International Endodontic Journal

doi:10.1111/j.1365-2591.2009.01610.x

Effect of acid-etching procedure on selected physical properties of mineral trioxide aggregate

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Abstract

Kayahan MB, Nekoofar MH, Kazandağ M, Canpolat C, Malkondu O, Kaptan F, Dummer PMH. Effect of acid-etching procedure on selected physical properties of mineral trioxide aggregate. *International Endodontic Journal* **42**, 1004–1014, 2009.

Aim To evaluate the effect of acid-etch procedures on the compressive strength and surface microhardness of tooth-coloured mineral trioxide aggregate (MTA).

Methodology White ProRoot MTA (Dentsply Tulsa Dental) was mixed and packed into cylindrical tubes of 4 mm in diameter and 6 mm in height. Three groups, each of 15 specimens were subjected to an acid-etch procedure either 4, 24 or 96 h after mixing. The compressive strength was measured and compared with unetched control groups. Differences between groups were analysed using the Kruskall–Wallis test. A further batch of cylindrical specimens of 6 mm in diameter and 12 mm in height were prepared for testing surface microhardness. Three groups of 15 specimens were subjected to the acid-etch procedure at either 4, 24 or 96 h following mixing. Data were subjected to one-way ANOVA. Changes in the surface microstructure before and after the acid-etch procedures were analysed using a scanning electron microscope (SEM).

Results There was a general trend for the compressive strength and surface microhardness of specimens to increase with time. In terms of compressive strength, the increase was significant between 4 h and the other time periods for both experimental and control groups ($P < 0.0001$); however, there was no significant difference between 24 and 96 h. The increase in surface microhardness was significant between 4, 24 and 96 h ($P < 0.0001$). In addition, there was a significant difference between experimental and control groups at all time periods ($P < 0.0001$). SEM examination revealed morphological differences between the intact and the etched MTA surfaces.

Conclusions Acid-etch procedures affected the compressive strength and surface microhardness of ProRoot MTA. This indicates that it may be better to postpone restorative procedures for at least 96 h after mixing MTA. Etching created surface changes that might have the potential to enhance bonding of resinous materials.

Keywords: acid etch, compressive strength, pH, SEM, Vickers surface micro hardness.

Received 16 February 2009; accepted 28 May 2009

Introduction

During the repair of root perforations, or during apexogenesis and apexification procedures, an appropriate material is placed adjacent to the connective tissues in order to restore tooth structure and act as a barrier against bacterial microleakage. The biocompatibility of materials used in such circumstances is a most critical requirement. However, it is also desirable for the selected material to induce or conduct bone deposition, release calcium hydroxide, provide a good seal against bacteria and fluids, have antibacterial property, set in a

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wet environment, be unaffected by blood contamination, and have reasonable compressive strength and hardness.

Mineral trioxide aggregate (MTA) has been shown to be biocompatible (Keiser *et al.* 2000, Ribeiro *et al.* 2006), and can be used safely when placed adjacent to pulp and periodontal tissues (Abedi & Ingle 1995, Torabinejad & Chivian 1999). It can also provide an environment that supports cementum re-growth (Alhadainy 1994). Low solubility (Fridland & Rosado 2005), the ability to set in a wet environment (Karabucak *et al.* 2005), facilitating control of bleeding (Tunca *et al.* 2007), prevention of bacterial leakage (Fuks 2002) and release of calcium hydroxide (Camilleri 2007) are some of the other properties of MTA. Thus, MTA appears to be a most promising material for use in a variety of clinical applications, including direct pulp capping, pulpotomy, orthograde root-end closure, sealing of perforations and as a root-end filling material after root-end resection.

Despite its unique combination of properties and great potential, the prolonged setting time of MTA is a major disadvantage (Antunes Bortoluzzi *et al.* 2006), along with its poor handling properties (Levenstein 2002). After mixing MTA with water, the resulting material has a 'grainy' and 'sandy' consistency, which is difficult to manipulate (Kogan *et al.* 2006). In addition, as MTA requires water to initiate and complete the setting reaction, placing a wet cotton pellet next to MTA is recommended following its placement (Torabinejad 2004). As a result, other filling materials should not be placed adjacent to it at the same appointment; obviously, this increases the number of appointments required and delays the placement of the final restoration.

