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Effect Of Various Additives On Setting Time & Hardness Of Mineral Trioxide Aggregate - An In Vitro Study

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Abstract
Mineral Trioxide Aggregate (MTA) since its introduction has proven to be an ideal retrograde material with many merits. Delayed setting time is one of the demerits of MTA which may limit its use in endodontic procedures. The purpose of this study is to identify types of MTA additives to enhance its setting properties and their effect on hardness of MTA. Additives tested included sodium phosphate, citric acid, calcium chloride and sodium chloride. The setting times and surface hardness were evaluated and it was observed that although the addition of additives significantly decreased the setting time of MTA, the hardness of set materials also got compromised. This combination might be a viable option in procedures where hardness of the material is not a critical issue.

Key Words
Mineral Trioxide Aggregate (MTA); Citric Acid; Sodium Phosphate Monobasic; Vickat apparatus; Vickers Hardness Test.

Introduction

It is estimated that over 24 million endodontic procedures are performed annually with up to 5.5% of those procedures involving endodontic apical surgery, perforation repair and apexification treatment.[1] Endodontic surgery is performed to resolve inflammatory processes that cannot be successfully treated with conventional techniques which may be due to complex canal and/ or apical anatomy and external inflammatory processes.[2] Surgical procedures may be indicated for the resolutions of misadventures including root perforations that may occur either during canal instrumentation or during post space preparation.[2],[3] Surgical treatment usually involves placement of a material designed to seal root canal contents from the periradicular tissues and repair root defects.[2]. This material should demonstrate the ability to form seal with dental tissues while also exhibiting biocompatible behavior with periodontal tissue.

Throughout the dental history, a wide variety of materials like Gold foil, silver posts, titanium screws, tin posts, amalgam and gallium alloys, ZnOE, IRM, Super EBA, Cavit, Zinc polycarboxylate, Zinc phosphate, Glass ionomer cement, Bone cement and other materials like Composite, Gutta percha, Ceramic inlay, Teflon, mixture of powdered dentin and Sulfathiozole and Cyanoacrylates have been used for retrograde fillings and perforation repair. Although a plethora of materials are available, none has been found that fulfils all or most of the properties for an ideal root end filling material. [4].

Mineral Trioxide Aggregate or MTA is a new biocompatible biomaterial with numerous exciting clinical applications in the field of endodontics. It has been used on experimental basis by endodontists for several years with anecdotally reported successes, some of it quite impressive [5]. Its first description was in "The Dental Scientific Literature" in 1993 by Lee and Torabinejad [6]. It was introduced by Mahmoud Torabinejad and colleagues at Loma Linda University. Its acceptance by the "US Federal drug Administration in 1998" led to its widespread use and became commercially available as ProRoot MTA. Since its introduction as a root end filling material, the use of MTA has expanded to many applications of root repair and bone healing. These applications include direct pulp capping, repair of root and furcation perforations and apexification. Although MTA has many favorable properties that support its clinical use, there are several drawbacks. The setting time of MTA has been reported to be about 3 hours, which means less shrinkage and better marginal adaptation .This makes its use difficult because of its initial looseness and compromised initial hardness. Hence an accelerator is required to improve the setting time of MTA, so that its utility in single visit endodontic procedures is enhanced. The effect of mixing MTA powder with different liquids has shown that the choice of preparation liquid can have an effect on setting time and compressive strength. Additives like saline, 2% lidocaine, 3% NaOCl gel, Chlorexidiene gluconate gel, K-Y jelly have been tried to evaluate the effect on setting time of MTA.[7]

The purpose of this in vitro study was to identify types of additives required to achieve optimal setting properties of MTA and to determine the effects of these additives on the hardness of the set MTA.

Materials And Methods

The endodontic material used in this

study was ProRoot MTA (Dentsply/Tulsa Dental, Tulsa, OK). Additives tested included Sodium Phosphate Monobasic (15% Na2HPO4), 0.2% Citric acid, 2% Calcium chloride (2% CaCl2), Sodium chloride (2% NaCl), Control group consisted of sterile water. The samples were divided into following groups:

I. MTA + 15% Sodium Phosphate Monobasic (15% Na2HPO4)
II. MTA+ 0.2% Citric Acid
III. MTA + 2% Calcium Chroride (2% CaCl2)
IV. MTA +2% Sodium Chloride (2% NaCl)
V. Control Group (MTA+ Sterile Water)

