IEJ Iranian Endodontic Journal

Setting Time and Surface Microhardness of Mineral Trioxide Aggregate and 1% and 5% Fluoride-Doped Mineral Trioxide Aggregate Mixed with Water and Gel-like Polymer

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ARTICLE INFO

Article Type: **Original Article**

Received: 15 Mar 2019
Revised: 08 Jul 2019
Accepted: 28 Jul 2019
Doi: *10.22037/iej.v14i4.24094*

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ABSTRACT

Introduction: Mineral trioxide aggregate (MTA) is extensively used in endodontics. However, it has limitations such as long setting time, low compressive strength and poor handling properties. Our study aimed to compare the setting time and surface microhardness of MTA and fluoride-doped MTA (FMTA) using gel-like polymer (GLP) or distilled water (DW) as liquid. **Methods and Materials:** An MTA-like cement was prepared by mixing Portland cement, bismuth oxide and gypsum (75%, 20% and 5%, respectively). FMTA (1% and 5%) was made by substituting 1% and 5% of MTA powder with fluoride. GLP, composed of methyl cellulose (MC) and propylene glycol (PG), was used as the hydrating liquid and compared with distilled water. Six experimental groups (n=10) were examined for each test. The samples were subjected to Vickers surface microhardness test after 4 and 28 days. Setting time was measured using ANSI/ADA standards. Data was analyzed using two-way and repeated measured ANOVA and the Tukey HSD tests. **Results**: The MTA-like cement hydrated with GLP showed a significantly reduced setting time (P<0.05); 1% FMTA, mixed with GLP, had the shortest initial and final setting times. The microhardness values of all samples increased at different rates during 28 days (P<0.00001). The microhardness significantly decreased by addition of fluoride to MTA (P<0.001). **Conclusions**: The results of the present *in vitro* study suggested that using GLP as the hydrating liquid enhances the setting time with no adverse effect on the surface microhardness. However, adding fluoride can negatively affect the microhardness of MTA.

Keywords: Fluoride-oped Mineral Trioxide Aggregate; Microhardness; Mineral Trioxide Aggregate; Setting Time

Introduction

An ideal orthograde or retrograde root end filling material should have adequate marginal adaptation, sealing ability, biocompatibility, proper consistency and antimicrobial activities [1]. It should also be insoluble in tissue fluids, radiopaque and dimensionally stable [1-3]. The research to find a proper endodontic root end filling material led to the introduction of mineral trioxide aggregate (MTA). This material was initially used for root end fillings and subsequently for pulp capping, pulpotomy, apexogenesis, apexification, root perforation repair and root canal filling [2]. In many endodontic procedures -such as perforation repair and apical surgeries -MTA can replace other materials *i.e.* amalgam, zinc oxide eugenol, Dycal and composite resin [4, 5]. Despite all efforts, its limitations such as long setting time, low compressive strength and poor handling have remained [2, 6].

Application of a gel-like polymer (GLP), composed of 3% methyl cellulose (MC) and 1.5% polyethylene glycol (PG) as the

mixing liquid, has been suggested to improve the handling properties of MTA [7]. Based on previous studies, the addition of PG to MTA improves its a) handling properties, b) push-out bond strength to dentin and c) calcium ion release [8, 9]. PG has no negative effect on the biocompatibility of MTA [10], its sealing properties [11] and decreases its film thickness [12]. MC has an -OH base, is water soluble and is used for viscosity control. Its anti-washout admixture binds water molecules within the cement, subsequently increases its cohesive strength and improves its handling and mechanical properties [13]. Moreover, MC enhances the compressive strength of MTA [14].

Furthermore, previous studies incorporated fluoride into the formulation of MTA powder to enhance its biological properties. Fluoride added to MTA powder suppresses osteoclastic activity, increases osteoblast proliferation/differentiation and stimulates new bone formation [15, 16]. Comparison between MTA and fluoride-doped MTA (FMTA) cements showed that FMTA cements demonstrated superior sealing ability during a period of up to 6 months and significant expansion; thus, FMTA may be suitable for use when fluid isolation cannot be achieved [17, 18]. Moreover, fluoride-doped calcium silicate cements showed higher bioactivity and accelerated apatite formation [19]. Furthermore, the human osteoblast-like cells on fluorapatite-collagen composites illustrated significantly higher proliferation and differentiation [20]. Fluoride-doped calcium silicate cements have shown greater calcium ion release [19].

On the other hand, the biological properties of MTA are attributed to its calcium ion release capacity [3]. Thus, it is expected that adding fluoride to MTA powder could promote its biological properties and result in optimal biocompatibility.

The aim of this study was to evaluate the effects of the addition of two different percentages of fluoride to MTA powder and the replacement of its liquid with gel-like polymer on its setting time and surface microhardness.

