

Original Article

Investigation of the effect of hydrochloric acid with different concentrations on mineral trioxide aggregate plug and dentin

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ABSTRACT

Background: Mineral trioxide aggregate (MTA) becomes a hard mass after setting and making it difficult to remove and can cause significant problems in the retreatment process. The aim of this study was to evaluate the effect of different concentrations of hydrochloric acid (HCl) on MTA dissolution and its effect on dentin.

Materials and Methods: In this *in vitro* study, 45 single-root premolars were selected. Artificially open apex was created in all samples with similar process. The samples were randomly divided into 4 experimental groups of 10, and a control group of 5. Four-millimeter thick apical plugs of Root MTA were placed in all samples in an orthograde manner. HCl was used at concentrations of 3.75%, 7.5%, 15%, and 22.5% (w/v) for the experimental groups and normal saline for the control group. Each sample was exposed to the desired solution for 15 min. Then, MTA retrieval and reaching the working length were attempted with k-file # 30. The times of each sample were recorded. Furthermore, after longitudinal incision of the roots with a disc, the dentin surfaces of canals were examined with a Dino-Lite microscope (×50). Results were analyzed by Shapiro–Wilk test and one-way analysis of variance tests. The level of significance *P* value was set at 0.05.

Results: The lowest average time of reaching working length was observed with group 22.5% that was significantly lower than 15% and 7.5% concentrations (*P* = 0.005 and *P* = 0.011). Furthermore, by examining with ×50 of Dino-Lite microscope, no difference was observed on the canal walls.

Conclusion: The optimum concentration of HCl was 7.5%. Furthermore, different concentrations of HCl had no significantly different effect on the dentinal canal wall using Dino-Lite microscope with ×50.

Key Words: Dentin, hydrochloric acid, solvents

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INTRODUCTION

Mineral trioxide aggregate (MTA) is a bioactive and biocompatible material that has a unique sealing ability.^[1] However, sometimes due to failures of endodontic treatment, previously applied MTA should be refresh or even exchange with new MTA

to reestablish the sealing characteristic. Hence, retrievability of MTA is an important interest.^[2] When MTA sets, it becomes a hard mass and retreatment could be tough and procedural problems may occur.^[3]

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Some investigations have discussed about retrieval of MTA by different solvents on MTA.^[4,5] Previous studies have evaluated the effect of different solvents: carbonic acid, hydrochloric acid (HCl), chlorhexidine, and sodium hypochlorite (NaOCl) on the surface hardness of MTA.^[5,6] Furthermore, different solutions have different results on the MTA surface. According to many studies,^[7-9] MTA has very low or even no solubility. However, some studies showed that low pH could affect MTA hardness,^[10] tensile strength,^[11] and push-out bond strength^[12] because of occurring porosities and voids.^[13] Acidic environment causes corrosion because of the decomposition of calcium hydroxide and calcium sulfoaluminate phases.^[14]

Hence, it is inevitable to have a solvent for MTA retrieval. However, we should keep in mind that acidic material for MTA solubility might pose the danger of weakening the tooth structure. Literature search showed that no study has been done to determine the minimum concentration of acidic material that can solve MTA and on the other hand has no effect on tooth structures. The present study was a pilot study to select the optimal concentration of HCl which can be used for the removal of MTA and had no effect on dentin.

MATERIALS AND METHODS

This clinical trial study was approved by the Vice-Chancellor of the Research and Ethical Committee in Mashhad University of Medical Sciences (MUMS) with ethic code of IR.MUMS.DENTISTRY.REC.1399.100. MTA (Root MTA, Iran) was used to form apical plugs in teeth prepared to simulate immature teeth with open apices.

Forty-five human single-rooted teeth with straight canal extracted for the therapeutic purpose were included in this study. Teeth with root fracture, cracks, resorption, calcification, and anatomical irregularities were excluded from the study. The teeth were stored in 0.5% NaOCl (Marvabon, Tehran, Iran) for 48 h to disinfection and then stored in saline solution (0.9% NaCl, Darupakhsh, Tehran, Iran). The teeth were decoronated by a diamond disc (Tizkavan, Tehran, Iran) and 12 mm of roots remained. Then, 2 mm of the apex of each tooth was cut, so the simulation of an open apex was done. The mechanical preparation was done by ProTaper Gold (Dentsply, Switzerland) rotary files (SX to F3). The apical part was prepared by #1, 2, and 3 Peeso

