Microhardness Properties of Mineral Trioxide Aggregate and Calcium-enriched Mixture Cement Plugs at Different Setting Conditions

Mehdi Tabrizizadeh¹, Mohammad Mahdi Dabbagh², Hamid Badrian³, Amin Davoudi⁴

Contributors:
¹Associate Professor, Department of Endodontics, Faculty of Dentistry, Shahid Sadoughi University of Medical Sciences, Yazd, Iran; ²Post Graduate Student, Department of Endodontics, School of Dentistry, Shahid Sadoughi University Medical Sciences, Yazd, Iran; ³Post Graduate Student, Department of Operative Dentistry, School of Dentistry, Shahid Sadoughi University Medical Sciences, Yazd, Iran; ⁴Dentistry Student, Dental Students Research Center, School of Dentistry, Isfahan University of Medical Sciences, Isfahan, Iran.

Correspondence:
Dabbagh MM. Department of Endodontics, School of Dentistry, Shahid Sadoughi University Medical Sciences, Daheye Fajr Street, Yazd, Iran. Tel.: +98-9132773114. Email: M.dabbagh91@yahoo.com

How to cite the article:
Tabrizizadeh M, Dabbagh MM, Badrian H, Davoudi A. Microhardness properties of mineral trioxide aggregate and calcium-enriched mixture cement plugs at different setting conditions. J Int Oral Health 2015;7(9):36-39.

Abstract:
Background: Providing an apical stop in open apex roots is one of the endodontic challenges. The aim of present study was to compare the surface hardness of both mineral trioxide aggregate (MTA) and calcium-enriched mixture cement (CEM) cement at different setting situations.

Materials and Methods: A total of 40 freshly extracted human teeth with a single root and normal apex and no obvious caries or curvatures were selected. The teeth were sectioned horizontally from the cement enamel junction and 2 mm above the apex. Standard technique was administered for cleaning and shaping of the canals. Open apex root canal was prepared by using Peso reamer. The prepared teeth were randomly divided into four groups in which two groups were filled by MTA and the other groups were filled by CEM. White MTA and CEM cement plugs were prepared and condensed up to the apical end. In two groups, moistened paper point was placed in the canals and in the other group dried paper points was used. Vickers test was done to evaluate the microhardness and the collected data were analyzed by Kruskal-Wallis and three-way ANOVA tests using SPSS software version 18 at a significant level of 0.05.

Results: The highest and lowest surface hardness was observed in CEM group at dried condition with 4 mm thickness indentation (145.10 ± 7.60 kg/mm²) and moist MTA group indented at 8 mm thickness (111.25 ± 5.37 kg/mm²). However, no significant difference was noticed (P > 0.05).

Conclusion: Humidity condition might not influence the microhardness properties of both MTA and CEM cement apical plugs at different tested indentation thickness.

Key Words: Apexification, calcium-enriched mixture cement, microhardness, mineral trioxide aggregate

Introduction
One of the endodontic challenges is to provide an apical stop in open apex roots either in immature tooth or mature tooth with apical root resorption. One of the suggested treatment plans is to induce preparation of a physiologic barrier by applying and further renewing of calcium hydroxide or other temporary endodontic materials for a long period of time.¹,² However, prolonged treatment visits and higher failure rate are important disadvantages of mentioned method.³ Mineral trioxide aggregate (MTA) has demonstrated a promise result as a root filling material which has a long setting time and needs humidity for setting.⁴⁻⁶ Calcium-enriched mixture (CEM) cement is another endodontic material which presents valuable properties similar to MTA but with easier manipulation and lower price.⁷⁻⁹ The remarkable advantage of using MTA and similar dental materials is significant reduction in duration of apexification treatment. However, the challenge is to ensure sufficient moisture for final setting and hardening. Another challenge is the possibility of displacement of apical plugs before final setting and during filling of the coronal regions. Although contemporary filling methods, such as thermo plasticized technique, might induce lower apical pressure than lateral or vertical condensation technique, filling of the canal with MTA might be another possible solution.⁴⁻¹⁰

According to the released literature, surface hardness is an indicator for measuring the hardness quality of MTA,¹¹⁻¹² as well as proper thickness of 4-5 mm above the apical plug.⁵ However, uncertainty still remains about using high thickness of MTA for final filling of the root canal.⁴ Since CEM cement have been introduced as a contemporary material and limited studies have been dedicated to its mechanical properties as a root canal filling material, the aim of the present study was to compare the surface hardness of both MTA and CEM cement at different setting situations.