Setting time was determined with a Vicat apparatus. The Vicat apparatus was assembled with an indenter needle that has a flat end with a diameter of 1.0 +0.02 mm. MTA powder was mixed with the respective additives in 3:1 powder liquid ratio. The Vicat indenter needle was placed on the samples with a load of 300 g. The setting time was recorded when the indenter needle failed to create an indentation in three separate areas of the sample within the sample. Samples were assessed at the following time intervals: 5 min, 10 min, 15 min, 20 min, 25 min, 30 min, 40 min, 50 min, 60 min, 1.5 h, 2 h, 2.5 h, 3 h, 3.5 h, and 4 h. All samples after setting were subjected to Vickers Hardness Test for estimating the effects of the additives on hardness of MTA. Vickers hardness machine consists of indentation the test material with diamond indenters in the form of a right pyramid with a square base and an angle of 136 degree between opposite faces subjected to a load of 1 to 100 kg. The load is normally applied for 10 to 15 seconds. The following formula was used to calculate the Vickers Hardness number (VHN) in Kg/mm2: VHN = 1.854 p/d2 (Where: P= load, D= average of diagonals.)

Results And Observation

Under the limitations of this study following observations were made:**(Table I)**

Comparison Of Effect Of Samples On Setting Time & Hardness Of Mineral Trioxide Aggregate

S No	Groups	Setting Time (Min)	Hardness (Kg/Mm2)
1	Group I Mta + 15% Na2hpo4	25	3.050
2	Group II Mta + 0.2% Citric Acid	45	3.121
3	Group III Mta+ 2% Cacl2	35	2.064
4	Group IV Mta+ 2% Nacl	100	2.6660
5	Group V Mta+ Sterile Water	120	4.880

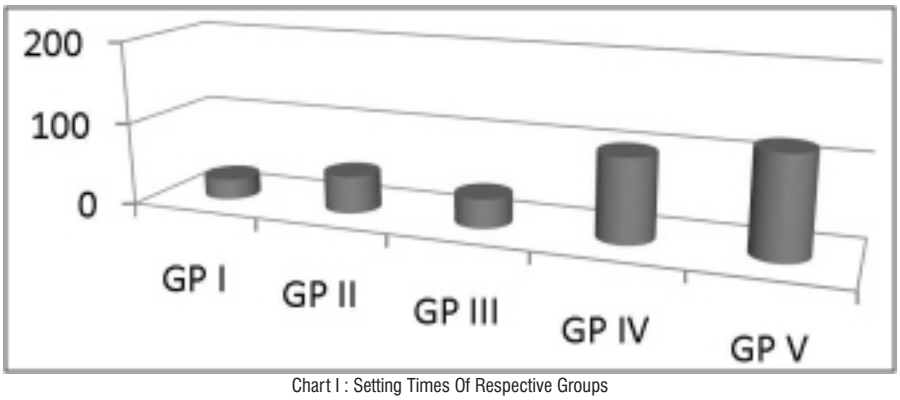

Chart I : Setting Times Of Respective Groups

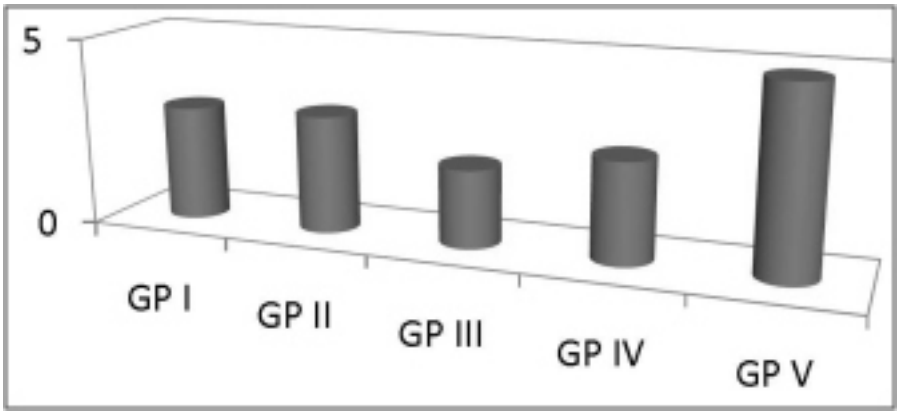

Chart II : Hardness Of Samples

1. Addition of 15% Sodium Phosphate Monobasic reduced the Setting time and hardness of MTA
2. Although 0.2% Citric Acid reduced the setting time, it had little effect on the Hardness.
3. 2% Calcium Chloride and 2%Sodium Chloride also reduced the setting time and hardness of MTA.

