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The effect of various mixing techniques on the surface microhardness of mineral trioxide aggregate

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Abstract

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Aim To evaluate the influence of various mixing procedures including ultrasonic vibration, trituration of customized encapsulated mineral trioxide aggregate (MTA) and condensation on the Vickers surface microhardness of MTA.

Methodology ProRoot® MTA Original, ProRoot® MTA (white), MTA-Angelus® (grey) and MTA White Angelus® (white) were prepared using several mixing techniques including ultrasonic vibration, trituration of customized encapsulated MTA and conventional condensation. Twelve experimental groups (four materials: three techniques) were evaluated, each with 35 samples. All samples were incubated after preparation and subjected to Vickers surface microhardness testing after 4 and 28 days. Data was were subjected to a two-way ANOVA.

Result At 28 days, the surface microhardness value was significantly greater for all experimental groups compared to 4 days after mixing ($P < 0.00001$). The application of ultrasonic energy to MTA produced significantly higher surface microhardness values compared to the other mixing techniques at both 4 and 28 days ($P < 0.0001$). However, no significant difference existed between condensation and trituration techniques at both time intervals. Regardless of the mixing technique employed, a significant difference ($P < 0.0001$) was observed in surface microhardness value between all types of MTA apart from between Angelus grey and ProRoot white at both 4 and 28 days, both of which produced the highest values.

Conclusion Compared to trituration and condensation techniques, the application of ultrasonic energy to MTA produced a significantly higher surface microhardness value at both 4 and 28 days. Irrespective of mixing technique, ProRoot white and Angelus grey had the highest surface microhardness values. Trituration of encapsulated, premeasured MTA and water provides a standardiszed method of mixing that produces MTA slurries with more controllable handling characteristics.

Keywords: condensation, mineral trioxide aggregate, mixing technique, trituration, ultrasonic, Vickers surface microhardness.

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Introduction

The clinical outcomes of the majority of dental restorative procedures are influenced by the chemical and physical properties of dental materials (Main *et al.* 2004, Behr *et al.* 2008, Drummond 2008). These properties can be effected by mixing technique (Nomoto & McCabe 2001, Nomoto *et al.* 2004), delivery system (Hachmeister *et al.* 2002), exposure to various clinical environments (Vanderweele *et al.* 2006), storage conditions (Roulet 1995) and the ratio of the constituent components (Fridland & Rosado 2003, Behr *et al.* 2008). Sensitivity to clinical techniques might also affect the optimum performance of materials

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(Kleverlaan *et al.* 2004), as well as their ease of handling, which when not good might contribute to its misuse (Maltezos *et al.* 2006).

Many dental materials are available as two components that are mixed before use, including 'powder and liquid', 'two paste' and 'paste and liquid' systems. When one component in the system is a liquid, the achievement of a homogenous mixture becomes less predictable (Powers & Wataha 2008). In addition, depending to the setting and hardening mechanisms of the material, manual mixing might result in inconsistencies that can be improved by mechanical mixing of their constituents (Gbureck *et al.* 2005). Mechanical mixing has the potential to reduce air spaces between adjacent particles, resulting in a more thorough wetting of the powder particles and lead to an improvement in the unification of the resultant paste (Nomoto *et al.* 2004). Trituration is one method of mechanical mixing that is used extensively for various dental materials. Initially, the individual components of the material were placed manually into a mixing device. Then, in an attempt to enhance consistency, various dental materials including, amalgam, glass–ionomer, self-cured composite resin, zinc phosphate and calcium hydroxide cements became available in capsules containing pre-set proportions of their components that were then triturated prior to use. The capsule might also contain a small rod-like pestle, which improves the mechanical mixing. This system has the potential to produce a consistently uniform, void or pore-free mixture (Powers & Wataha 2008).