Although MTA has been suggested for use in a variety of clinical applications, there is limited information about the effect of various treatments on the physical properties of MTA. In particular, there is no information about the effect of conditioning process, including exposure to phosphoric acid, on the physical properties and crystalline structure of MTA, which is a reflection of the hydration process (Lee *et al.* 2004, Camilleri 2007, Nekoofar *et al.* 2007). The effect of phosphoric acid, which is used to increase the retention and sealability of composite resin restorations, on the properties of pulp capping and root repair materials is a fundamental problem and one that could affect their durability and effectiveness (El-Araby & Al-Jabab 2005).

Fuss *et al.* (1990) evaluated the effect of acid-etch technique on glass ionomer cements when used as a lining material and reported that 15 s etching had no detrimental effect. In a similar study, the surface structure of glass ionomer was evaluated under scanning electron microscopy (SEM) and no significant loss of cement was reported after acid-etching procedures (Smith & Martin 1990). However, no information is available on MTA.

The purpose of this study was to evaluate the influence of acid-etch procedures 4, 24 or 96 h after mixing on surface microhardness, compressive strength and the surface crystalline structure of tooth-coloured ProRoot MTA (Dentsply Tulsa Dental; Johnson City, TN, USA).

Methods and materials

The parameters investigated were surface hardness (Vickers microhardness), compressive strength and assessment of surface morphological characteristics using SEM. The material investigated was the tooth-coloured formula of ProRoot MTA with LOT number 083006 (Dentsply Tulsa Dental).

Compressive strength

The material was mixed according to the manufacturer's instructions. Each sachet containing 1 g of MTA was mixed gradually with a 0.33-g aliquot of water, not with the water supplied by the manufacturer which could be inconsistent (Nekoofar *et al.* 2009). The mixed material was packed incrementally into 90 stainless steel cylindrical moulds having an internal diameter of 4 mm and height of 6 mm placed on a glass slab. The samples were then subjected to a constant vertical force (3.22 MPa applied for 1 min) that was translated into a transverse and equally distributed pressure that compacted the MTA evenly into the cylindrical mould (Nekoofar *et al.* 2007). The extruded material was removed and a wet cotton gauze was placed onto the MTA and samples were then stored at 37 °C and 95% humidity in an incubator.

After 4 h, 30 samples were randomly selected and divided into two groups of 15. In the first group, the exposed surfaces of the samples were covered by phosphoric acid 37% (3M ESPE Co., St Paul, MN, USA) for 15 s then rinsed using tab water for 15 s and dried gently using a stream of air for 15 s. The second group of 15 specimens was used as the control group and were not subjected to the acid-etch procedure. The

acid-etch procedure was repeated 24 and 96 h after mixing on a further 30 samples at each time period respectively. The compressive strength test was conducted on each specimen just after etching and on the control specimens using a universal testing machine (Instron, 3345, Norwood, MA, USA). 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. Compressive strength was calculated by dividing the maximum load at fracture by the original cross-sectional area of the specimen. The failure load was recorded and the compressive strength σ (sigma) was calculated using the following equation:

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

where P was the force (N) applied and d (mm) was the diameter of the specimen. The compressive strength of all specimens was recorded in mega Pascal (MPa).

The data obtained for compressive strength of all six groups were found to be nonparametrically distributed. Therefore, differences between groups were analysed using the Kruskall–Wallis test.

Surface microhardness

The material was mixed according to the manufacturer's instructions. Each sachet containing 1 g of MTA was gradually mixed with an aliquot of 0.33 g water, not with the water supplied by the manufacturer which could be inconsistent (Nekoofar *et al.* 2009). The mixed material was packed incrementally into 45 polycarbonate cylindrical tubes having an internal diameter of 6 mm and height of 12 mm placed on a glass slab. The samples were then subjected to a constant vertical force (3.22 MPa applied for 1 min) that was translated into a transverse and equally distributed pressure that compacted the MTA evenly into the cylindrical mould. The extruded material was removed and a wet cotton gauze was placed onto the MTA and samples stored at 37 °C and 95% humidity in an incubator.

After 4 h, 15 samples were randomly selected and the MTA removed from the mould. The exposed end surface of each sample was covered by phosphoric acid 37% (3M ESPE Co.) for 15 s then rinsed using tab water for 15 s and dried gently using a stream of air for 15 s. The other end surface of each sample was used as the control and was not subjected to the acid-etch procedure. Both surfaces of the specimens were wet polished at room temperature using silicon carbide-based sandpapers of varying particle size ('WetordryTM' 600-grit, 737 SF 'Tri-M-iteTM' and 1000-grit 'WetordryTM'; 3M, St Paul, MN, USA) with minimum hand pressure to provide smooth surfaces for ease of indentation. By employing wet polishing and gentle pressure, the influence of sample processing on the structure and surface microhardness was minimized. The polished specimens were cleaned gently with distilled water to remove surface debris. To prevent dissolution or water sorption, the surfaces were dried immediately using a gentle air spray. The Vickers hardness test of each specimen was performed using a Micromet 5114 microhardness tester (Buehler Ltd., Lake Bluff, IL, USA) using 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 (HV) is calculated based on the following formula:

$$\mathrm{HV} = \frac{2F\sin\frac{136^\circ}{2}}{d^2}, \quad \mathrm{HV} = 1.854\frac{F}{d^2} \text{ approximately}$$

where F is the load (kg^{-1}) and d is the mean of the two diagonals of the impression made by the indenter in millimetres. The mean Vickers surface microhardness values obtained were calculated to determine the surface microhardness for each specimen. The same etching procedures and surface microhardness tests were repeated on specimens 24 or 96 h following mixing on a further 15 samples at each time.

Differences between the Vickers surface microhardness values obtained from the surfaces that were exposed to the acid-etch procedure at different time periods and control surfaces was compared statistically using one-way ANOVA followed by a *post hoc* Tukey's test.

Scanning electron microscopy

For the morphological evaluation, new specimens were prepared as described above and kept under the same

storage conditions. After 4, 24 and 96 h, samples were randomly divided into two groups of five. The exposed surfaces of samples in the test groups were covered by phosphoric acid 37% (3M ESPE Co.) for 15 s then rinsed using tap water for 15 s and then dried gently using a stream of air for 15 s. Samples in the control group were not subjected to the acid-etch procedure. 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) SEM (S.E.M. Tech Ltd, Woodbridge, UK). Gold sputter coating in the vacuum chamber was not possible on specimens removed after 4 h due to the amount of moisture in the samples; therefore, morphological evaluation by SEM after 4 h was impossible. The micrograph images from the SEM analysis of two other etched and control groups after 24 and 96 h were compared in terms of the surface morphology, and type of crystal formation.

Results

There was a general trend for the compressive strength and surface microhardness of specimens to increase with time (Figs 1 and 2).

Compressive strength

The results of the compressive strength testing are shown in Fig. 1. In the experimental groups, the lowest compressive strength (mean = 6.438 MPa, SD = 2.360) was observed on the specimens etched after 4 h. There was a statistically significant difference between compressive strength values of specimens etched after 4 h compared with those groups etched at 24 and 96 h ($P < 0.0001$). However, there was no statistically significant difference between specimens etched after 24 and 96 h respectively.

Statistically significant differences in compressive strength values between test and control groups occurred only after 4 h ($P < 0.0001$).

Surface microhardness

The results of the surface microhardness test are shown in Fig. 2. In the group that was exposed to the acid-etch procedure, the lowest Vickers surface microhardness value (mean = 17.269, SD = 6.382) was observed after 4 h. There was a statistically significant difference between specimens etched after 4, 24 and 96 h respectively ($P < 0.0001$).

There was a statistically significant difference in Vickers surface microhardness values between test and control groups after 4, 24 and 96 h respectively ($P < 0.0001$). The surface microhardness values of the test group were lower than the control group at all time intervals (Fig. 2).

In the control group, there was a statistically significant difference in Vickers surface microhardness values between specimens after 4 h compared with the other control groups ($P < 0.0001$). However, there was no statistically significant difference between control specimens after 24 and 96 h respectively.

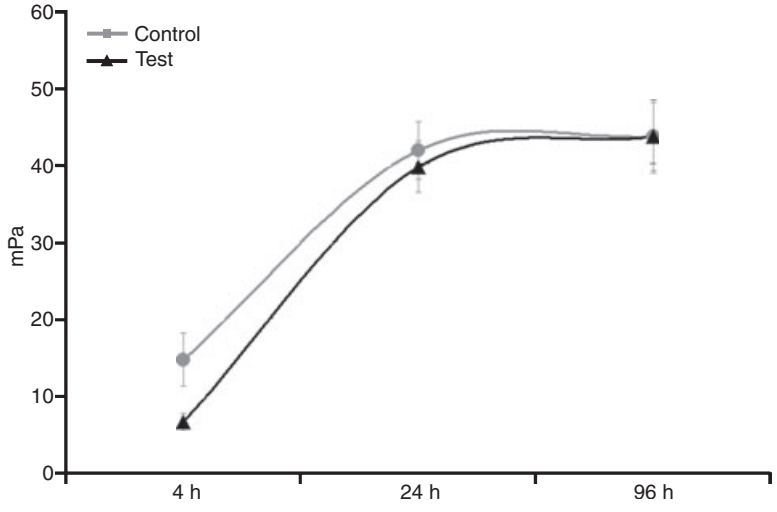

Figure 1 Mean compressive strength (MPa) of intact and etched specimens after 4, 24 and 96 h.

