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Effects of different accelerators on the setting time and physo-chemical properties of mineral trioxide aggregate

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ABSTRACT

This study aimed to evaluate the effects of different accelarators added to Mineral Trioxide Aggregate (MTA) liquid on the final setting time, push-out bond strength as well as the physicochemical properties of MTA using SEM analysis.

Keywords: *accelerators, automatic Vicat, MTA, setting time, sodium carbonate, push-out.*

1. INTRODUCTION

Although Mineral Trioxide Aggregate was initially introduced as a root-end filling material, it is used in a variety of clinical applications (such as pulp capping, apexification, apexification, pulpotomy perforation, and others) due to its superior biocompatibility, effective sealing capability, and ability to improve regeneration of the pulp and peripheral root tissues [1].

MTA has extensive clinical use. It is a powder containing hydrophilic particles, which set in the presence of moisture. In the setting process, the paste obtained from the contact between the MTA powder and liquid loses its plasticity and gains density [2, 3]. Highly soluble calcium compounds are the most frequently used accelerants to achieve Ca^{2+} ion support to shorten the setting period of MTA [4, 5]. Calcium chloride ($CaCl_2$) and calcium lactate gluconate (CLG) highly penetrate into cement pores, which are hygroscopic, and they also act as chemically engager to provide increased consistency for MTA [4]. The buffer solution disodium hydrogen orthophosphate (Na_2HPO_4) has been found to shorten MTA's setting period by different rates,too [6, 7].

Disodium hydrogen phosphate (Na_2HPO_4) is a salt of phosphoric acid. The Na_2HPO_4 compound is a highly hygroscopic and water-soluble powder [6]. Furthermore, Na_2HPO_4 is frequently used to catalyze the setting time of Portland cement and MTA [7, 8]. The effects of the varying ratios of Na_2HPO_4 on the setting time of white MTA were studied. It was reported that 15% Na_2HPO_4 is characterized with the shortest setting time in MTA cements mixed with 5%, 10% and 15% Na_2HPO_4 solutions [5]. In another study, setting times of the cements obtained by mixing white MTA with 5%, 10%, and 15% Na_2HPO_4 solutions were measured. It was reported that the cement mixed with 15% Na_2HPO_4 had the shortest setting time [9]. In their study assessing the biocompatibility of MTA cements enriched with varying ratios of Na_2HPO_4, Ding et al.[6] found that 15% Na_2HPO_4 represented the highest rate of cell viability. Sodium carbonate (Na_2CO_3) is the sodium salt of carbonic acid. Na_2CO_3 is a potent alkali salt that is highly soluble in water and can be used as a pH stabilizer[10]. Na_2CO_3 is reported to be an additive to Portland cement as hydration catalyst in the construction industry, as a foaming agent for toothpastes in dentistry, as an abrasive particle in air abrasion systems in periodontology, and, furthermore, as a catalyst for bone fillers with silica content [9, 11, 12]. Na_2CO_3 is one of the most effective accelerator of the hydration and setting of Portland cement in the construction industry [3]. Studies report that Na_2CO_3 added to the bone filler with calcium silica content (CS) shortened the setting time of cement, and it further enhanced the bioactivity of the filler and formation of the bone-like apatite layer [11]. At present, no studies have considered the addition of Na_2CO_3 to MTA as an accelerator and its impact on the setting time.

The MTA cement's hardening time is usually measured using Vicat or Gillmore needles. The use of these device is prone to some setbacks such as user faults and data loss. Automatic Vicat device has been suggested as an alternative in order to minimize these issues. The automatic Vicat device provides a standard needle downtime and force, allowing for a repeated test performed in a clear manner. On-screen display and transfer of data to the computer provides great user. In literature, the number of study using automatic vicat device was very rare. In this study, the final setting time was mesured with automatic vicat device to standardize the test mechanism.

This study investigates the effects of 10% Na_2CO_3 and other accelerators (10% $CaCl_2$, 15% Na_2HPO_4, and 23.1% CLG) added to the distilled water used in MTA cement preparation. These accelerants are intended to decrease the long setting time, which is one of the negative attributes of MTA. The final setting times, push-out bond strength, and SEM analysis parameters of MTA cements prepared using different accelerators were evaluated using *in vitro* tests.

2. EXPERIMENTAL SECTION

During our study, the chemicals $CaCl_2$, Na_2HPO_4, Na_2CO_3, and CLG were added in distilled water as 10%, 15%, 10%, and 23.1%, respectively.

The study was conducted with confirmty approval from the local ethics comittee.