Whilst trituration uses conventional mechanical vibration, there might be a potential for ultrasonic energy to be more effective. Ultrasonic vibration has a dispersing effect on the particles of the material, which frequently cluster together. Indeed, ultrasonic energy has been used to enhance the mechanical properties of materials, such as compressive strength (Kleverlaan *et al.* 2004, Barata *et al.* 2008), tensile bond strength (Algera *et al.* 2005, Fagundes *et al.* 2006), hardness (Towler *et al.* 2001) and fill density (Yeung *et al.* 2006). Ultrasonic energy might also increase the total reactive surface area, improve particle interaction and decrease setting time (Kleverlaan *et al.* 2004, Algera *et al.* 2005). In addition, by changing rheological properties, it can improve the placement and handling characteristics of materials (Witherspoon & Ham 2001, Lawley *et al.* 2004, Schmidlin *et al.* 2005), as well as marginal adaptation to the cavity wall (Schmidlin *et al.* 2007).

One of the increasingly used materials in endodontic treatment is mineral trioxide aggregate (MTA). MTA is a type of hydraulic cement that can set in the presence of water. In simple terms, hydraulic cements are finely ground materials (powders) that when mixed with water gradually or instantly set and harden in air or in water; the reaction resulting in the formation of hydrated compounds whose strength increases with time (Camilleri 2007, 2008).

At the outset, MTA was commercially available as ProRoot® MTA Original (Dentsply Tulsa Dental, Johnson City, TN, USA); however, recently, another commercially available cement has been launched as MTA-Angelus® (Angelus Dental Industry Products, Londrina, Brazil) (Duarte *et al.* 2003). Both manufacturers produce two types of MTA (grey and white), which are available as 'powder and water' systems.

Hachmeister *et al.* (2002), in an attempt to improve the sealing ability of MTA in an immature root canal model, emphasized the importance of the delivery system rather than the material itself. Aminoshariae *et al.* (2003) compared two placement techniques for MTA, concluding that hand condensation of MTA provided less porosity, better adaptation and fewer voids than ultrasonic vibration. Nekoofar *et al.* (2007) examined the effect of condensation pressure on the surface microhardness, microstructure and compressive strength of MTA and concluded that higher condensation pressures produced lower microhardness values, suggesting that high condensation pressure might affect the hydration, strength and microhardness of MTA. Roberts *et al.* (2008) highlighted the use of ultrasonic vibration for MTA placement.

In all studies to date, MTA was mixed according to the manufacturer's instructions that recommend a powder to water ratio of 3–1. For example, each sachet of ProRoot MTA (Dentsply Maillefer) contains 1 g of cement that should be gradually mixed with the water supplied by the manufacturer in small plastic ampoules. However, Nekoofar *et al.* (2009) reported that the ampoules of water supplied with ProRoot MTA by the manufacturer are inconsistent in terms of volume, with potential consequences for the physical properties of the set material. Furthermore, the recommended ratio of powder to water by the manufacturer has been challenged (Aminoshariae *et al.* 2003, Pelliccioni *et al.* 2007). In experimental studies and clinical usage, adhering to a consistent ratio is important as deviation might produce inconsistent setting and reduced physical properties (Nekoofar *et al.* 2009).

To achieve consistency, it could be hypothesized that encapsulated pre-set proportions of MTA could confer advantages to the practitioner and the material.

Because amalgamators are available in most dental clinics, it might be efficient to use such mixing equipment for encapsulated pre-set proportions of MTA. There is no information about the effect of mixing or manipulation technique on the surface microhardness of MTA, which is a reflection of the hydration process (Nekoofar *et al.* 2007, Namazikhah *et al.* 2008). The purpose of this study was to evaluate the influence of various mixing techniques including ultrasonic vibration, trituration of customized encapsulated MTA and condensation on the surface microhardness of MTA.