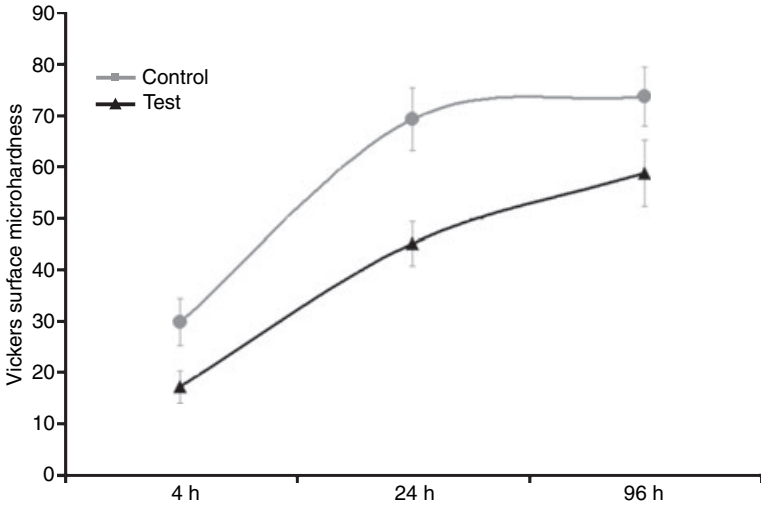

Figure 2 Mean Vickers surface microhardness of intact and etched surfaces after 4, 24 and 96 h.

Scanning electron microscopy

The SEM examination revealed distinct morphological differences between the intact (Figs 3–7) and the etched MTA surfaces (Figs 8–13). No morphologically dissimilarity was observed within the test and control groups at 24 and 96 h after mixing (Figs 3, 4, 9 and 10). The surface microstructure of the unetched MTA (control groups) after both time period were similar and showed an amorphous poorly crystallized superficial gel structure at 36× magnification (Figs 3 and 4). The presence of needle-like crystals was a common finding in control groups at 500× and 700× magnification after 24 and 96 h (Figs 5 and 6). At 1187× magnification, a plain poorly crystallized superficial gel structure containing globular aggregate particles was observed in the control group (Fig. 7). After etching procedures at 24 or 96 h, a selective loss of matrix from around the crystalline structures were observed at 36×, 500× and 700× magnifications that produced a relatively uniform 'honeycomb' etched pattern without penetrating deeply or removing substantial amounts of the cement (Figs 8–10). No needle-like crystals were observed over the etched surfaces. Loss of the needle-like crystals was a significant morphological difference between etched and intact (control) surfaces (Figs 5, 6, 9 and 10). In addition, acid-etch procedures after 24 and 96 h revealed notable crystalline structures such

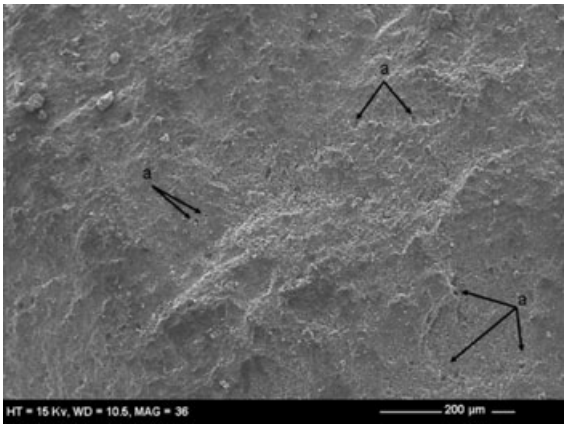

Figure 3 Intact surface of unetched mineral trioxide aggregate after 24 h. An amorphous poorly crystallized superficial gel structure and the cross-section of some microchannels (a) can be seen (original magnification: 36×).