Methods and Materials

Powder preparation

The three materials evaluated in this study were (*i*) MTA like cement (MTA) (*ii*) 1% and (*iii*) 5% F-doped MTA-like cement (FMTA 1%, 5%). The MTA like cement was prepared by mixing the Portland cement (75%) with bismuth oxide (20%) and gypsum (5%). FMTA was made by substituting 1% and 5% of the powder with fluoride. The components were homogenized in a bench-top planetary ball mill (Retsch PM100; Retsch GmbH, Haan, Germany) for 10 min [21, 22].

Sample preparation

Mixing of the materials was standardized by mixing 1 g of the prepared powder with 0.33 g of either distilled water (DW) or GLP. For this purpose, the MTA-like cement, FMTA 1% and FMTA 5% were individually mixed with GLP and DW, with a powder-to-liquid ratio of 3/1 on a glass slab at room temperature.

Setting time evaluation

Sixty samples with 10 mm in diameter and 2 mm in height_ were fabricated for the evaluation of the setting time (6 groups; n=10 in each group). The samples were fabricated using stainless steel cylindrical moulds. The initial and final setting times of all samples were measured in accordance with the American Society for Testing and Materials (ASTM) International Standard C266-08 (2008) and the American National Standards Institute/American Dental Association (ANSI/ADA) Specification No. 57 (2008) [23]. The test was performed in an incubator at 37°C (all samples were kept incubated at all times except for the measurement times) using a Gillmore apparatus (CT-5; ELE International Inc., Loveland, CO, USA). The apparatus included two needles: the needle for testing of the initial setting time weighed 113.4 g and had an active tip with 2.12 mm diameter (initial needle). The needle for the final setting time weighed 453.6 g and had an active tip with 1.06 mm diameter (second needle). The incubation times in-between measurements were determined in a pilot study. The samples were incubated for 5 min between measurements. The initial needle was lightly pressed on the surface of each sample until it no longer created a complete circular depression on the specimen surface. For each sample, the time lapse between completion of mixing and unsuccessful indentation was recorded in min and defined as "initial setting time" for the initial needle. The "final setting time" was determined using the second needle [24].

Surface microhardness evaluation

For evaluation of surface microhardness, the mixture was placed in polycarbonate cylindrical moulds with an internal diameter of 4 mm and a height of 6 mm. Considering 3 types of powders and 2 types of liquids, our samples were divided into six experimental groups (a total of 60 samples, n=10 in each group). The filled moulds were incubated at 37°C and 100% humidity for 4 days. After 4 days, all samples were removed from the incubator and their surfaces were wet-polished using 600, 1000 and 1200-grit fine-grain sandpapers (Buehler-Met®; Agar Scientific Limited, Cambridge, UK), at room temperature with minimum hand pressure to obtain smooth, flat surfaces. The Vickers microhardness test for each specimen was performed in accordance with the BS EN 843-4 (2005) [25] using a Micromet 5114 tester (Buehler Ltd, Lake Bluff, IL,

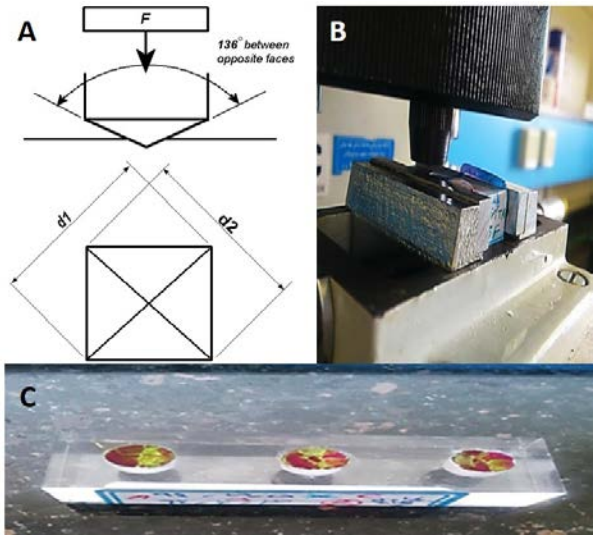

Figure 1. *A*) Schematic view of Vicker's microharness indenter. Two diagonals of the indentation left on the surface is measured and the microhardness is calculated; *B*) Microhardness testing machine. An indentation is made on the surface of specimen; *C*) Specimens in polycarbonate cylindrical mold shown after testing

USA) with a square-based pyramidal-shaped diamond indenter and maximum load of 500 g for 30 sec at room temperature. This produced a quadrangular depression with two equal orthogonal diagonals on the polished surface of the cement. Ten indentations were randomly made on the polished surface of each specimen. The two diagonals, produced from the indentation, were measured immediately under a microscope, and the Vickers microhardness value displayed on the digital readout of the tester was recorded in megapascals (MPa) (Figure 1). After testing, the samples were immediately placed in an incubator. After 28 days, all samples were removed from the incubator and subjected to surface microhardness test using the same methodology. The mean value of 10 indentations was considered as the Vickers surface microhardness number [26, 27].

