were related to the water to powder ratio, the humidity around the material and the amount of air trapped in the mixture. To allow MTA to harden sufficient moisture must be present during the hydration process. Based on various clinical applications, moisture may be available from adjacent periodontal or pulpal tissues and from a damp cotton or sponge pellet that should be placed near the unset material. It is possible that the degree of condensation pressure may change the molecular distance between water molecules and MTA particles and change the space required for the hydration reaction, this in turn may change the optimum water to powder ratio (Bordallo *et al.* 2006). Condensation pressure may also have an effect on air trapped in the material and the number of inclusions (Bendtz 1997).

The aim of this study was to examine the effect of condensation pressure on the surface hardness, microstructure and compressive strength of MTA.

Materials and methods

The parameters investigated were surface hardness (Vickers microhardness), compressive strength and assessment of morphological characteristics using scanning electron microscopy (SEM). The material investigated was the tooth-coloured formula of ProRoot MTA with LOT number of 03081235 (Dentsply Tulsa Dental, Johnson City, TN, USA).

Microhardness test

The material was mixed according to the manufacturer's instructions. Each sachet containing 1 g of MTA was mixed with the recommended volume of water supplied by the manufacturer. The mixed material was weighed and then divided into four equal specimens that were packed into polycarbonate cylindrical tubes having an internal diameter of 6 mm and height of 12 mm.

Six groups of 10 specimens were prepared using condensation pressures of 0.06, 0.44, 1.68, 3.22, 4.46 or 8.88 MPa by varying the weight applied. The pressure on each specimen was applied for 1 min using a custom-made device containing a stainless steel piston with the similar internal diameter of polycarbonate cylindrical tubes (Fig. 1). The samples were thus subjected to a vertical force that was translated into a transverse and equally distributed pressure that compacted the MTA evenly into the cylindrical mould. A wet cotton pellet was placed onto MTA within the polycarbonate tube and each specimen placed in a glass

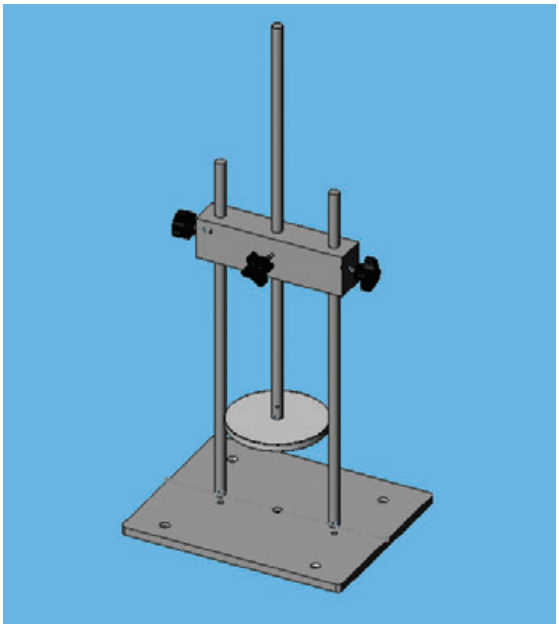

Figure 1 The custom-made device for application of condensation pressure on test specimens.

vial with 100% humidity for 4 days at room temperature (20°C). The MTA was then removed from the mould.

Both surfaces of the specimens were wet polished at room temperature using minimum hand pressure and silicon carbide-based sandpapers of varying particle size ('Wetordry™' 600-grit, 737 SF 'Tri-M-ite™' and 'Wetordry™' 1200-grit; 3M, St Paul, MN, USA) to provide smooth surfaces for ease of indentation testing. The polished specimens were cleaned gently under light pressure distilled water to remove surface debris. To prevent dissolution or water sorption the surfaces were dried gently by air spray. The Vickers hardness test of each specimen was performed using a Mitutoyo microhardness tester MVK G_1 (Mitutoyo Corp., Tokyo, Japan) and a square-based pyramid-shaped diamond indenter with a full load of 50 g for 5 s at room temperature that produced a quadrangular depression with two equal orthogonal diagonals in the polished surface of the cement. The angle between the opposite faces of the diamond indenter was 136°. Five indentations were made on the polished surface of each specimen at separated locations no closer than 1 mm to adjacent indentations or the specimen periphery. The diagonal of the resulting indention was measured immediately under the microscope and the Vickers hardness value displayed on the digital readout of the microhardness tester. The Vickers hardness (H_V) is calculated based on the following formula:

$$H_V = \frac{2F\sin(136°/2)}{d^2}, \quad H_V = 1.854\frac{F}{d^2} \text{ approximately}$$

where F = load (kg^{-1}) and d = the mean of the two diagonals of the impression made by the indenter in millimetres. The mean value of the hardness value obtained was calculated to determine the hardness value for each specimen. Differences between the experimental groups were analysed by one-way ANOVA.