Materials and Methods
Sample preparation
In this analytical-observational in vitro study, 40 freshly extracted human teeth with a single root and normal apex, and no obvious caries or curvatures were selected. Radiographs were taken to assure the existence of single root canal with appropriate length and diameter, and to roll out teeth with...
canal calcifications, internal resorption, or any abnormality. After removing debris or attached tissues, the teeth were decontaminated by sodium hypochlorite 5.25% (Chloran, Tehran, Iran) for 1-h, then transferred to the normal saline solution. The teeth were sectioned horizontally from the cement enamel junction by diamond burs (Drenal and Zweiling Diament, Lemgo, Germany). Furthermore, 2 mm above the apex was sectioned to remove any possible apical deltas or accessory canals. Working length was measured by using K-file size # 15 (Mani, Tochgi, Japan) until observing the file's tip at the apex under ×10 magnification. After enhancing straight-line access, standard technique was administered for cleaning and shaping of the canals by using K files size # 25-50. To prepare an open apex root canal, Peso reamer size # 1-4 (Mani peso reamer, Mani Inc., Tochgi, Japan) was used to provide cylindrical shape canal, with 1.3 mm diameter, from the coronal to the apical. Sodium hypochlorite 2.5% was used for canal irrigation during all steps of canal preparation.

**Grouping category**
The prepared teeth were mounted in wet flower sponges and randomly divided into four groups (n = 10) in which two groups were filled by MTA and the other two groups were filled by CEM. Canals were dried with paper points (Ariadent, Tehran, Iran). White MTA (Angelus, Londrina, PR, Brazil) was prepared based on manufacturer’s instruction and was transferred into the canals with MTA carrier (Dentsply Maillefer, Ballaigues, Switzerland). The MTA plugs were gently condensed up to the apical end by using hand plungers (size # 4) (Dentsply Maillefer, Ballaigues, Switzerland) with a rubber stop positioned 9 mm shorter than the working length. In one group, moistened paper point was placed in the canals and in the other group dried paper points was used. Then the density, quality and thickness of MTA plugs were observed by radiographs. Temporary restoration material (Coltosol; Ariadent, Tehran, Iran) was used to restore the access cavities. The same procedure was executed for CEM cement (Bionique Dent, Tehran, Iran) groups. Finally, all of the specimens were stored at 37°C and 100% humidity for 21 days.

**Surface hardness evaluation**
To prepare the specimens for Vickers test, they were mounted in self-cured acrylic resin (Asia ChemiTeb Co., Tehran, Iran). Then, the specimens were split longitudinally and their surfaces were rinsed by normal saline to remove any remained debris for 1 min. The procedure was followed by polishing the surface and using 300-1200 grit papers. The specimens were subjected to Vickers test (Micromet, Buehler Ltd., IL, USA) based on ISO/IEC 17025 under 500 g force. Due to the square pyramid indenter shape of the device, a rectangular recess was prepared on the surface of polished surfaces. The angle between the opposite faces of the diamond indenter is 136°. Hence, at 4 and 8 mm thickness of the polished surface, three indentations were created randomly with minimum distance from each other. The diameter of the resulting indentation was measured immediately under ×40 magnification and the final surface hardness calculated based on below formula:

\[
HV = \frac{2F \sin 136^\circ}{d^2} = 1.854 \frac{F}{d^2}
\]

"F" stands for the loaded force (kg) and "d" is the mean of indentation diameter (mm).

At last, the collected data were analyzed by Kruskal-Wallis and three-way ANOVA tests using SPSS software version 18 at a significant level of 0.05.

**Results**
To simplify the comparison, three variables were defined as follow: V1 as tested materials (MTA and CEM), V2 as the moist condition of test materials (dry and moist), and V3 as the thickness of indentation (4 mm and 8 mm).

Table 1 manifests the mean surface hardness of studied groups at different conditions. Based on analyzed results, the highest surface hardness was observed in CEM group at dried condition and 4 mm thickness indentation (145.10 ± 7.60 kg/mm²). Furthermore, the lowest value belonged to moist MTA group indented at 8 mm thickness (111.25 ± 5.37 kg/mm²). However, no significant difference was noticed among different groups of study (P > 0.05).

**Discussion**
According to the results of present study, no significant difference was observed between the use of CEM and MTA, as root-end filling materials, in one-visit or two-visit endodontic treatments in terms of quality and hardness.