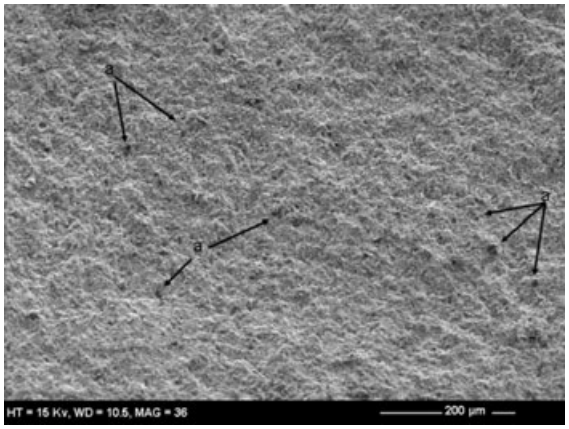

Figure 4 Intact surface of unetched mineral trioxide aggregate after 96 h. An amorphous poorly crystallized superficial gel structure and the cross-section of several microchannels (a) can be seen (original magnification: 36×).

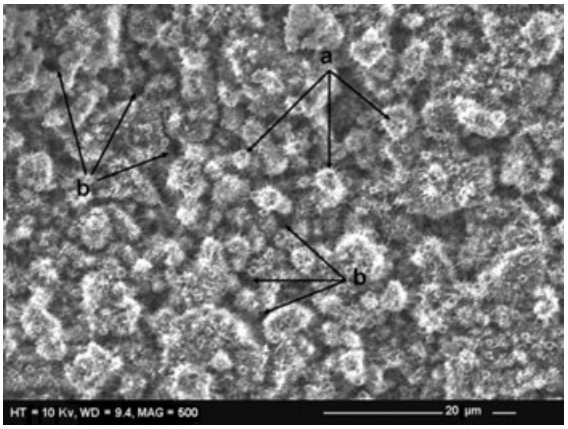

Figure 5 Intact surface of unetched mineral trioxide aggregate after 24 h. Irregular needle-like crystals that cover globular formations (a) and cross-sections of several microchannels (b) can be seen (original magnification: 500×).

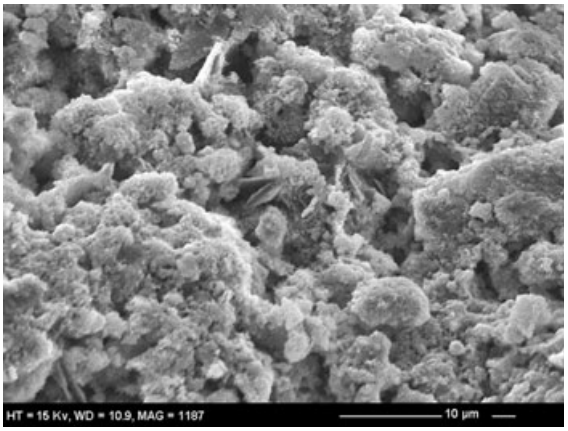

Figure 7 Intact surface of unetched mineral trioxide aggregate after 96 h. The relatively uncrystallized superficial gel structure containing the globular particles can be seen (original magnification: 1187×).

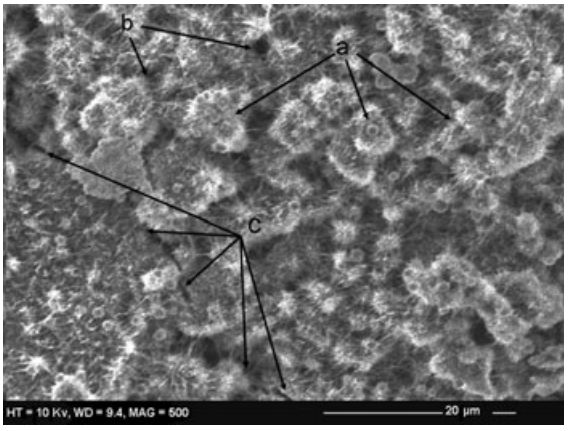

Figure 6 Intact surface of unetched mineral trioxide aggregate after 96 h. Irregular needle-like crystals that cover globular formations (a), cross-sections of some microchannels (b) and a microchannel running transversely (c) can be seen (original magnification: 500×).

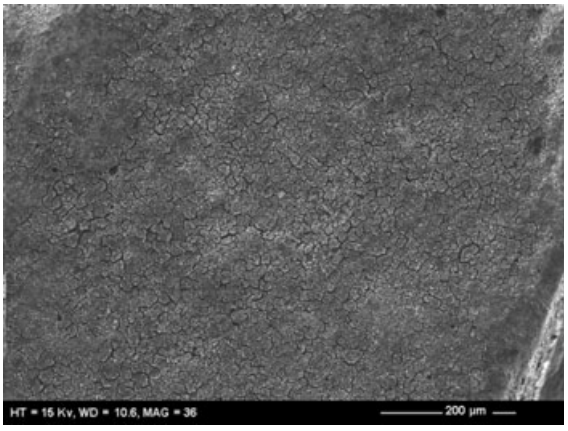

Figure 8 Etched surface of mineral trioxide aggregate after 24 h. A relatively uniform 'honeycomb' etched pattern can be seen (original magnification: 36×).

as plate-shaped and laminated crystals on the MTA surface (Figs 11–13). There was no evidence of superficial gel structure throughout the surface of the etched groups after 24 and 96 h at the various magnifications (Figs 8–13).