Compressive strength

The same specimen preparation procedure was employed for testing the compressive strength. Cylindrical specimens of 4 mm in diameter and 6 mm in height (in accordance with ISO 4049:2000) were prepared in polycarbonate cylindrical moulds. The same custom-made device (Fig. 1) was used to condense the samples. Five groups of 10 specimens were prepared using pressures of 0.06, 0.44, 1.68, 3.22 or 4.46 MPa. The compressive strength test was conducted using a Lloyd LR MK1 machine (Lloyd Instruments, Fareham, UK). A flat steel rod was used at a crosshead speed of 1 mm min^{-1} whilst specimens were mounted vertically so that the compressive load was applied along the long axis of the specimen. The failure load was recorded and the compressive strength was calculated using the following equation:

$$CS(\sigma) = \frac{4P}{\pi d^2}$$

where P is the force (N) applied and d (mm) is the diameter of the specimen. The compressive strength of all specimens was recorded in MPa. The data obtained from compressive strength test were subjected to statistical analysis using one-way ANOVA.

Scanning electron microscopy

For the morphological evaluation by SEM, new specimens were prepared using the same set of pressures to condense the material and kept for 4 days under the same storage conditions. To analyse the microstructure of the inner surfaces, the specimens were sectioned into two halves by a disposable surgical scalpel blade No. 15. The surfaces were sputter-coated with gold using a Polaron Sputter Coater (Quorum Technologies, Newhaven, UK) and specimens were analysed with an EBT1 (Electron Beam Technology) Scanning Electron Micro-

scope (S.E.M. Tech Ltd, Woodbridge, UK). The micrograph images from the SEM analysis showing the qualitative internal microstructure of the set MTA prepared with different condensation pressures were evaluated in terms of the presence of microchannels and type of crystal formation.

Results

Microhardness

The results of the microhardness testing are shown in Fig. 2. Little difference occurred in mean surface hardness value up to a condensation pressure of 3.22 MPa. However, a condensation pressure of 8.88 MPa produced specimens with significantly lower values in terms of surface hardness than the other groups ($P < 0.001$). A condensation pressure of 3.22 MPa conferred the maximum hardness value; however, it was not significantly different from the other groups.

Compressive strength

The results of the compressive strength testing are shown in Fig. 3. Maximum compressive strengths occurred at a condensation pressure of 0.06 and 1.68 MPa. The lowest of compressive strength was related to a condensation pressure of 0.44 MPa. There was no statistically significant difference between the groups.

SEM analysis

The internal microstructure of all specimens that were prepared with various condensation pressures showed microchannels, depressions caused by air bubbles, pores, asymmetrical crystalline formation in the form of laminated cross-stratified structures, and bundles of jagged needle-like formations in a homogeneous matrix that resembled an epitaxial growth pattern (Figs 4–8). It was not possible to score each characteristic and thus compare them quantitatively between groups. However, the SEM images demonstrated that higher condensation pressures resulted in fewer voids created by entrapped air. In addition, fewer microchannels could be seen in specimens prepared with higher condensation pressures (Fig. 9). More typical crystalline structures were observed in specimens prepared with lower condensation pressures; they tended to appear around microchannels (Fig. 4).

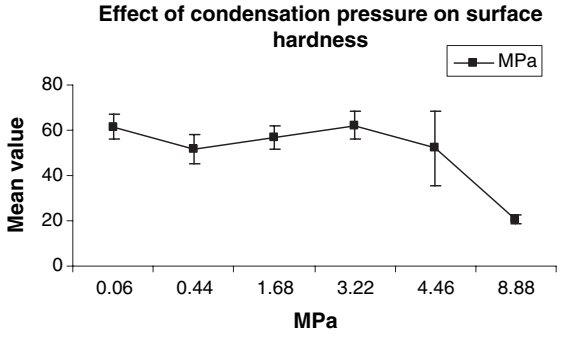

Figure 2 Mean surface hardness of specimens applied at various condensation pressures.