Discussion

The purpose of this study was to improve the handling characteristics of MTA by the use of certain accelerators. Delayed setting times may limit the use of mineral trioxide aggregate (MTA) in endodontic procedures. There have been reports that the amount of sulphur at the surface of set MTA is three times higher than the powder forms of MTA and this layer protects the cement from further hydration and increases the setting time. The longer setting time of MTA can be attributed to the lower levels of sulphur and tricalcium aluminate in MTA. [1],[8] Most accelerants for MTA are calcium compounds with high solubility typically used for calcium ion supplements.[9]

Sodium phosphate monobasic (Na_2HPO_4) is recommended as a liquid phase accelerator due to bio compatibility. Studies have proved that Na_2HPO_4 solution could be used as a cement liquid to accelerate the setting of the cement prepared from tricalcium phosphate as a cement powder. Phosphate increases the rate of hydroxyapatite formation. Strong ionic interactions between phosphate and calcium ions lead to the formation of calcium silicate hydrate phase. Chow et al have suggested that the rate of hydroxyapatite formation, an indicator of the setting reaction in the calcium phosphate cement system, could be increased by the presence of phosphate in the solution. This phosphate solution may influence the kinetics of the setting reaction.[10] Citric acid can also be used as an potential accelerator in concentrations of 0.1%, 0.2% & 0.4%. It has mild antibacterial properties and is also used as an irrigant. It is known to remove smear layer.[1]

Although there are many types of accelerators, calcium chloride ($CaCl_2$) continues to be one of the most preferred owing to its excellent biocompatibility. Calcium chloride is classified as a Type C accelerating chemical admixture for

concrete by ASTM C494. Calcium chloride has the ability to accelerate cement hydration and reduce set time by as much as two thirds.It is also known to reduce the rate of micro leakage.[7] In addition, Accelerated Portland Cement (APC) containing $CaCl_2$ as the accelerator has been shown to be nontoxic and may have the potential to promote bone healing. (Abdullah D et al 2002).

Sodium chloride in low concentration of 2% -5% has been used as accelerator for controlling the rate of setting time of a variety of cements both in dentistry and otherwise. This fact can be attributed to its ease of availability and biocompatible nature. However if used at higher concentrations sodium chloride acts as a retarder and slows the setting reaction of the cement.

In the present study, the effect of additives on the setting time of mineral trioxide aggregate was also assessed. Setting time was estimated using a Vicats apparatus. There are different methods for estimation of setting time of endodontic cement. The initial setting time of cement can be assessed using a Vicat needle (ASTM C191-04, 100-g flat-end indenter with a diameter of 1.0 mm), Gilmore (ASTM C266-03,113.4-g indenter with a diameter of 2.12 mm), or an indenter (ISO 6876:2001, 100-g indenter with a diameter of 2 mm). "However, there is no uniformly accepted test procedure for the initial setting time of MTA".[11],[12] The rationale behind choosing Vickats apparatus was due to its ability to assess the setting time of the sample over short intervals.

The surface hardness is another important property that needs to be considered in addition to setting time. Holt DM et al evaluated the Compressive Strength of Two Types of Mineral Trioxide Aggregate Mixed with Sterile Water or 2% Chlorhexidine Liquid. It was concluded that addition of chlorexidiene as a liquid reduced the strength of mineral trioxide aggregate.[13] Torabinejad et al. showed that the hardness of MTA has discrepancy which can be attributed either to differences in protocols that were followed in testing for the hardness and/or changes in the composition of the MTA powder since it was first introduced. [14]

Conclusion

Under the limitations of this study it could be concluded that although the tested additives significantly reduced the setting time and hardness of MTA. Of the tested samples, both 15% sodium phosphate monobasic and 0.2% citric acid significantly reduced the setting time of MTA. The hardness of MTA decreased on addition of the tested additives, but it was far less than what was observed in case of 2% Calcium Chloride and 2% Sodium Chloride.

This combination might be a viable option in single visit procedures where hardness of the material is not a critical issue. Further studies are indicated to incorporate some of the additives tested into clinical practice. The mixtures that showed potential benefits should be tested for their biocompatibility, antimicrobial properties, and sealing abilities before any recommendation for clinical use.

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