Material and methods

The parameter investigated was surface microhardness (Vickers microhardness)

The materials investigated were

- ProRoot® MTA Original (Dentsply Tulsa Dental, Johnson City, TN, USA) with LOT number of 05003087 (grey)
- ProRoot® MTA (Dentsply Tulsa Dental, Johnson City, TN, USA) with LOT number of 083006 (white)
- MTA-Angelus® (Angelus Soluções Odontologicas) with LOT number 6701 (grey)
- MTA White Angelus® (Angelus Dental Industry Products, Londrina, Brazil) with LOT number 6389 (white)

One gram of each powder was mixed with a 0.34 g aliquot of distilled water using three various mixing techniques including conventional condensation, trituration and ultrasonic energy. There were 12 experimental groups each with 35 samples.

Condensation

The measured distilled water was added to the powder and left until it was absorbed. It was then placed with minimal pressure using the tip of a dental spatula into customized polycarbonate cylindrical moulds having an internal diameter of 4 mm and height of 6 mm, which were placed on a glass slab. The MTA within the moulds was then subjected to a constant vertical compaction force of 3.22 MPa applied for one minute (Nekoofar *et al.* 2007) that was translated into a transverse and equally distributed pressure that compacted the material evenly into each cylindrical mould. The extruded material was wiped away and a wet cotton pellet placed onto the MTA. The polycarbonate moulds were then placed onto a damp paper towel in a sealed plastic container and incubated at 37 °C temperature and 95% humidity.

Trituration

An aliquot of 0.34 g of distilled water was added to the MTA powder in an empty, clean amalgam capsule until it was absorbed. A plastic pestle was then added, and encapsulated material was triturated immediately for 30 s at 4500 rpm using an amalgamator (Silamat®; Ivoclar, Vivadent AG, Liechtenstein). The mixed material was placed with minimal pressure using the tip of a dental spatula into customized polycarbonate cylindrical moulds having an internal diameter of 4 mm and height of 6 mm, which were placed on a glass slab. The extruded material was wiped away, and a wet cotton pellet was placed on top of the MTA. The polycarbonate moulds were then placed onto a damp paper towel in a sealed plastic container and incubated at 37 °C temperature and 95% humidity.

Ultrasonic vibration

An aliquot of 0.34 g of distilled water was added to the MTA powder and left until it was absorbed. It was then placed with minimal pressure using the tip of a dental spatula into customized polycarbonate cylindrical moulds having an internal diameter of 4 mm and height of 6 mm, which were placed on a glass slab. The samples were then each treated with ultrasonic energy for 30 s at scale five using a CPR-2D tip (Obtura Spartan, Fenton, MO, USA) with a Suprasson P5 ultrasonic booster (Satelec, Merignac, France). The tip of the ultrasonic device was placed in the centre of the material and not in contact with the walls or floor of the mould. The extruded material was then wiped away, and a wet cotton pellet was placed on top of the MTA. The polycarbonate moulds were then placed onto a damp paper towel in a sealed plastic container and incubated at 37 °C temperature and 95% humidity.

Surface microhardness

After 4 days, all samples were removed from the incubator and subjected to a surface microhardness test. The surfaces that were in contact with the damp paper towel were wet polished at room temperature using minimum hand pressure and silicon carbide grinding papers (Buehler-Met®; Agar Scientific Limited, Cambridge, UK) of 600-grit, 1000-grit and 1200-grit, respectively. The Vickers surface microhardness of each

specimen was then performed in accordance with the European and British Standard (BS EN 843-4:2005) using a Micromet 5114 tester (Buehler Ltd, Lake Bluff, IL, USA) with a square-based, pyramid-shaped diamond indenter with a full load of 500 g for 30 s at room temperature. This 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°. Ten indentations were randomly made on the polished surface of each specimen at separated locations to adjacent indentations or from the specimen periphery. The diagonal produced from the indention was measured immediately under the microscope, and the Vickers surface microhardness value was displayed on the digital readout. 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 load (kg^{-1}) and d is the mean of the two diagonals produced by the indenter in millimetres. After microhardness testing, all samples were immediately replaced into the incubator. After 28 days, samples were removed from the incubator and subjected to the surface microhardness test using the same methodology.