Despite the recommendation of applying moisturized cotton for two-visit apexification therapies, some studies stated that the periapical interstitial fluid provides enough moisture for the condensed apical plug to become hard enough. Nevertheless, controversies are still remained about the application of higher thickness of the apical plug in one-visit apexification therapies. Shokouhinejad et al. concluded that the placement of moisturized cottons is essential ProRoot MTA plugs with 4-6 mm thicknesses. On the other hand, DeAngelis et al. claimed that using moisturized cottons is not necessary for 4 mm plugs of MTA Angelos when the diameter of apical perforation site is more than 1 mm. Even one study indicated that applying dry powder pack of MTA in open apex root canal could be set only by absorbing moisture from the periapical tissue. Variation in the results might be due to the design of the study. Shokouhinejad et al. used cylindrical resin samples resembled to open apex root canals. Cementum permeability
seems to provide moisture especially in young sound teeth. However, they were different to clinical situations as by using resin samples absorption of moist would be limited only to the apical region, not axial walls. Therefore, dentin tubules of axial walls, especially in young root canals, might provide an important source of water supply required for setting of apical plugs. Furthermore, the capillary rise mechanism might help the moist transformation from the apical sites. That’s why the teeth were decoronated and mounted in flower wet sponges in the present study to provide sufficient moisture for plug settings.

The mean hardness of present study was higher than similar articles. The values of present study were more than 100 kg/mm², however, similar experiments reported the range of 60-90 kg/mm². In a study on the hardness of CEM, the average reported value was in the range of 3-9 kg/mm². Since the numerical values might be influenced by various factors, the normal distribution of recorded data seems to be more important. For instance, in the mentioned study MTA Angelos, the mean hardness was 78 kg/mm² but they were varied from 25 to 188 kg/mm², which might criticized the required equal situation for each experiment. Another issue that must be considered is that the physical strength of MTA might be increased over time and its maximum physical straight is reported after 21 days. Hence, the samples were incubated for 21 days in current study, in contrast to other experiments in which 3-7 days incubation was ordered.

**Conclusion**

Within the limitations of in vitro studies, it can be concluded that the humidity situation might not effectively influence the microhardness properties of both MTA and CEM cement apical plugs at different tested indentation thickness. However, further study is recommended to reach out determinant and comprehensive conclusion.

**Acknowledgments**

The current study was based on a thesis with ID number of 3442 which has been approved by Ethical Research Committee of Shahid Sadoughi University of Medical Science.

**References**

1. Steiner JC, Dow PR, Cathey GM. Inducing root end closure of nonvital permanent teeth. J Dent Child 1968;35(1):47-54.
2. Sonarkar S, Purba R. Bioactive materials in conservative dentistry. Int J Contemp Dent Med Rev 2015;2015:Article ID: 340115. doi: 10.15713/ins.ijcdmr.47.
3. Andreasen JO, Farik B, Munksgaard EC. Long-term calcium hydroxide as a root canal dressing may increase risk of root fracture. Dent Traumatol 2002;18(3):134-7.
4. Parirokh M, Torabinejad M. Mineral trioxide aggregate: A comprehensive literature review – Part III: Clinical applications, drawbacks, and mechanism of action. J Endod 2010;36(3):400-13.
5. Chávez-Andrade GM, Kuga MC, Duarte MA, Leonardo Rde T, Keine KC, Sant’Anna-Junior A, et al. Evaluation of the physicochemical properties and push-out bond strength of MTA-based root canal cement. J Contemp Dent Pract 2013;14(6):1094-9.
6. Khandelwal A, Karthik J, Nadig RR, Jain A. Sealing ability of mineral trioxide aggregate and biodentine as root end filling material, using two different retro preparation techniques – An in vitro study. Int J Contemp Dent Med Rev 2015;2015:Article ID: 150115. doi: 10.15713/ins.ijcdmr.48.

---

| Table 1: The mean surface hardness (kg/mm²) of studied groups at different conditions. |
| Variables | Mean±SD | P value |
| --- | --- | --- |
| **V1** | | 0.95 |
| CEM | 129.82±3.80 | |
| MTA | 129.50±3.80 | |
| **V2** | | 0.13 |
| Dry | 139.20±3.80 | |
| Moist | 120.12±3.80 | |
| **V3** | | 0.26 |
| 4 mm | 135.87±3.80 | |
| 8 mm | 123.45±3.80 | |
| **V1 and V2** | | 0.28 |
| CEM | | |
| Dry | 141.55±5.37 | |
| Moist | 136.85±5.37 | |
| MTA | | |
| Dry | 118.10±5.37 | |
| Moist | 122.15±5.37 | |
| **V1 and V3** | | 0.50 |
| CEM | | |
| 4 mm | 135.00±5.37 | |
| 8 mm | 124.65±5.37 | |
| MTA | | |
| 4 mm | 136.75±5.37 | |
| 8 mm | 122.25±5.37 | |
| **V2 and V3** | | 0.23 |
| Dry | | |
| 4 mm | 142.75±5.37 | |
| 8 mm | 135.65±5.37 | |
| Moist | | |
| 4 mm | 129.00±5.37 | |
| 8 mm | 111.25±5.37 | |
| **V1 and V2 and V3** | | 0.70 |
| CEM | | |
| Dry | | |
| 4 mm | 145.10±7.60 | |
| 8 mm | 138.00±7.60 | |
| Moist | | |
| 4 mm | 140.40±7.60 | |
| 8 mm | 133.30±7.60 | |
| MTA | | |
| Dry | | |
| 4 mm | 124.90±7.60 | |
| 8 mm | 111.30±7.60 | |
| Moist | | |
| 4 mm | 133.10±7.60 | |
| 8 mm | 111.20±7.60 | |

