The Effect of pH on Surface Hardness of Mineral Trioxide Aggregate with Various Additives

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Citation

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Abstract

Background and Aim
:
To evaluate the surface microhardness of mineral trioxide aggregate (MTA) mixed with various additives after exposure of their surface to acidic pH during hydration.Materials and Methods
: White ProRoot MTA was mixed with various additives like saline, lidocaine, 2% calcium chloride and distilled water and packed into cylindrical polycarbonate tubes. Two groups, each of 32 specimens was prepared, with four subgroups and 8 samples under each subgroup. The two groups were exposed to 4.4 and 7.4 pH for 4 days. Vickers microhardness of the surface of each specimen was measured after exposure. The data obtained were subjected to one-way ANOVA and post hoc Tukey's test.Results
: The greatest mean surface hardness value (74.4 ± 4.6) was observed following exposure to pH 7.4 of MTA with 2% $CaCl_2$ and least value (17.2 ± 2.7) with saline. Tukey post-hoc test revealed that difference between the values of specimens for $CaCl_2$, saline, lidocaine and distilled water were statistically significant ($p< 0.05$). But among saline and distilled water at pH 4.4, it was not statistically significant.Conclusion
: Under the conditions of this study, surface hardness of MTA with 2%$CaCl_2$ was not impaired in an acidic environment.

INTRODUCTION

MTA was developed at the Loma Linda University in 1990s (1). It was first described in the dental scientific literature in 1993 (2) and was given approval for endodontic use by the U.S Food and Drug Administration in 1998 (3). A patent was taken out for MTA in 1995. This states that MTA consists of 50% – 75% (by weight) calcium oxide and 15% - 25% silicon dioxide. These two components together compromise 70-95% of the cement.

When these raw materials are blended, they produce tricalcium silicate, dicalcium silicate and tricalcium aluminate. On addition of water, the cement hydrates to form silicate hydrate gel. The physiochemical basis for the biological properties of MTA is attributed to the production of hydroxyapatite when the calcium ions released by the MTA came into contact with tissue fluid. These released calcium ions promotes alkaline pH. MTA has been therefore utilized to repair root perforation; as root end filling material; for pulp capping and partial pulpotomy procedures (4, 5). In addition, because of its sealing ability, it was also suggested as an apical barrier in the treatment of teeth with open apices and necrotic pulp (6, 7).

The hydration rate is characteristic of the progress of cement setting (8). Sufficient water is required during the setting of the cement. The use of MTA as a root end filling was identified, as it sets in the presence of water.

MTA is the material of choice for sealing perforation, and as a retrograde filling material. It is also used for vital pulp therapy, as a barrier during internal bleaching of discoloured tooth and repair of vertical fractures (5). However, it also has some shortcomings; such as extended setting time, which favours its solubility, disintegration or dislodgement of the material. Its granular consistency complicates its insertion in cavities (9). In many clinical applications, MTA is placed in an environment where inflammation is present and low pH is likely (10). Torabinejad et al (1995) demonstrated that MTA has a pH of 10.2 initially, which increases to 12.5 three hours after mixing. There may be variation in the pH value of host tissue response because of pre-existing pathological conditions, which affects its physical and chemical

properties (11).

Recently, number of studies was made to improve the physical and chemical properties of MTA. Because MTA cement contains 75-80% Portland cement (12), new additives used in civil engineering are being studied to solve the clinical deficiencies.

An acidic pH in the environment impedes MTA setting, and reduces its strength and hardness (8). An S.E.M. study by Namazikhah et al evaluated the morphological and microstructural features of samples immersed in different pH and their response to MTA was analyzed. Under the conditions of his study, surface hardness of MTA was impaired in an acidic environment. MTA was mixed with distilled water in this study mentioned. However, MTA mixed with other commonly available additives may increase its compressive strength.

The aim of the current study was to evaluate the effect of pH on surface hardness of MTA mixed with various additives.

MATERIALS AND METHODS

The parameter investigated was Surface Hardness (Vickers microhardness), and the material investigated was tooth coloured formula of ProRoot MTA (Dentsply Tulsa Dental, Johnson City, TN, USA).