The mean Vickers surface microhardness value and standard errors were calculated for each group and subjected to a two-way ANOVA. All analysis was performed using the Statistical Package of Social Science (SPSS Inc., Chicago, IL, USA).

Results

A trend was observed that at 28 days, the surface microhardness values were significantly greater for all experimental groups compared to 4 days after mixing ($P < 0.00001$).

Regardless of the type of MTA used, the application of ultrasonic energy produced the highest surface microhardness at 4 and 28 days (Fig. 1). There was a statistically significant difference between the values of surface hardness following ultrasonic energy and the other mixing techniques at 4 and 28 days ($P < 0.0001$). No significant difference existed between condensation and trituration techniques at both time intervals.

When comparing mixing technique by experimental materials, for ProRoot MTA (white), the highest surface microhardness values were found in the ultrasonic group (Table 1). There was a statistically significant difference between the ultrasonic mixing compared to

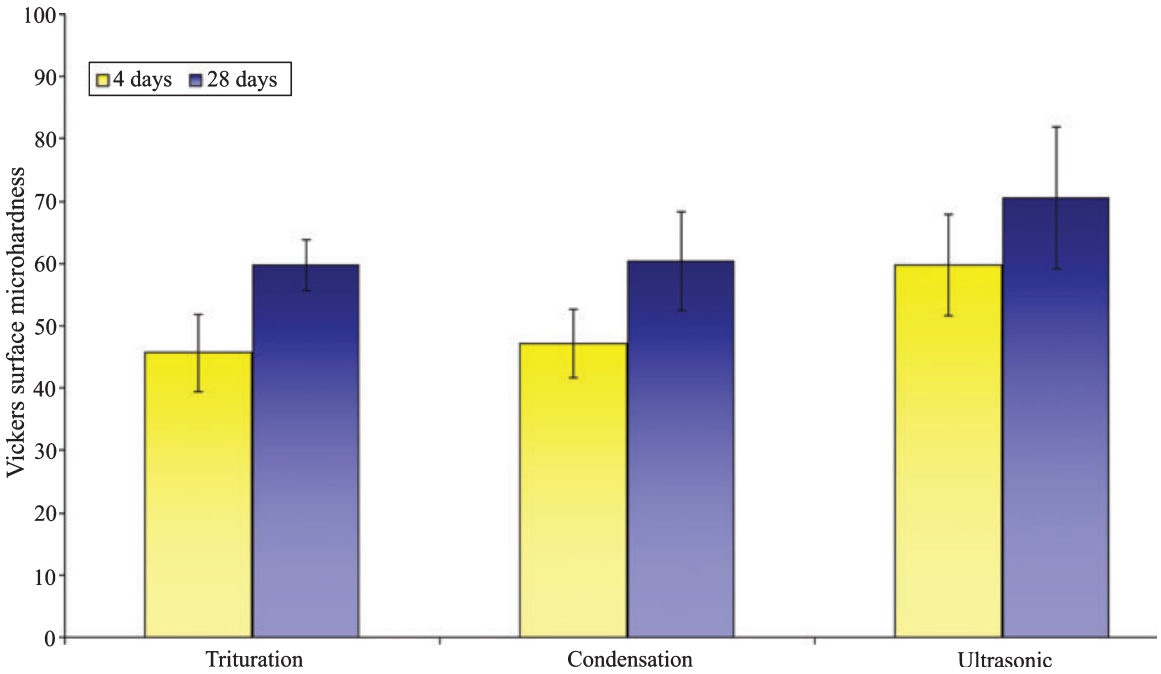

Figure 1 Vickers surface microhardness values at 4 and 28 days irrespective of the type of mineral trioxide aggregate used.