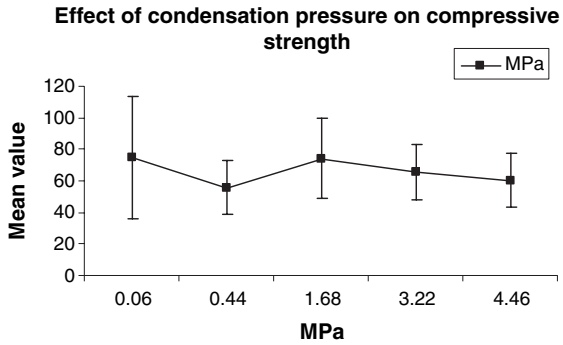

Figure 3 Mean compressive strength of specimens applied at various condensation pressures.

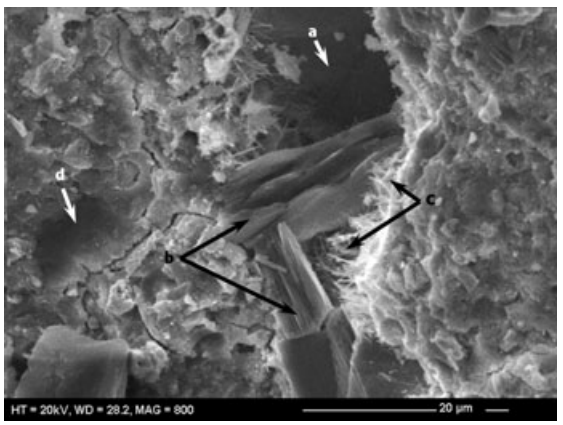

Figure 4 Scanning electron microscope image of a 3.22 MPa condensation force specimen. A cross-section of a microchannel can be seen (a), together with laminated (b) and needle-like (c) crystalline formation and a depression from an air bubble (d) (original magnification, ×800).

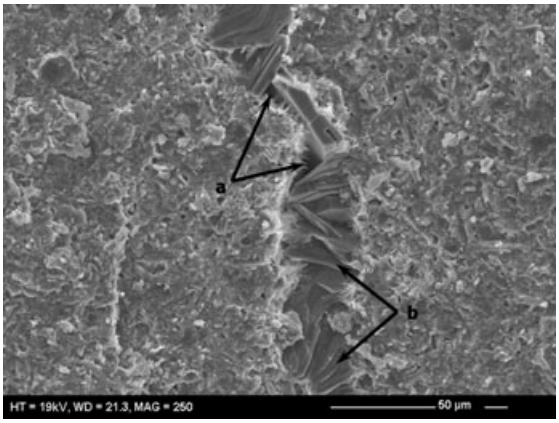

Figure 5 Scanning electron microscope image of a 0.06 MPa condensation force specimen showing a cross-section of a microchannel (a), and crystalline formation in the form of laminated cross-stratified structure (b) (original magnification, ×250).

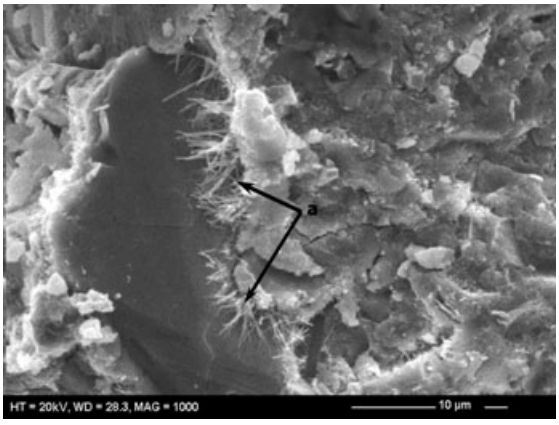

Figure 6 Scanning electron microscope image of a 0.06 MPa condensation force specimen. Bundles of jagged needle-like formations can be seen (a) (original magnification, ×1000).