Discussion

Mineral trioxide aggregate was introduced initially for the repair of root perforations (Lee *et al.* 1993) and was subsequently recommended as a root-end filling material following root resection (Torabinejad *et al.* 1993, 1995). It has also been suggested for use in vital pulp therapy (Pitt Ford *et al.* 1996). As hard tissue induction is one of its exceptional properties, it has been recommended for use as an apical barrier in the treatment of immature teeth with necrotic pulps and open apices (Bakland 2000) and for repair of resorptive root defects (Schwartz *et al.* 1999). Yildirim & Gencoglu (2009) reported new hard tissue formation in two horizontal root fracture lines after a 5-year follow-up suggesting the use of MTA in the treatment of such cases. Gomes-Filho *et al.* (2008) showed that a sealer

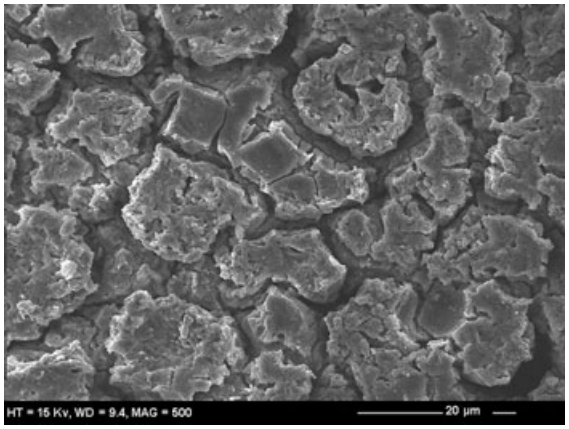

Figure 9 Etched surface of mineral trioxide aggregate after 24 h. Selective loss of matrix from around the crystalline structures and relatively uniform 'honeycomb' etched pattern with minimal loss of the cement can be seen. No needle-like crystals were observed (original magnification: 500×).

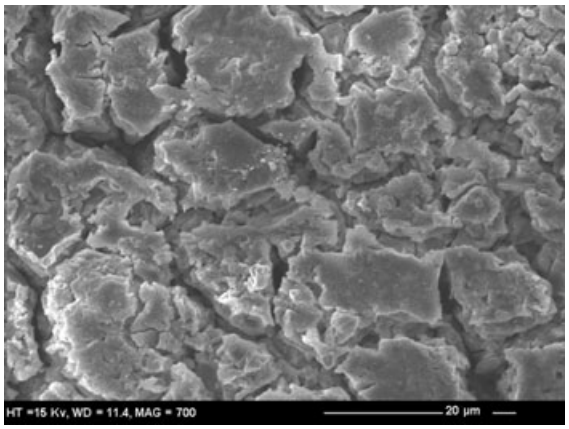

Figure 10 Etched surface of mineral trioxide aggregate after 96 h. Selective loss of matrix from around the crystalline structures and relatively uniform 'honeycomb' etched pattern with minimal loss of the cement can be seen. No needle-like crystals were observed (original magnification: 700×).

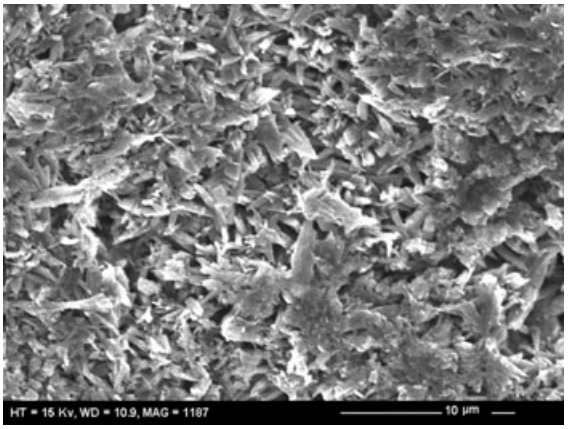

Figure 11 Etched surface of mineral trioxide aggregate (MTA) after 96 h. Selective elimination of matrix with minimal loss of the cement can be seen. Laminated and plate-shaped crystals are notable and visible on the MTA surface. No needle-like crystals were observed (original magnification: 1187×).