Statistical analysis

Statistical analysis was performed using SPSS version 18 (SPSS Inc., IL, USA). The two-way ANOVA and repeated measures ANOVA were used to compare the setting time and microhardness of the groups, respectively. The Tukey's HSD test was performed for pairwise comparisons.

Results

Figures 2 and 3 present the mean and standard deviations of the initial and final setting times in all experimental groups. Both hydrating liquid and fluoride concentrations had a statistically significant effect on the initial and final setting times. In both DW and GLP groups, different fluoride concentrations had significant effects on the initial and final setting times (P<0.001).

In DW group, the initial and final setting time values of MTA and FMTA 5% groups did not significantly differ but there was a statistically significant difference between MTA and FMTA 1% groups (P<0.001), and also between FMTA 1% and FMTA 5% groups (P<0.001); with FMTA 1% group having the shortest initial and final setting times. In GLP group, a significant difference (P<0.001) in the initial and final setting times was found between all percentages of fluoride, with FMTA 1% having the shortest initial and final setting times. In other words, FMTA 5% exhibited longer setting time than FMTA 1% (Figures 2 and 3). In addition, a statistically significant difference in the initial and final setting times was found between DW group compared with GLP group, with GLP group having shorter setting time (P<0.001). Of all tested cements, FMTA 1%, mixed with GLP, had the shortest initial and final setting times.

At 28 days, the surface microhardness values for all experimental groups were significantly greater than those at 4 days after mixing (P~0).

Addition of different percentages of fluoride caused a statistically significant difference in surface microhardness at both 4 and 28 days (P=0.001). Addition of 5% fluoride produced the lowest surface microhardness value compared to other groups (P<0.001). As the amount of fluoride increased, the surface microhardness decreased.

According to Table 1, there was no significant difference in microhardness between DW and GLP groups with corresponding concentrations of fluoride at 4 or 28 days.

Table 1. Mean (SD) values of Vickers surface microhardness for different groups at 4 and 28 days

	4 days			28 days		
Fluoride content	**DW group**	**GLP group**	***P*-value**	**DW group**	**GLP group**	***P*-value**
0%	46.8900 (3.3781)	47.3500 (2.7163)	0.297	81.6700 (3.7007)	79.2800 (3.2788)	0.120
1%	26.7700 (1.9049)	28.8900 (1.7847)	0.297	58.3100 (2.1439)	59.9100 (2.4821)	0.106
5%	23.2300 (2.1458)	22.5900 (1.9145)	0.297	54.4100 (2.1031)	52.7000 (2.3860)	0.140
***P*-value**	<0.001	<0.001		<0.001	<0.001	

GLP: Gel-like polymer; DW: Distilled water

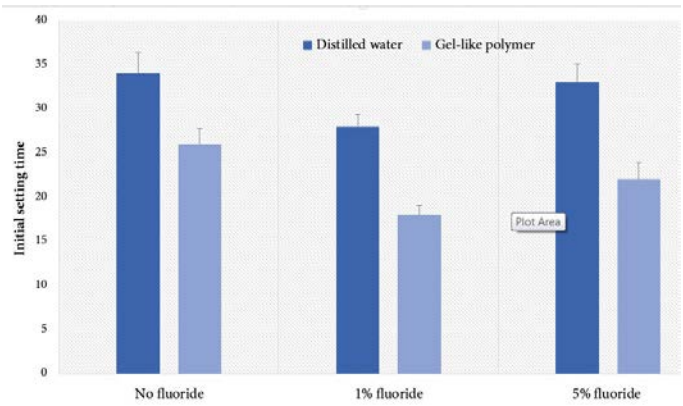

Figure 2. Initial setting time values in minutes for all 6 experimental groups

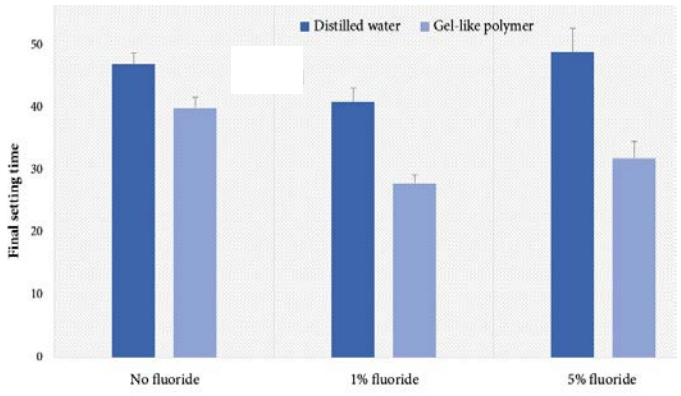

Figure 3. Final setting time values in minutes for all 6 experimental groups

Discussion

This *in vitro* study aimed to assess the effects of adding fluoride to MTA and mixing it with GLP (PG+MC) instead of water on its setting time and microhardness. The results showed that using GLP hydrating liquid could decrease the setting time without significantly impacting microhardness values. However, adding fluoride in either concentrations could decrease the microhardness values in both time periods.