2.1. Study Group Preparation.

Group 1: This group was composed of a mixture of white ProRoot™ MTA (Dentsply Maillefer, Switzerland) powder and distilled water at a powder to liquid ratio of 3/1.

Group 2-3-4: This group was composed of a white ProRoot MTA powder mixed with 10% $CaCl_2$, 15% Na_2HPO_4, 10% Na_2CO_3 solution. These weights were calculated according to the mole percentages of accelerators and put in a glass beaker and completed to volume by 100 ml using distilled water. The solution was homogenized using a magnetic stirrer and mixed in the magnetic stirrer (Brunco, US) until the end of the tests. Fresh solutions were prepared for each test.

Group 5: This group was prepared using a mixture of ProRoot MTA powder and 23.1% CLG solution by weight. The above "density" and "molarity" formulas were used in order to ensure preparation by weight of the 23.1% CLG solution. After the procedure, 27.0 g lactic acid ($C_3H_6O_3$) (MA=90.1 g/mol), 35.6 gr glucono delta lactone ($C_6H_{10}O_6$) (MA= 178 g/mol) and 15.28 g calcium oxide (CaO) (MA= 56.0 g/mol) were weighed, placed inside a glass beaker and completed to volume by 100 ml using distilled water.

Table 1. Accelerators used to mix with MTA.

	Contents
Group 1	MTA (1 g) + Distilled water (0,33 ml)
Group 2	MTA (1g) + % 10 $CaCl_2$ (0,33 ml)
Group 3	MTA (1 g) + % 15 Na_2HPO_4 (0,33 ml)
Group 4	MTA (1 g) + % 10 Na_2CO_3 (0,33 ml)
Group 5	MTA (1 g) + % 23,1 CLG (0,33 ml)

2.2. Measurement of Final Setting Times.

The sample for the measurement of the setting time was prepared on the basis of the ANSI/ADA #57 specification. Stainless steel molds were prepared in order to preserve identical volume and form during the setting time measurement. This stainless steel molds have an internal diameter of 10 mm and a thickness of 2 mm.

Setting time was measured using the automatic Vicat device (Vicatronic E044, Matest, Italy). Both the device and needle calibrated for needle penetration depth, penetration time and shape in accordance with the predefined parameters identified prior to the measurements. The needle penetration depth was defined as 2 mm in line with the mold height. According to the study by Kogan et al, the needle penetration range was defined as 0, 5, 10, 15, 20, 25, 30, 40, 50, 60, 90, 120,150, 180, 210, and 240 minutes. The automatic Vicat needle weighing 300 g and measuring 1.0 ± 0.2 mm in diameter was penetrated into the specimen in “driven mode” [10]. The test groups were placed inside stainless steel molds. The excess cement was removed from the surface of the sample holder by a single sweep with the edge of a glass slide. The final setting time was logged as the period elapsing until the cement formed a depth less than 1 mm on the top surface of the stainless steel container from the initial cement mixture. The test was repeated for 10 times for each test group.

2.3. Push-Out Bond Strength.

Extracted 100 single root human teeth with straight canals were selected for this study. The number of specimens in the groups composed of 20 teeth. The crowns of all teeth were removed, and the midroot dentin was sectioned horizontally into slices with a thickness of 2.0 mm by using a water-cooled low-speed IsoMet diamond saw (Buehler, Lake Bluff, NY). Each section was enlarged to achieve a canal gap using #5 Gates Glidden drill for standard cavities of 1.3 mm diameter. Afterwards, it was rinsed in 1% NaOCl and saline in ultrasound device to remove debris. The control group and accelerator added cements were placed inside the dentine discs obtained. A wet cotton pellet was placed over and bottom to each specimen and allowed to set the cement. The specimens were kept in the incubator (EN 120, Konya, Turkey) at 37°C for 72 hours. The push-out bond strength values were measured by using a universal testing machine (Instron Universal test machine; Elista, Istanbul, Turkey). The samples were placed on a metal slab with a central hole to allow the free motion of the plunger. The force was applied at a 1 mm/min. until the MTA cement was completely removed from the canal.

Bonding surface area of each disc was calculated as mm^2 for the conversion of the bond strength into MPa unit. MPa values convert to Newton with $F/ 2\pi r \times h$ formule.