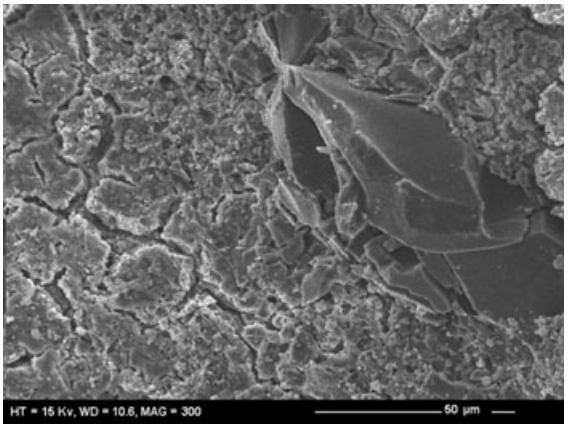

Figure 12 Etched surface of mineral trioxide aggregate (MTA) after 96 h. Selective elimination of matrix with minimal loss of the cement can be seen. Laminated and plate-shaped crystals were a notable feature on the MTA surface. No needle-like crystals were observed (original magnification: 300×).

based on MTA was biocompatible and stimulated mineralization.

Because of its many potential advantages, MTA is increasingly being used in various clinical modalities such as repair of root perforations (Uyanik *et al.* 2009) and as a pulp capping agent in vital pulp therapy (Accorinte *et al.* 2009), when the final coronal restoration will be in intimate contact with MTA. There is anecdotal evidence that some operators tend to place the final coronal restoration in the same appointment as the MTA. However, the effect of various restoration procedures on the chemical and mechanical characteristics of MTA and the appropriate time of restoration after mixing of MTA are important issues that have not been evaluated adequately.

The chemical bonding of MTA to dentine as a result of a physiochemical interaction has been suggested (Sarkar *et al.* 2005). These authors indicated that after the placement of MTA adjacent to dentine, hydroxyapatite crystals grow around the

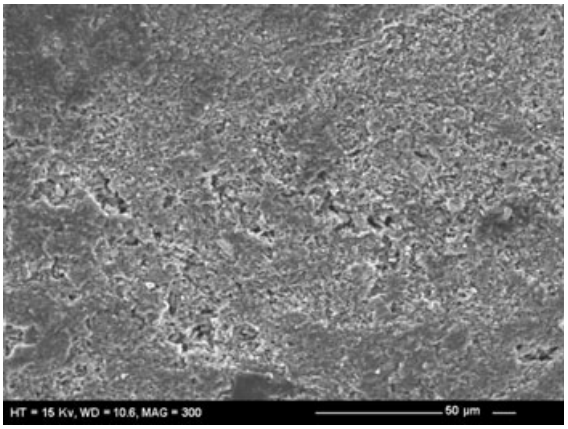

Figure 13 Etched surface of mineral trioxide aggregate (MTA) after 24 h. Selective elimination of matrix with minimal loss of the cement can be seen. Laminated and plate-shaped crystals were a notable feature on the MTA surface. No needle-like crystals were observed (original magnification: 300×).

MTA particles and filled the microscopic gap between the material and dentine. Yan *et al.* (2006) evaluated the bond strength of MTA and dentine in different environments and demonstrated that there was no statistically significant difference between the strength of the bond even when the dentine had been exposed previously to sodium hypochlorite and chlorhexidine (Sarkar *et al.* 2005). Tunc *et al.* (2008) evaluated the adhesive properties of MTA and restorative materials by investigating the shear bond strength of two resin composites used with two different bonding systems to tooth-coloured Pro Root MTA. They recommended that composite resins used with a total-etch, one bottle adhesive system was an appropriate final restoration in contact with MTA.

In the present study, the effects of an acid-etch procedure on the surface microhardness, compressive strength and surface morphology of tooth-coloured Pro Root MTA (Dentsply Tulsa Dental) was investigated. It is known already that exposure of MTA to a low pH environment may influence its physical and chemical properties (Watts *et al.* 2007, Camilleri & Pitt Ford 2008, Namazikhah *et al.* 2008, Saghiri *et al.* 2008, Shie *et al.* 2009). Because of the prolonged hydration and setting process of MTA and lack of knowledge about the effect of restoration procedures during this time, the effect of phosphoric acid on MTA was investigated 4, 24 and 96 h after mixing. Compressive strength and surface microhardness are indicators of the setting process and strength of the material (Bentz 1997, Danesh *et al.* 2006). They can also indicate the effect of various setting conditions on the overall strength of a material (Blake 1985).