MICROHARDNESS

The material was mixed according to manufacturer's instruction. Each sample of MTA was mixed with recommended volume of water, same volume of 2% $CaCl_2$, lidocaine and saline.

Eight specimens were obtained under each group. The mixed material was weighed and packed into polycarbonate tube of internal diameter 6mm and height of 5mm. Two groups each of 24 specimens were prepared using a pressure of 3.22 MPa applied for 1min (13). The samples were then subjected to a constant vertical force using amalgam condenser containing internal diameter similar to that of the cylindrical tubes. A wet cotton pellet was placed onto the MTA within polycarbonate tube and samples were stored at room temperature (30°) within glass vials for 4 days. The bottom of each vial contained a piece of 2×2 cm gauge that had been soaked in butyric acid buffered at pH 4.4 and 7.4. The latter acted as control group. Based on pilot experimentation the acid soaked piece of gauge were replaced with fresh acid every 24 hrs to ensure consistent pH during experiment period. The opening of glass vials was then covered with moist gauge to ensure the presence of sufficient humidity inside the vial.

After 4 days, MTA was replaced from the vial. The surface exposed to acid on each specimen were then wet polished at room temperature using minimum hand pressure and silicon carbide based sand paper of varying particle size (600, 800 and 1200 grit) to impart a smooth surface for ease of indentation testing. Polished specimens were cleaned gently under light pressure distilled water to remove surface debris and then gently air dried (14).

The Vickers microhardness test of each specimen was performed using Leica microhardness tester (Leica Microsystems GmbH, Wetzlar, Germany) and square based diamond indenter with full load of 50gms for 5 seconds at room temperature (15) that produced quadrangle depression with two equal orthogonal diagonals.

Five indentations were made on polished surface of each specimen at separate location 1mm apart on the specimen area. Vickers microhardness reading was recorded for each specimen.

STATISTICS

One-Way ANOVA and Post-Hoc Tukey Test analyzed differences between the experimental groups after calculating the Mean ± Standard Deviation.

RESULTS

MICROHARDNESS

Mean of the microhardness values obtained under each group with their standard deviation at 95% confidence interval are listed in table 1 and graphically depicted in graph 1. A mean of 74.4 ± 4.6 was obtained for the $CaCl_2$ group at pH 7.4, which was the highest value obtained among all the subgroups and groups. A mean of 17.2 ± 2.7 was obtained for the saline group at pH 4.4, which was the lowest value obtained among all the subgroups and groups. The difference between means was analyzed by One-Way ANOVA and it showed that the difference between means was statistically significant for pH 7.4 and for pH 4.4.

Comparison between means of the subgroups under each group were done using Tukey post-hoc test. At pH 7.4, there was no statistically significant difference between lidocaine and distilled water (P = 0.776), but there was statistically significant difference seen between all the other subgroups. At pH 4.4, there was no statistically significant difference between saline and lidocaine (P = 0.166) and between

lidocaine and distilled water (P = 0.116). But there was statistically significant difference seen between all the other subgroups.

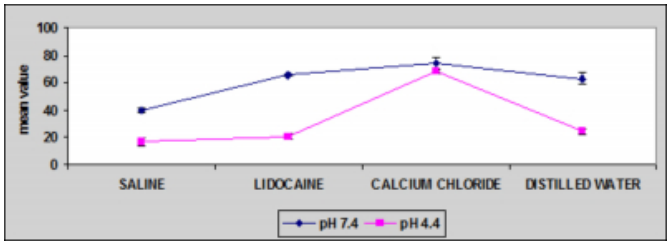
Figure 1

Table (1): Mean surface microhardness of specimens.

Additives	Surface Microhardness (MPa)	
	pH 4.4 Mean ± Std.Deviation	pH 7.4 Mean ± Std.Deviation
Saline	17.2±2.7	39.7±1.6
Lidocaine	20.6±2.4	65.2±0.37
2%Calcium chloride	68.1±1.3	74.4±4.6
Distilled water	24.5±2.1	63.1±3.8

Figure 2

Fig (1): Mean surface microhardness of specimens with their SD at pH 4.4 and 7.4



DISCUSSION

Mineral trioxide aggregate is the material of choice for root perforations (2), retrograde fillings (16, 17), apexification (6, 7) and vital pulp therapy (4, 5). MTA has been shown to release soluble fractions of CaOH in both short and long term (18), sufficient to maintain pH of the surrounding environment at a high level (pH 11-12).