Table 1 The mean, median and standard deviation values of Vickers surface microhardness for each type of MTA mixed by various techniques at 4 and 28 days

		4 days			28 days		
		Mean	Median	Standard deviation	Mean	Median	Standard deviation
White ProRoot-MTA	Trituration	65.18	66.75	6.42	64.12	64.30	5.77
	Condensation	52.42	56.50	8.94	69.97	69.00	8.74
	Ultrasonic	74.20	73.30	6.79	92.30	93.00	9.83
Grey ProRoot-MTA	Trituration	40.46	40.30	3.57	61.03	60.10	8.23
	Condensation	50.65	50.90	6.66	59.46	60.60	7.26
	Ultrasonic	43.81	43.95	4.98	57.52	54.40	16.56
White MTA-angelus	Trituration	38.48	37.50	3.17	50.28	50.50	4.57
	Condensation	36.32	34.40	11.99	38.26	38.00	3.58
	Ultrasonic	43.82	42.70	10.99	49.71	45.40	10.70
Grey MTA-angelus	Trituration	38.60	40.00	5.96	63.32	62.70	4.99
	Condensation	55.24	53.85	5.38	75.49	74.55	4.25
	Ultrasonic	73.06	71.00	10.62	84.33	85.45	19.94

MTA, mineral trioxide aggregate.

trituration and condensation methods at 4 and 28 days ($P < 0.0001$).

For ProRoot grey at 4 days after mixing, (Table 1) a significantly higher ($P < 0.005$) difference was found between the condensation mixed group compared to trituration and ultrasonic experimental groups. At 28 days, no statistically significant difference existed between the mixing techniques.

For Angelus white MTA at 4 days after mixing (Table 1), no significant differences were found between ($P < 0.005$) surface microhardness compared to both other experimental groups. No significant difference the experimental groups. However, after 28 days, the condensation technique revealed a significantly lower in surface microhardness was found between trituration and ultrasonic mixing.

A significant difference ($P < 0.0001$) was found between all mixing techniques at 4 and 28 days for Angelus Grey (Table 1), with ultrasonic mixing producing the highest surface microhardness values.

Figure 2 shows the surface microhardness according to the material irrespective of the mixing technique. A significant difference ($P < 0.0001$) was observed in surface microhardness between all types of MTA apart from between Angelus grey and ProRoot white at both 4 and 28 days after mixing, both of which produced the highest values.

Discussion

Mineral trioxide aggregate is a biocompatible material (Shahi *et al.* 2006) that is increasingly used in endodontic treatment. Despite its advantageous biocompatibility and sealability (Stefopoulos *et al.* 2008), the handling properties and prolonged setting time of MTA have been described as disadvantages (Antunes Bortoluzzi *et al.* 2006).

The hydration reaction of MTA results in the formation of hydrated compounds whose strength increases with time (Lee *et al.* 2004, Nekoofar *et al.* 2007). The effect of condensation pressure, trituration of encapsulated MTA and ultrasonic vibration on the surface microhardness of various types of MTA was evaluated in the present study. The results showed that the surface microhardness value of all experimental groups increased significantly after 28 days (Fig. 1). The surface microhardness value of a material is not a measure of a single property and is influenced substantially by other fundamental properties of the material such as crystal structure stability (Taylor 1997, Hewlett 2004). Thus, it can be used as an indicator of the hydration process of hydraulic cements and their setting process (Lee *et al.* 2004, Camilleri 2007). It can also indicate the effect of various setting conditions on the overall strength of a material (Namazikhah *et al.* 2008). The hydration process is a complex phenomenon that if modified might influence the biological, chemical and physical properties of the resulting product (Camilleri 2007). Fridland & Rosado (2003) studied the effect of various water-to-powder ratios of MTA on its solubility and porosity. They reported that an increased water-to-powder ratio resulted in higher solubility, porosity and release of calcium hydroxide. The latter is the main compound