According to the British Standard (BS EN 843-4 2005) for a reliable measurement of surface microhardness, the test specimen surfaces should be parallel to obtain symmetrical indentations. In addition, the test surface should be polished prior to surface microhardness measurement to obtain a smooth scratch-free area that allows for a more reliable measurement of the indentations.

The results of this study demonstrated that acid etch applied 4 h after mixing MTA with water, significantly reduced its resultant compressive strength compared with the controls. However, after 24 and 96 h, these differences were not significant. Therefore, to reduce the potential adverse effects of the acid on the compressive strength of MTA, it could be suggested that it is only necessary to postpone the acid-etch procedure and the restoration of a tooth for 24 h. On the other hand, the acid-etch procedure significantly reduced the surface microhardness of MTA when applied 4, 24 and 96 h after mixing, which is in accordance with the findings of Lee *et al.* (2004) and Namazikhah *et al.* (2008) who reported that MTA does not harden as well in a low pH environment. At 24 and 96 h following mixing, the superficial gel-like amorphous structure and needle-like crystals were missing in the etched samples when they were observed under SEM. This selective loss of matrix from around the crystalline structures with minimal loss of the cement resulted in a relatively uniform 'honeycomb' etched pattern and exposure of crystalline structures that could provide a satisfactory surface for bonding resin materials. Due to the humidity of the specimens, it was not possible to evaluate the specimens after 4 h of mixing. To overcome this limitation, the use of an environmental SEM that does not require a vacuum (Bergmans *et al.* 2005) may be advantageous. Thus, taken together the results would suggest that delaying tooth restoration for at least 96 h or longer is desirable. According to the result of this study, there was also a trend for increasing the compressive strength and surface microhardness over time in both test and control groups; therefore by delaying the placement of the final coronal restoration the material can acquire sufficient compressive strength to withstand the acid-etch procedure and/or condensation pressures used during the placement of a restoration.

Bodanezi *et al.* (2008) evaluated the short- and long-term solubility of MTA-Angelus (Angelus; Londrina, PR, Brazil) and suggested that at least 72 h was

necessary to achieve the desirable sealability. Their conclusion was based on the finding that during the first 72 h after mixing, the degree of solubility of the material was high. Vanderweele *et al.* (2006) recommended that MTA should be allowed to set untouched for 72 h or longer to decrease the chance of material displacement. Sluyk *et al.* (1998) also reported that for achieving the desirable sealability, MTA should be untouched for 3 days when used to repair root perforations.

According to the findings of the SEM evaluation, the surface morphology of MTA after acid-etch procedures created a selective loss of matrix from around the crystalline structures that resulted in a relatively uniform 'honeycomb' etched pattern without penetrating deeply or removing substantial amounts of the cement (Figs 8–10). This differential etching pattern is an essential characteristic for achieving a satisfactory bond to resin restorations and is one of the requirements when selecting a material for the composite resin combined or 'sandwich' technique (Sheth 1989, Fuss *et al.* 1990). Accordingly, it would appear that the combined MTA-composite restoration could provide a reliable chemical bond to dentine, as well as have the potential for micromechanical bonding of the composite to MTA surfaces (Sarkar *et al.* 2005, Yan *et al.* 2006, Tunc *et al.* 2008).

Despite the fact that the type and duration of acid exposure was not the same as in this study, the 'honeycomb' pattern of the etched surfaces has been reported previously by Lee *et al.* (2004) as well as the exclusive removal of the needle-like crystals. In addition, in the present study, the acid-etch procedures after 24 and 96 h created notable structures such as plate-shaped and laminated crystals on the MTA surface (Figs 11–13). This finding is in accordance with Namazikhah *et al.* (2008), although in their study MTA samples were exposed to butyric acid for 4 days and not the phosphoric acid of the present study.

The significance of these morphological changes is unclear. Lack of sufficient information about the hydration of MTA makes the interpretation of the SEM findings difficult. However, removal of the superficial gel-like amorphous structure, lack of needle-like crystals throughout the etched samples and exposure of remarkable crystalline structures were common findings in the etched samples. Further studies are suggested to determine the significance of these changes in terms of bonding to composite resins.

Conclusion

It is recommended that restorative procedures are postponed at least for 96 h after mixing MTA to allow the material to achieve its optimum physical properties. Changes to the surface of MTA as a result of acid etching 96 h after mixing appear to indicate a structure that may have potential beneficial effects on bonding prior to the placement of composite resin restorations. However, future work is required to confirm this hypothesis.

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