Discussion

Hydraulic cements are finely ground materials that when mixed with water gradually or instantly set and harden either in air or in water. The reaction results in the formation of hydrated compounds whose strength increases with time. MTA is a type of hydraulic cement that can set in the presence of water. The initial setting time of MTA is approximately 4 h (Torabinejad *et al.* 1995a) and can create problems in terms of retention of the material in clinical situations (Lee 2000).

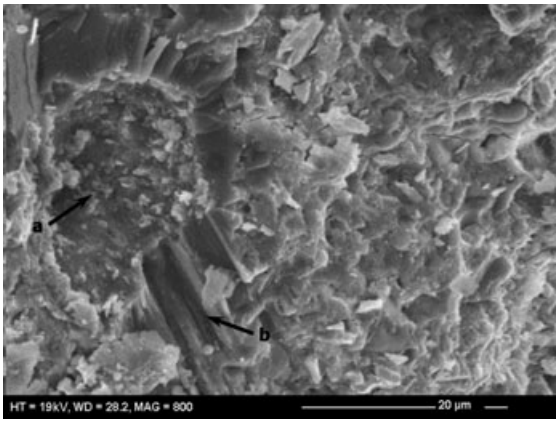

Figure 7 Scanning electron microscope image of a 1.67 MPa condensation force specimen. Cross-section of bundles of jagged needle like formations (a) can be seen together with a laminated crystalline structure (b). This structure resembles an epitaxially growth pattern.

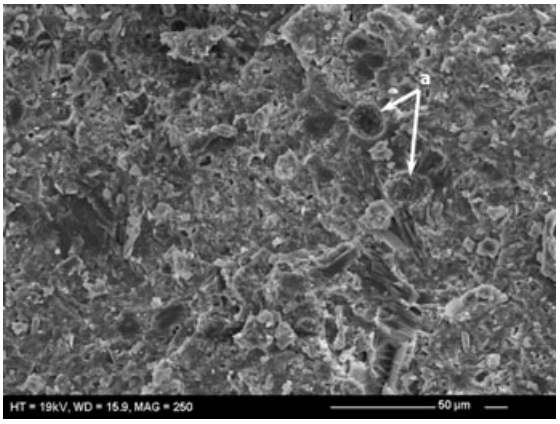

Figure 8 Scanning electron microscope image of a 0.06 MPa condensation force specimen. Depressions from an air bubble (a) can be seen (original magnification, ×250).

According to the United States Patent nos. 5 415 547 and 5 769 638 the basic constituent of MTA is Portland cement (Torabinejad & White 1995, 1998) and X-ray diffraction analysis has confirmed this similarity (Islam *et al.* 2006). The basic untreated materials of Portland cement are calcium oxide (CaO), silica (SiO_2), iron oxide (Fe_2O_3) and alumina (Al_2O_3). After grinding and heating of the raw materials, gypsum ($CaSO_4 \cdot 4H_2O$), which is the source of sulphate in the cement, is added to prolong its setting time. In fact, Dammaschke *et al.* (2005) reported that the quantity of gypsum in MTA is approximately half of that in the Portland cement.

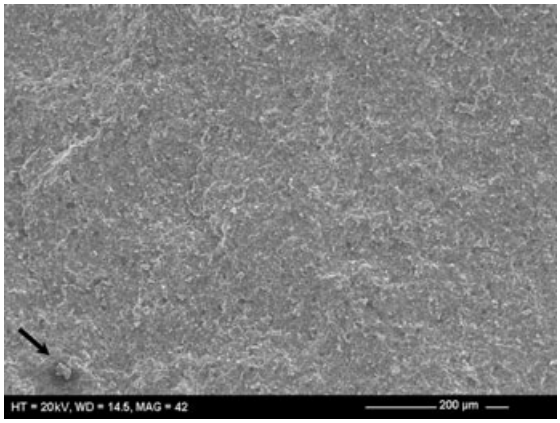

Figure 9 Scanning electron microscope image of a 4.46 MPa condensation force specimen. Greater condensation pressure resulted in fewer voids and microchannels. Some unidentified debris can be seen (arrow).