MTA powder when combined with various additives improves the working properties of the mixture (19). Studies done by Namazikhah et al. (14) showed that MTA placed under acidic pH 4.4 significantly affected its microhardness and as the pH was increased the surface microhardness also increased. He also stated that at pH 4.4, physical properties of MTA mixed with distilled water were significantly affected when compared to MTA kept under pH 5.4 and 6.4. To overcome this, MTA was mixed with other commonly available additives like saline, lignocaine and calcium chloride, which are easily available in clinical practice.

Microscopic analysis of hand placement method of placing MTA in 5mm polycarbonate tube showed less or no voids (20) when compared to ultrasonic condensation.

It is recommended by Song et al. (21) that MTA be allowed to set untouched for 72hrs or longer to decrease the chance of MTA displacement. Hence, for attainment of maximum strength specimens were kept for 4 days in the study. To simulate clinical conditions, MTA was kept in contact with wet cotton pellet on one side for attainment of maximum strength. In this study, butyric acid – a byproduct of anaerobic bacterial metabolism (22, 23) was used to simulate the clinical condition of inflamed periradicular tissue.

Microhardness of a material is not a measure of single property. It is influenced by other fundamental properties of materials such as yield strength, tensile strength, modulus of elasticity (24) and crystal structure stability (25). It is an indicator of setting process and overall strength or resistance to deformation. It can also indicate the effect of various setting condition on the overall strength of the material.

Vickers microhardness test in this study showed that when MTA mixed with $CaCl_2$ at pH 4.4 showed maximum strength compared to others in the same group and there was also not much difference in its microhardness value at pH 7.4. $CaCl_2$ mixed with MTA has already been confirmed to be nontoxic to human cells in vitro (26). An accelerated 35 minute initial setting time was also observed with $CaCl_2$ mixed MTA (27). However, this might not be considered by clinicians to be fast enough to provide significant clinical benefits. The $CaCl_2$ by itself had no effects on handling characteristics either. A significant increase in calcium release was seen during the first 24 hours, when $CaCl_2$ was added to White MTA (28). Also, it has been confirmed that high amounts of calcium in a cell culture environment might down-regulate cell proliferation (29). It was also seen that greater than 2% $CaCl_2$ adversely affects the cement by increasing the risk of drying shrinkage and reducing ultimate strength (30). MTA when mixed with saline did not set completely even after 4 days of the experiment and at a pH of 4.4 showed the least Vickers microhardness value. In conclusion, the surface hardness of MTA with 2% $CaCl_2$ was not seen to be impaired in an acidic environment.

References

1. Camilleri and Pitt Ford TR: Mineral trioxide aggregate: a review of the constituents and biological properties of the material. Int Endod J; 2006; 39: 747-54.
2. Lee S J, Monsef M, Torabinejad: Sealing ability of MTA for the repair of lateral root perforation. J Endod; 1993; 19: 541-45.
3. Schmitt D, Bogen G: Multifaceted use of pro-root MTA