F= Applied Force (Newton), max. force removing the canal fill from the canal

Surface Area $=2\pi r \times h$ (π= 3.14, r = root canal radius, h = height of dentin disc)

After the Push-Out test, the coronal and apical surfaces of all discs were examined under X40 magnification using a stereomicroscope (Olympus SZ4045 TRPT Olympus, Osaka, Japan) and each sample was categorized into I of III the failure modes: I. Type – Adhesive, test material and removal from the dentin interface; II. Type – Cohesive, test material or internal rupture; III. Type – Mixed, both, collective adhesive and cohesive rupture

2.4. SEM Analysis.

The samples were dried in hot air at 37°C for one day and their surface was coated with gold film in vacuum environment. Examinations were performed at x5000 magnification using back scattered electron micsroscope(LEO 440, Leike Zeiss, K61n, Germany).

2.5. Statistical Analysis.

Final setting times mean values were compared using the non-parametric Kruskall-Wallis H test while paired comparisons were performed using the Mann-Whitney U test. The push-out bond strength test mean values of the groups was performed using the Kruskall-Wallis H test and paired comparisons were made using the Mann-Whitney U test. The statistical difference is displayed on the basis of a 0.05 significance level.

3. RESULTS SECTION

3.1. Setting Time.

The setting times for MTA mixed with various additives are shown in Figure 1. MTA mixed with sterile water set in 71 min. There was a significant decrease in setting time between the control group and the addition of CLG and the Na_2HPO_4 additives. There was no significant difference between Na_2CO_3 and the $CaCl_2$ additive groups. The reduction in the final setting time of 23.1% CLG and 10% $CaCl_2$ groups is likely attributed to the fact that highly-soluble Ca^{+2} compounds quickly rendered the solution saturated. Studies on the test of setting time of added Na_2CO_3 are avaible in the literature for hydralic cement, but not especially MTA. Thus, the results of the final setting time could not be directly compared with any MTA studies. The results of this study revealed that the control group set in 71 min and the 10% Na_2CO_3 group set in 54 min. The compound 10% Na_2CO_3 used as an accelerator in our study shortened the setting time of MTA. The results of our study reveal that the 15% Na_2HPO_4 group decreased the final setting time to 43 min, from 71 min.

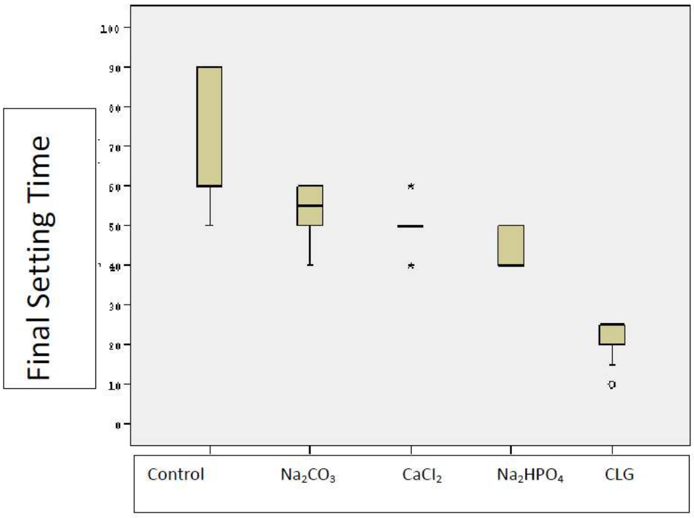

Figure 1. The means of final setting times values of test groups.

3.2. Push-out Test.

Results of the Kruskal–Wallis test revealed that the mean push-out bond strength values of white MTA (control), Na_2CO_3, $CaCl_2$, Na_2HPO_4 and CLG were 3.28 ± 0.92, 9.91 ± 2.91, 2.73 ± 0.91, 4.77 ± 1.39 and 1.36±0.43 MPa, respectively. The push-out bond strength values of Na_2CO_3 group was the highest, with statistically significant differences between the groups ($p < 0.05$) (Figure 2). The lowest push-out bond strength was observed in the CLG group ($p < 0.05$). The percentages of the failure modes of the samples are presented in Table 2. Inspection of the samples under a stereomicroscope at 40 magnification revealed that the bond failure was adhesive type in the majority of the all groups however, some of the control, Na_2CO_3 and $CaCl_2$ exhibited cohesive failure patterns (Table 2). In this study, we think that varying fracture modes may be due to the different sizes of accelerators added.