MTA: Mineral trioxide aggregate, CEM: Calcium-enriched mixture, SD: Standard deviation
7. Attavar SH, Nadig P, Sujatha I. Management of open apex with mineral trioxide aggregate – 2 case reports. Int Dent Med J Adv Res 2015;1:1-4.

8. Asgary S, Shahabi S, Jafarzadeh T, Amini S, Kheiriez S. The properties of a new endodontic material. J Endod 2008;34(8):990-3.

9. Chakraborty A. Will portland cement be a cheaper alternative to mineral trioxide aggregate in clinical use? A comprehensive review of literature. Int J Contemp Dent Med Rev 2015;2015:Article ID: 110215. doi: 10.15713/ins.ijcdmr.69.

10. Gancedo-Caravia L, Garcia-Barbero E. Influence of humidity and setting time on the push-out strength of mineral trioxide aggregate obturations. J Endod 2006;32(9):894-6.

11. Camilleri J. Hydration mechanisms of mineral trioxide aggregate. Int Endod J 2007;40(6):462-70.

12. Lee YL, Lee BS, Lin FH, Yun Lin A, Lan WH, Lin CP. Effects of physiological environments on the hydration behavior of mineral trioxide aggregate. Biomaterials 2004;25(5):787-93.

13. Khalilak Z, Vali T, Danesh F, Vatanpour M. The effect of one-step or two-step mta plug and tooth apical width on coronal leakage in open apex teeth. Iran Endod J 2012;7(1):10-4.

14. Torabinejad M, Chivian N. Clinical applications of mineral trioxide aggregate. J Endod 1999;25(3):197-205.

15. Tsujimoto M, Tsujimoto Y, Ookubo A, Shiraishi T, Watanabe I, Yamada S, et al. Timing for composite resin placement on mineral trioxide aggregate. J Endod 2013;39(9):1167-70.

16. Budig CG, Eleazer PD. In vitro comparison of the setting of dry ProRoot MTA by moisture absorbed through the root. J Endod 2008;34(6):712-4.

17. DeAngelis L, Chockalingam R, Hamidi-Ravari A, Hay S, Lum V, Sathorn C, et al. In vitro assessment of mineral trioxide aggregate setting in the presence of interstitial fluid alone. J Endod 2013;39(3):402-5.

18. Chedella SC, Berzins DW. A differential scanning calorimetry study of the setting reaction of MTA. Int Endod J 2010;43(6):509-18.

19. Hasheminia SM, Feizi G, Razavi SM, Feizianfard M, Gutknecht N, Mir M. A comparative study of three treatment methods of direct pulp capping in canine teeth of cats: A histologic evaluation. Lasers Med Sci 2010;25(1):9-15.

20. Shokouhinejad N, Jafargholizadeh L, Khoshkhournejad M, Nekooofar MH, Raoof M. Surface microhardness of three thicknesses of mineral trioxide aggregate in different setting conditions. Restor Dent Endod 2014;39(4):253-7.

21. Goldberg F, Massone EJ, Soares I, Bittencourt AZ. Accessory orifices: Anatomical relationship between the pulp chamber floor and the furcation. J Endod 1987;13(4):176-81.

22. Lee SJ, Monsef M, Torabinejad M. Sealing ability of a mineral trioxide aggregate for repair of lateral root perforations. J Endod 1993;19(11):541-4.

23. Bolhari B, Nekooofar MH, Sharifian M, Ghabrai S, Meraji N, Dummer PM. Acid and microhardness of mineral trioxide aggregate and mineral trioxide aggregate-like materials. J Endod 2014;40(3):432-5.

24. Torabinejad M, Hong CU, McDonald F, Pitt Ford TR. Physical and chemical properties of a new root-end filling material. J Endod 1995;21(7):349-53.