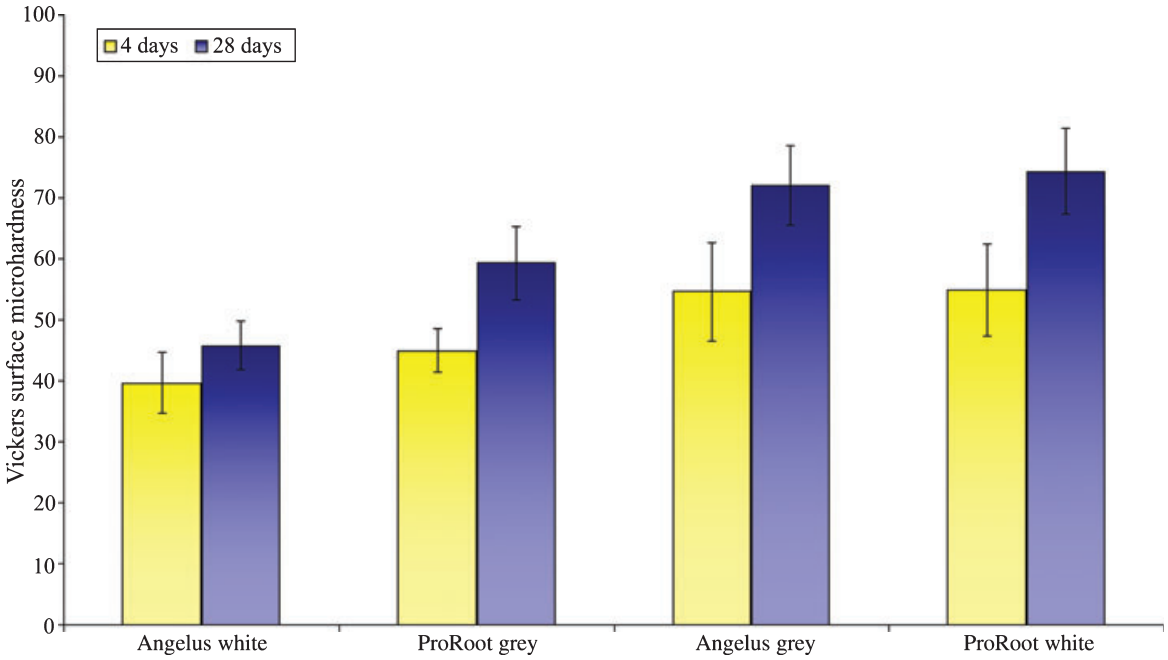

Figure 2 The Vickers surface microhardness value of each type of mineral trioxide aggregate irrespective of the mixing technique employed.

released by MTA in an aqueous environment (Fridland & Rosado 2003, Camilleri 2008) that might be advantageous. However, the presence of excess water in the mixture might cause difficulty in handling and placement of the material. The amount of water mixed must be balanced with the requirements of the material such as bioactivity, handling and physical properties. Nekoofar *et al.* (2009) reported an inconsistency in the amount of water supplied within ProRoot MTA packages. This variation in the volume of water might produce uncontrolled and undesirable characteristics in some clinical and laboratory situations, such as incomplete setting and poorer handling properties. Encapsulation of pre-set proportions of MTA powder and water appears advantageous as it enables the powder to liquid ratio and the mixing technique to be regularized by the manufacturer. In turn, this reduces the variability that might occur when the material is dispensed and mixed conventionally. However, the results of the present study showed that trituration of customized encapsulated MTA did not improve surface microhardness value compared to other experimental groups regardless of the type of MTA used. In terms of consistency, the materials mixed using the novel trituration method (patent pending) were subjectively found to be consistently creamier and of a less grainy quality that made handling more controllable. To quantify these handling characteristics, further rheological investigations are recommended.