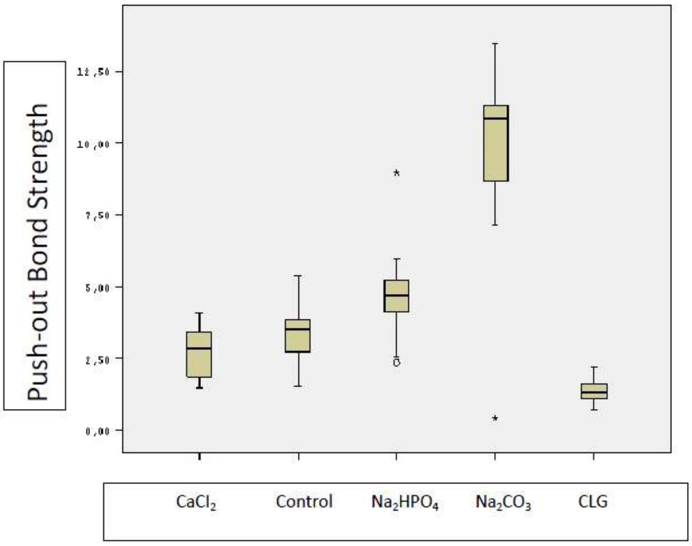

Figure 2. The means of push-out bond strength values of test groups.

Table 2. Failure modes of test groups.

Groups	Failure Modes(%)		
	Adhesive	Cohesive	Mix
Control	65	0	35
% 10 Na_2CO_3	95	0	5
% 10 $CaCl_2$	60	0	40
% 15 Na_2HPO_4	100	0	0
% 23,1 CLG	100	0	0

No studies where the efficiency of the CLG solution, added to shorten the setting time of MTA and reduce its fluidity in bonding to the reticular dentin, have been published. Among the test groups to which an accelerator was added, least bonding strength was found in the 23.1% CLG with 1.36 MPa. In our opinion, both the chemical and the mechanical bonding in the 23.1% CLG group demonstrated that the lowest bonding strength is weak. CLG, with good Ca^+ deionization kinetics, towards dentin as Ca^+ ions move towards cement pores while the cement setting reaction is catalyzed. Poor mechanical bonding may be related with surface morphology of MTA enriched with 23.1% CLG. SEM images obtained in our study revealed large particles and smooth surfaces in the CLG group. Also, poor mechanical bonding may also be attributed to the surface morphology of CLG group. We are of the opinion that retention between dentin tubules and cement interface may be poor due to such large particles. The literature review revealed no studies assessing the impact of the Na_2CO_3 catalyst, when added to MTA, on bonding to the root canal dentin. In our study, the highest bonding strength appears in the 10% Na_2CO_3 group with 9.91 MPa.

3.3. SEM Analysis.

In the SEM examination, globular and angular shapes of variable sizes were observed in the MTA control group (Figure 3A). In Na_2CO_3 group, irregular small-sized particles positioned on surface were observed (Figure 3B). The $CaCl_2$ group showed a slight granular surface, which consisted of small and globular crystals with the undeveloped hexagonal plates (Figure 3C). The Na_2HPO_4 group showed large irregular and hexagonal crystals, and the surfaces of the irregular structures appeared uneven (Figure 3D). The characteristic "rose leaves" view was observed in the Na_2HPO_4 group. CLG group showed a relatively smooth surface with a marked increase in size and decrease in number (Figure 3E).

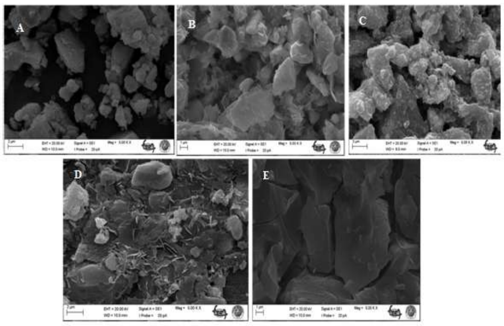

Figure 3. SEM pictures of. MTA test groups. A: Globular and angular shapes in control group. B: irregular small-sized particles in Na_2CO_3 group. C: Small and globular crystals with the undeveloped hexagonal plates in $CaCl_2$ group. D: Large irregular and hexagonal crystals in Na_2HPO_4. E: A relatively smooth surface in CLG group.

4. CONCLUSIONS

Using of Automatic Vicat needle may be advice to standardize the setting time test. All catalysts of 10% $CaCl_2$, 15% Na_2HPO_4, 10% Na_2CO_3, and 23.1% CLG were found to decrease the setting time of MTA. Although the fastest setting was in the 23.1% CLG group, the decreases in physicochemical properties limit its clinical use. Considering other parameters besides time, the study reveals that, although the 15% Na_2HPO_4 and 10% Na_2CO_3 groups do not have the fastest setting time, they stand out with the increases dentin push-out strength. We believe that push-out strength data will contribute to the literature.

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