The powder of MTA is comprised of hydrophilic particles of tricalcium silicate ($3CaO\ SiO_2$), dicalcium silicate ($2CaO\ SiO_2$), tricalcium aluminate ($3CaO\ Al_2O_3$) and tetracalcium aluminoferrite ($4CaO\ Al_2Fe_2O_3$) (Schwartz *et al.* 1999, Lee *et al.* 2004, Dammaschke *et al.* 2005, Islam *et al.* 2006). Bismuth oxide (Bi_2O_3) is the only component of MTA that has not been traced in Portland cement (Funteas *et al.* 2003, Dammaschke *et al.* 2005, Islam *et al.* 2006).

The setting of Portland cement and thus MTA takes place in two stages. After mixing with water the hydration reaction of both silicates begins and results in the formation of a gel consisting of calcium silicate hydrates, which liberates calcium hydroxide. The calcium hydroxide then gradually reacts with the minerals to form other hydrated compounds. The calcium silicates contribute most to the binding power and strength of the material. It is also the main binding agent of crystalline calcium hydroxide that leaches most readily from the gel. Tricalcium aluminate exhibits flash setting on hydration. Tetracalcium aluminoferrite reacts at a slower rate than tricalcium aluminate (Eglinton 1987).

The characteristic of the resultant set material is likely to be dependent on various factors including water to powder ratio, temperature, environmental humidity and pH, entrapped air and water, the rate of packing and the condensation pressure applied (Torabinejad *et al.* 1995a, Fridland & Rosado 2003, Lawley *et al.* 2004, Lee *et al.* 2004, Fridland & Rosado 2005). Although a range of biological properties of MTA have been investigated, the effects of various clinical factors on the hydration process of MTA are not completely understood. Moreover, its physical and chemical properties have not been evaluated fully. Because of the various clinical applications of MTA such as direct pulp capping, sufficient strength is necessary to withstand condensation pressures applied to restorative materials. Compressive strength and surface hardness are indicators of the setting process and strength of the material (Bendtz 1997) and formed the basis of this investigation. In addition, in an attempt to evaluate its microstructure, a SEM evaluation was also carried out.

The effect of condensation pressure on the physical properties of MTA has not been reported. However, one of the operator variable factors when placing condensable materials is the condensation pressure used. In addition, although great emphasis has been placed on the effect of condensation pressure on the properties of other dental materials such as amalgam (Basker & Wilson 1968, Rakow *et al.* 1978, Ogura *et al.* 1983, Mahler & Nelson 1984, Lussi & Buergin 1987, Symons *et al.* 1987) there are no recommendations about the degree of condensation pressure required for MTA. For example, increased condensation pressure on dental amalgam results in removal of excess mercury and a faster setting reaction (Lussi & Buergin 1987, Lussi *et al.* 1995). The British Standards Institution (BS 2938) suggested applying a force of 10 pounds (4.5 kg) when amalgam test samples are prepared for clinical or laboratory investigations (Basker & Wilson 1968). Because of the lack of standardization for MTA in the field of dentistry, some studies follow the standards of endodontic sealers (Chng *et al.* 2005) with samples being prepared on the basis of the standards of restorative materials (Torabinejad *et al.* 1995a, Fridland & Rosado 2005, Danesh *et al.* 2006). In fact MTA is not a restorative material nor is it an endodontic sealer. It would seem to be essential that MTA, being a new and unique material with various clinical applications, should have its own standard. The standard testing techniques for Portland cement are not always applicable for MTA, even though they have been used (Chng *et al.* 2005, Dammaschke *et al.* 2005, Danesh *et al.* 2006).

In most of the laboratory and clinical studies on MTA, specimens were prepared in accordance with the manufacturer's instruction. On the other hand, in the manufacturer's instruction there is no information about the minimum or maximum condensation pressure that should be applied. In addition, there appears to be no other data on the optimum condensation pressure or the manipulation time of MTA. Therefore,

condensation pressure was an uncontrolled variable in most experimental studies reported to date (Torabinejad *et al.* 1995a, Eidelman *et al.* 2001, Aminoshariae *et al.* 2003, Camilleri *et al.* 2005a, Dammaschke *et al.* 2005, Walker *et al.* 2006, Yeung *et al.* 2006). Because of this lack of standardization and use of uncontrolled hand placement methods, the results obtained in some of these studies may be inconsistent. Conversely, temperature, gradual incorporation of water, methods of drying, humidity, water to powder ratio, size of samples, time, humidity and other environmental conditions have been considered (Camilleri *et al.* 2005a, Dammaschke *et al.* 2005, Walker *et al.* 2006).