Reamers (Mani, City, Japan) in a retrograde manner, which resulted in a diameter of 1.1 mm.^[15] Therefore, 45 roots with 10 mm length and similar shaping were prepared. Two milliliters of 5.25% NaOCl was used for root canal irrigation between each instrument. To simulate the periapical soft tissues during plug placement, the wet cotton was placed at the apical part of roots and then the roots were mounted in acrylic resin (Acropars, Tehran, Iran). Canals were dried with paper point (DiaDent, South Korea). MTA (L Root, Tabriz, Iran) was prepared according to the manufacturer's instructions and carried into the canals in an orthograde manner by MTA carrier (MAP System, Switzerland),^[16] guided by the tip of a #80 paper point and compressed by an appropriate hand plugger (Dentsply, Switzerland) to a thickness of 4 mm. For confirmation of the appropriate MTA plug placement, the radiographs were taken. The rest of the canals were left unfilled. A wet paper point was placed on the MTA plug. All the canals were sealed with temporary restoration (Coltosol, Ariadent, Tehran, Iran) and then stored at 37°C and 100% humidity for 21 days.^[4] Then, the specimens were randomly divided into four experimental groups ($n = 10$) and exposed to the different concentration of HCl (3.75%, 7.5%, 15%, and 22.5% [w/v]) (Merck, Darmstadt, Germany). These concentrations were conducted in the Department of Chemistry of Mashhad Ferdowsi University. The control group ($n = 5$) was exposed to normal saline. The chronometer was activated and MTA plugs were continuously exposed to the HCl (3 drops/5 min) for 15 min. Samples were then rinsed by distilled water for 1 min. K-file # 30 was used with watch-winding movements and gentle apical pressure and attempt was made to cross the MTA plug and reach the working length. Finally, the times of each sample were recorded.

To evaluate the internal morphology of canals that were exposed to HCl, the specimens were vertically grooved on both sides with a disk and then split longitudinally with a chisel. The amount of erosion in both sides of each sample was assessed under the Dino-Lite Microscope (Hsinchu, Taiwan) with $\times 50$.

Data were analyzed using the one-way analysis of variance. The level of significance was set at 0.05.

RESULTS

In this *in vitro* study, 40 single-root tooth samples in the form of 4 concentrations (3.75%, 7.5%, 15%, and

22.5%) and 5 control samples were examined for the time required to penetrate the MTA plug. The 3.75% group and the control group were excluded from the statistical analysis because they could not penetrate and solve in the MTA. The normality of data distribution in the other three groups was evaluated using Shapiro–Wilk test, and it was found that the data had a normal distribution. A concentration of 3.75% was excluded from statistical comparison with other groups due to the lack of dissolution of MTA in 15 min and the fact that no time was recorded for the samples.

In this study, the mean time required to penetrate the MTA plug was significantly different between the groups ($P = 0.001$). Comparing the concentrations, it was found that the mean time required to penetrate the MTA plug in the groups of 7.5% and 15% was not significantly different from each other ($P = 0.324$), but the mean time at the concentration of 22.5% compared to the concentration 7.5% and 15% was significantly lower ($P = 0.005$ and $P = 0.011$, respectively).

The statistical indicators of mean, standard deviation, and minimum and maximum amount of time required to penetrate the MTA plug at each concentration and the result of the statistical test are given in Table 1.

After longitudinal incision of the teeth with a disc, the two halves of each tooth were examined under a Dino-Lite microscope. Due to the lack of evaluation and damage in the dentin walls of the samples, no statistical analysis was performed for the groups [Figures 1-5].

DISCUSSION

The results of the present study showed that HCl 3.75% was the only acidic concentration that could not dissolve MTA in 15 min. In terms of the time required for penetrating in MTA, the lowest average recorded time belonged to the 22.5% group and the highest to the 7.5% group. Furthermore, in terms

Table 1: Comparison of mean, standard deviation, and analysis of time required to remove mineral trioxide aggregate plugs between different concentrations (s)

Concentration (%)	Mean±SD (s)	Min (s)	Max (s)	Test
7.5	436.60±197.33 ^a	110	770	$F=8.84$
15	326.70±124.76 ^a	92	483	$P=0.001$
22	171.00±76.98 ^b	43	303	

^aSimilar letter shows there were no significant differences between the parameters. ^bSD: Standard deviation

of the effect of different concentrations of acid on the dentin structure, no significant difference was observed between the groups, using Dino-Lite microscope.

Removing MTA from the root canal has always been of interest to endodontists, especially in cases that MTA needs to be refreshed or retreated. HCl has been introduced in the industry to remove cement.^[17] It has been observed that after the use of acids such as dilute HCl, the removal of cement is easily done by dissolving or decomposing the cement. Furthermore in previous studies, concluded that HCl (with pH value of 1.8), was more effective than other tested materials such as acetic acid, 5.25% NaOCl, and phosphate buffer, to loosen Portland cement structure.^[5] Hence, in the present study, HCl had been selected in different concentrations to analyze their ability on the removal of MTA.