An extended setting time poses a disadvantage for root-end filling materials, because it facilitates leakage and cement dislodgement during apical surgery [28]. Addition of different agents to dental materials often results in changes in their physical properties, and may subsequently affect their clinical efficacy [29]. Addition of light-cure resin monomers to MTA has been proposed to enhance its properties and reduce its setting time [30]. However, resinous MTA-based materials inhibit calcium ion release by the MTA and compromise its ability to stimulate tissue repair [31, 32]. Thus, Jacinto *et al.* [33] used 2% chlorhexidine. Although chlorhexidine increased calcium ion release and antimicrobial activity of MTA, it prevented its polymerization. Also, Zapf *et al.* [34] used phosphate buffered saline, calcium chloride 5% ($CaCl_2$) and sodium hypochlorite 3% (NaOCl) and showed that only $CaCl_2$ accelerated the setting reactions. Another substance proposed to improve the handling properties of MTA was polyvinyl alcohol (3 and 5 wt%). Polyvinyl alcohol, a modified liquid for MTA, increased its setting time and decreased its compressive strength [35]. However, nanoparticulate calcium carbonate (NPCC) accelerated the setting time but again decreased the compressive strength [36].

According to Natu *et al.* [37] the ratio of water to PG affects the properties of MTA mixed with PG. Adding large amounts of PG (more than 50%) increases the setting time to a degree that is clinically unacceptable, which is attributed to the formation of larger porosities. Our GLP also contained PG and reduced the setting time. This effect was probably due to the fact that our GLP contained about 2% to 3% of PG, and MC was its main constituent. This result is in accordance with that of Natu *et al.* [37].Thus, it seems necessary to find the optimal percentage of PG to reduce the setting time without negatively affecting other properties of MTA. To the best of our knowledge, there is no evidence in the literature regarding the effect of fluoride on the setting time of MTA, Portland or MTA-like cements. In this study, FMTA 1%mixed with GLP had the shortest initial and final setting time amongst all the tested cements. Future studies with different amounts of fluoride are suggested.

Surface microhardness indicates the degree to which, a material is hydrated during the setting reactions [22]. There are a few reports about MTA surface microhardness following incorporation of different materials into its formulation. Assessment of the effect of bismuth oxide on MTA demonstrated that lack of bismuth oxide was associated with an increase in surface microhardness [23] . Salem Milani *et al.* [9] stated that placing MTA mixed with PG in the root canal was easier than placing MTA mixed with water, and PG had no negative effect on the biocompatibility of MTA. This effect occurs in certain concentrations of PG (20% PG+80% water); at higher levels, it decreases the calcium ion release. It also decreases the surface microhardness as it negatively affects the setting process. Milani [9] also stated that their suggested amount would ideally improve MTA handling with no significant reduction in its setting quality.

In the present study, the surface microhardness of MTA for all groups was evaluated after 4 and 28 days with a significant difference in values between the two time periods. GLP did not decrease surface microhardness of the MTA in none of the groups. Although some studies have assessed the effect of fluoride on MTA, there is no study on its effect on MTA microhardness. In this study, the results did demonstrate that addition of fluoride to MTA had a detrimental effect on its microhardness and adding 5% fluoride produced the lowest surface microhardness compared to other groups. Adding fluoride to MTA may negatively affect its microhardness. However, GLP did not probably affect the microhardness of MTA.

Conclusion

Fluoride and GLP modified the properties of MTA-like cement. Addition of GLP to MTA was proposed to improve its handling properties; the setting time decreased significantly, and GLP showed no detrimental effect on the microhardness of MTA. However, the addition of fluoride to MTA, although decreased its setting time, had an undesirable effect on its microhardness.

Acknowledgment

The authors wish to thank Research Council of Shahid Beheshti Dental School for financial support.

Conflict of Interest: 'None declared'.

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Please cite this paper as: Dianat O, Mahdian A, Sanagoo N, Mozayeni MA, Eskandarion S. Setting Time and Surface Microhardness of Mineral Trioxide Aggregate and 1% and 5% Fluoride-doped Mineral Trioxide Aggregate Mixed with Water and Gel-like Polymer. Iran Endod J. 2019;14(4): 265-70. *Doi: 10.22037/iej.v14i4.24094.*