root canal repair material. Pediatr Dent; 2001; 23: 326-30.
4. Abedi HR, Ingle JI: Mineral trioxide aggregate: a review of a new cement. J Calif Dent Assoc; 1995; 23: 36-39.
5. Torabinejad M, Chivian N: Clinical applications of mineral trioxide aggregate. J Endod; 1999; 25: 197-205.
6. Shabahang S, Torabinejad M: Treatment of teeth with open apices using mineral trioxide aggregate. Pract Periodontics Aesthet Dent; 2000; 12: 315-20.
7. Witherspoon DE, Ham K: One-visit apexification: technique for inducing root-end barrier formation in apical closures. Pract Proced Aesthet Dent; 2001; 13: 445-60.
8. Taylor HFW: Cement Chemistry; 1997; 2nd edn. London Thomas Telford Ltd.
9. J Endod; 1995; 21: 228.
10. Cummings GR, Torabinejad M. Mineral trioxide as an isolating barrier for internal bleaching. J Endod 1995; 21:228.
11. Malamed SF: Local anaesthetic considerations in dental specialties. Handbook of local anaesthesia; 4th edn. St.Louis, Mosby-Year Book; 1997.
12. Lee YL, Lee BS, Lin FH, Yun Lin A, Lan WH, Lin CP: Effects of physiological environments on the hydration behaviour of mineral trioxide aggregate. Biomaterials; 2004; 25: 787-93.
13. Bortoluzzi EA, Broon NJ, Duarte MAH, Demarchi: ACCO, Bramante CM: The use of setting accelerator and its effect on pH and calcium ion release of Mineral trioxide aggregate and White Portland cement. J Endod; 2006; 32: 1194-97.
14. Nekoofar MH, Adusei G, Sheykhrezae MS, Hayes SJ, Bryant ST, Dummer PM: The effect of condensation pressure on selected physical properties of mineral trioxide aggregate. Int Endod J; 2007; 40: 453-61.
15. Cross WM, Sabnis KH, Kjerengtroen L, Kellar JJ: Microhardness testing of fiber-reinforced cement paste. ACI Materials journal; 2000; 97: 162.
16. Namazikhah MS, Nekoofar MH, Sheykhrezae MS, Salariyeh S, Hayes SJ, Bryant ST et al: The effect of pH on surface hardness and microstructure of mineral trioxide aggregate. Int Endod J; 2008; 41: 108-116.
17. Torabinejad M, Watson TF, Pitt Ford TR: Sealing ability of a mineral trioxide aggregate when used as a root end filling material. J Endod; 1993; 19: 591-5.
18. Aqrabawi J: Sealing ability of amalgam super EBA cement and MTA when used as retrograde filling materials. Br Dent J; 2000; 188: 266-8.
19. Fridland M, Rosado R: Mineral trioxide aggregate MTA solubility and porosity with different water-to-powder ratios. J Endod; 2003; 29: 814-7.
20. Kogan PJ, Glickman G N, Wantabe I: Effect of various additives on sealing properties of MTA. J Endod; 2006; 32; 567-72.
21. Aminoshariae A, Hartwell G, Moon P: Placement of Mineral Trioxide Aggregate using two different techniques. J Endod; 2003; 29(10): 679-683.
22. Song JS, Mante FK, Romanow WJ, Kim S: Chemical analysis of powder and set forms of Portland cement, gray ProRoot MTA, white Proroot MTA, and gray MTA-Angelus. Oral Surg Oral Med Oral Pathol Oral Radiol Endod; 2006; 102: 809-15.
23. Zeikus JG: Chemical and fuel production by anaerobic bacteria. Annual. Review of Microbiology; 1980; 34: 423-64.
24. Barker HA: Amino acid degradation by anaerobic bacteria. Annual Review of Biochemistry; 1981; 50: 23-40.
25. Bentz DP: Cement hydration: building bridges and dams at the microstructure level. Materials and Structures; 2007; 40: 397-404.
26. Gilman JJ: Chemical and physical hardness. Materials Research Innovations; 1997; 1: 71-6.
27. Abdullah D, Pitt Ford TR, Papaioannou S, Nicholson J, McDonald F: An evaluation of accelerated Portland cement as a restorative material. Biomaterials; 2002; 19: 4001-10.
28. Wiltbank KB, Schwartz SA, Schindler WG: Effect of selected accelerants on the physical properties of Mineral Trioxide Aggregate and Portland Cement. J Endod; 2007; 33(10): 1235-39.
29. Antunes Bortoluzzi E, Juarez Broon N, Antonio Hungaro Duarte M, de Oliveira Demarchi AC, Monteiro Bramante C: The use of a setting accelerator and its effect on pH and calcium ion release of mineral trioxide aggregate and white Portland cement. J Endod; 2006; 32: 1194–7.
30. Midy V, Dard M, Hollande E: Evaluation of the effect of three calcium phosphate powders on osteoblast cells. J Mater Sci Mater Med; 2001; 12: 259–65.
31. Kosmatka S, Pnarese W: Design and control of concrete mixtures. 13th ed. Skokie IL: Portland Cement Association; 1988.

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