Previous studies used microhardness test for analyzing the chemical solution ability on the MTA removal, presumed that by decreasing the MTA microhardness, it could be removed more easily than hard cement.^[17] Abraham *et al.* showed that 2% acetic acid has maximum efficacy in reducing the surface microhardness of partial and completely set MTA, followed by 2% carbonic acid.^[2] Shojae *et al.*^[5] reported that HCl and H₂CO₃ were effective in significantly reducing the surface hardness of MTA on both days 1 and 21.

Since we did not find the clinical technique in the literature for MTA retrieval from the canal, the present pilot study was designed to find the lowest concentration of HCl that can remove MTA from the



Figure 1: Dentin appearance after exposure to 22.5% solution.

canal in a clinically acceptable time. In this study, to be more similar to the clinical situation, MTA was used inside the root canal to find the optimal concentration of HCl that had no effect on dentin. Single root teeth were used to facilitate the alignment of samples. The researchers showed that a thickness of 4 mm MTA is more resistant than a thickness of 1 mm.^[18] Hence, in this study, a thickness of 4 mm MTA was used. It has been reported that the maximum compressive strength and complete chemical setting of MTA take place by 21 days.^[4] Hence, in this study for achieving completely set MTA, acid exposure of samples was done after 21 days. Because there is no study on how long an acidic substance can be used to remove MTA in the root canal, a pilot study of 5 samples was done and found that 15 min could be a good time and clinically seems reasonable. The reason for washing

the HCl every 5 min by normal saline was to remove the dissolved debris or MTA and also to add fresh solution maximally 3 times for better effectiveness.

Because this acidic compound may have a dissolving and abrasive effect on the dentinal walls of the canals, the canal walls were evaluated by a Dino-Lite microscope. However, in future studies, it is better to use more accurate microscopes with higher magnification to study more detailed effects of HCl on the canal walls. Previous studies have only focused on the dissolution of MTA regardless of the acid's effect on dentin. Therefore, we could not compare this part of the study with other studies.

Some parameters such as, solution concentration, the exposure time, type of MTA, and its thickness can effect on the solution penetration into the MTA.



Figure 2: Dentin appearance after exposure to 15% solution.



Figure 4: Dentin appearance after exposure to 3.75% solution.



Figure 3: Dentin appearance after exposure to 7.5% solution.



Figure 5: Dentin appearance after exposure to normal saline solution.

In the following, these parameters in this study were discussed with similar articles. Shojaee^[5] and Nandini *et al.*^[4] used stainless steel cylinders and molds in their studies. However, in this study, MTA was placed inside the root canal of extracted teeth. Many solutions with high concentrations are able to dissolving the MTA.^[2] But it should be considered that the selected acid concentration had no negative effect on the root Canal walls and periodontium. In this study, the lowest concentration of acid that removes MTA at a given time was called the optimal concentration. Based on the results of Saghiri *et al* study,^[17] it was found that the optimal concentration of HCl was 37% and this solution could reduce surface hardness more than acetic acid which can be due to the lower pH value of HCl (pH = 1.8) in comparison with acetic acid (pH = 3.5). However, in this study, it was shown that the optimal concentration of HCl for MTA dissolution was 7.5%. The difference between the two studies is probably due to the different methodologies. The diameter and height of the MTA in the Saghiri *et al* study^[17] were 8 and 10 mm, respectively, and in this study, the height of the MTA was 4 mm and the diameter of the MTA was equivalent to the diameter of Peeso Reamers #3. The exposure time in the Saghiri study was 1 min, whereas in this experiment, up to 15 min was considered for each sample. The type of MTA may also affect the results. In this study, Root MTA (Tabriz, Iran) was used, and in the Saghiri study,^[17] Portland cement was used. Angelus MTA was used in Abraham *et al.*'s study^[2] and ProRoot MTA was used in Aggarwal *et al.*'s study.^[19]

As mentioned, in terms of the time, there was no significant difference in low concentrations of HCl. This may indicate that as the safest concentration of HCl is to be used, it is best to use a concentration of 7.5%. In the present study, unlike other previous studies, the effect of acid concentrations on dentin was investigated, but no visible change was found. In some samples, a thin layer of discoloration was observed in the canal walls, which was removed by washing with normal saline, and in general, no significant change was observed in the $\times 50$ by Dino-Lite microscope.

It is suggested that in future studies, other parameters such as the effect of HCl on the structures of the periodontium will be examined. Furthermore, other acidic materials with different concentrations compare with each other. More accurate methods such as electronic microscopy can be used to examine surface

changes in dentin after the application of acidic solution for MTA removal.

CONCLUSION

According to the result of the present study, 7.5% HCl is the optimal concentration for MTA retrieval. This study can be used as a pilot study for further investigations, and if the biocompatibility of 7.5% HCl will be determined, the clinical usage of this material for MTA retrieval may be possible.

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Conflicts of interest

The authors of this manuscript declare that they have no conflicts of interest, real or perceived, financial or nonfinancial in this article.

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