To achieve optimum properties, the hydraulic cement particles should be thoroughly mixed with water. The method chosen for mixing a material is fundamental to produce effective contact between powder particles and liquid and a set material with optimum physical, chemical and biological properties (Nomoto *et al.* 2004). Aminoshariae *et al.* (2003) compared the effect of ultrasonic and hand condensation on the adaptation of MTA to experimental polypropylene containers as well as the occurrence of voids within the material. They concluded that ultrasonic techniques resulted in more voids than hand condensation. The presence of voids might not be a disadvantage for the MTA hydration process as they might provide pathways for the water to diffuse into the material (Fridland & Rosado 2003, 2005, Nekoofar *et al.* 2007, Namazikhah *et al.* 2008). In contrast, the results of the present study demonstrated that ultrasonic vibration produced a significantly higher surface microhardness value compared to condensation and mechanical trituration (Fig. 1). This could be explained by the dispersion effect of the ultrasonic energy on the material particles that might provide sufficient space for

water molecules and better water diffusion producing a greater degree of hydration and subsequently a higher surface microhardness value. Total reactive surface area and particle interaction are increased by ultrasonic energy and might decrease setting time (Kleverlaan *et al.* 2004, Algera *et al.* 2005). In addition, ultrasonic vibration, by changing the rheological properties of a material, might also improve their handling characteristics (Witherspoon & Ham 2001, Lawley *et al.* 2004, Schmidlin *et al.* 2005). Yeung *et al.* (2006) in their *ex-vivo* study compared the fill density of MTA in simulated straight and curved canals using hand condensation and indirect ultrasonic vibration. They reported a heavier and denser filling in the latter group, suggesting the beneficial effects of ultrasonic vibration on MTA that is in accordance with the results of the present study. In addition, the advantages of the application of ultrasonic vibration were shown by Lawley *et al.* (2004). They evaluated the effect of ultrasonic energy on MTA in relation to bacterial penetration in an apexification model and found that it improved significantly the seal after 45 days. In the studies of Aminoshariae *et al.* (2003) and Yeung *et al.* (2006), the amount of pressure applied during condensation was an uncontrolled variable. Nekoofar *et al.* (2007) demonstrated that higher condensation pressures during placement of MTA produced lower surface microhardness values, suggesting that high condensation pressure affects the hydration, strength and surface microhardness of the material. Optimum physical properties were reported at a condensation pressure of 3.22 MPa (Nekoofar *et al.* 2007), which was the selected pressure used in the present study for the condensation experimental group. The results of the present study demonstrated that even though ultrasonic vibration produced a significantly higher surface microhardness value compared to condensation and mechanical trituration, no statistical difference was observed in surface microhardness value between the two latter experimental groups. The findings for the condensation group of MTA might be explained by the concept that compacting the material limited the formation of microchannels, compromising the ingress of water to hydrate the material adequately. In relation to the type of MTA at 4 days, the ultrasonic experimental group showed significantly higher surface microhardness value in all four types of MTA used. At 28 days after mixing, ultrasonic application produced the highest surface microhardness for ProRoot white and Angelus grey. Whilst for ProRoot grey and Angelus white, trituration technique produced the highest surface microhardness value; however, the differences were not significant compared to the ultrasonic groups (Fig. 2).

Regardless of the mixing technique employed, the Vickers surface microhardness value of each type of MTA is illustrated in Fig. 2. No significant difference was observed between Angelus grey and ProRoot white at both 4 and 28 days after mixing. Angelus white produced a significantly lower surface microhardness value compared to all experimental groups ($P < 0.0001$) that might be related to its rapid setting because surface microhardness value of a cement is an indication of its adequate hydration. However, this is not in accordance with Angelus grey that sets more quickly than ProRoot white and which both produced the highest surface microhardness values.

Conclusions

The application of ultrasonic energy to MTA produced a significantly higher surface microhardness value compared to other experimental groups at both 4 and 28 days after mixing. Regardless of the mixing techniques used, ProRoot white and Angelus grey produced the highest surface microhardness values. Trituration of encapsulated, premeasured MTA and water provides a consistent method of mixing that produces MTA slurries with more controllable handling characteristics.

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