In the present study to eliminate the effect of time of condensation as a confounding variable (Lussi *et al.* 1995), an application time of 1 min was adopted based on pilot studies. In order to apply even and equally distributed pressure on the specimens, a custom device was designed and constructed so that the diameter of the piston matched the internal diameter of cylindrical polycarbonate tubes. In this way the entire MTA surface was condensed and upward seeping of material prevented.

Based on the findings of this study, when greater pressures are applied to MTA during placement its surface hardness is reduced significantly (Fig. 2). Conversely, its maximum compressive strength occurred with the minimum pressure (Fig. 3). Therefore, in certain clinical situation, when condensation pressure should be controlled to prevent MTA from being extruded into the adjacent tissues, such as repair of perforations, placing apical barriers within immature apices and during direct pulp capping, the resultant material will be relatively strong.

In a laboratory study on ProRoot MTA and two Portland cements (Danesh *et al.* 2006) a vibration intensity of 6000 min^{-1} was used, to avoid air entrapment and to control confounding variables where samples were prepared for measurement of Vickers microhardness. But in the same study samples that were prepared for radiopacity and solubility were neither vibrated nor condensed with a controlled pressure; however, the molecular distance might have some effect on solubility and radiopacity. In an attempt to improve the placement and seal of MTA in immature root canals, Lawley *et al.* (2004) used ultrasonics. They compared this method with conventional hand condensation, but failed to control the condensation pressures applied. Clearly, use of controlled condensation pressure for the future studies is suggested.

In the present study it was anticipated that a greater condensation pressure would result in a harder material, although the result showed that when the condensation pressure was more than 3.22 MPa surface hardness was reduced (Fig. 2). This may occur because of insufficient intermolecular space for the ingress of water to hydrate the material adequately. In addition, SEM images demonstrated that higher condensation pressures were associated with fewer voids that could result in a less than optimal volume of intermolecular space with a negative effect on the hydration process (Fig. 9). Thus, applying a greater pressure in an attempt to achieve a harder material appears futile. The results also revealed that the condensation pressures applied during the preparation of samples did not have a statistically significant effect on compressive strength. Compressive strength is the capacity of a material to withstand axially directed pressure generating compressive stress as a result of compression force. It could be hypothesized that with the application of higher condensation pressures, the formation of microchannels was limited as a result of the material being more compact. It may also reduce the amount and the rate of water diffusion through the material that may impair the setting reaction resulting in a reduced compressive strength and surface hardness. The role of water molecules during the setting reaction is crucial. It does not only mix with powder, but it also chemically binds with various phases of the cement and has a direct effect on the setting process (Camilleri *et al.* 2005a, Santos *et al.* 2005, Walker *et al.* 2006). In other words, MTA hardens and gains strength as it hydrates, this process occurs rapidly at first and then slows down with time. When MTA powder is mixed with water, a special structure of microchannels is created (Figs 4 and 5). The continuity of microchannels is damaged during the setting process (Fridland & Rosado 2005). Therefore, the hardened cement presents pores and broken microchannels. The role of microchannels and pores during the hydration reaction is important; they provide pathways for the water to diffuse into the material and thus take part in the slow hydration process of the cement, when water becomes bound into the structure (Fridland & Rosado 2003, 2005). In the present study, specimens prepared with lower condensation pressures had more typical crystalline structures around microchannels. This might be related to better water diffusion and therefore a greater degree of hydration leading to well formed crystalline structures in the form of laminated cross-stratified and bundles of jagged needle-like formations (Figs 4–8). In

other words, a higher condensation pressure may pack the powder molecules closer together to produce a drop in surface hardness and a reduction in crystalline formation due to lack of sufficient space for water molecules.

Conclusion

Condensation pressure may affect the strength and hardness of MTA. A trend was observed for higher condensation pressures to produce lower surface hardness values. Use of controlled condensation pressure in sample preparation for future studies on ProRoot MTA is suggested.

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