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Post retention with mineral trioxide aggregate and accelerated Portland cement

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**POST RETENTION WITH MINERAL TRIOXIDE
AGGREGATE AND ACCELERATED PORTLAND CEMENT**

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Thesis submitted to the
School of Dentistry
at West Virginia University

In partial fulfillment of the requirements
For the degree of
Master of Science
In Endodontics

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Morgantown, West Virginia
2005

ABSTRACT

POST RETENTION WITH MINERAL TRIOXIDE AGGREGATE AND ACCELERATED PORTLAND CEMENT

Pamela P. Harrington, D.D.S.

Perforations occurring during post placement are problematic. Mineral trioxide aggregate (MTA) is slow to set. A trial demonstrated that CaCl_2 accelerated the set of Portland cement. Forty single-rooted teeth were sectioned, treated with endodontic therapy and post prepared. The first group contained 20 teeth, evaluated at 72 hours. MTA, Portland cement with 10% CaCl_2 , 15% CaCl_2 and zinc phosphate were used as post cements. The second group was evaluated at 28 days. The posts were cemented with either MTA or zinc phosphate. Instron mechanical testing was used to determine forces needed for post dislodgement. The 72 hour group showed statistically significant higher resistance to dislodgement with zinc phosphate. ($p < .05$) No significance was found between resistance in the experimental groups. The 28 day group posts seated with zinc phosphate had a higher resistance to dislodgement than those with MTA. ($p < .05$) At 28 days, MTA was found to be 28 % weaker than zinc phosphate.

DEDICATION

For Ryan.

ACKNOWLEDGEMENTS

To the West Virginia Staff and Residents: It is said that a child laughs one hundred times a day, yet an adult laughs only four to five times a day. Thanks for making me feel like a kid.

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CHAPTER I

INTRODUCTION

The placement of posts is necessary in certain endodontically treated teeth.(1) Iatrogenic perforations during post placement can compromise long term prognosis. Microorganisms pass through the perforation site and lead to the subsequent destruction of the surrounding periodontal ligament and bone.(2) Mineral trioxide aggregate, MTA, is a new material approved by the Food and Drug Administration in 1998. It has been shown to have many clinical applications in endodontics: direct pulp-capping, pulpotomy, root end filling material, lateral and furcal perforation repair, apexification and internal resorption.(3-5) Underlying these many uses of MTA, are the properties of MTA. MTA has been shown to be highly biocompatible, to form fibroblast attachments, and to promote bone healing.(6;7)

In situations where post placement perforates the root, MTA would be ideal. Placement of perforation repair material should occur at the time of the incident to lessen the inflammatory response.(2) Dental cements have a mild to moderate inflammatory pulpal response.(8) MTA has a reparative pulpal response.(9;10)

One disadvantage of MTA as a restorative material has been its long setting time of two hours and 45 minutes.(11) MTA is 75% Portland cement. Abdullah et al. reported the setting time of Portland cement was successfully reduced by adding calcium chloride.(12) This SEM study provided evidence that accelerated Portland cement was non toxic and

biocompatible. This study did not, however, report the time of set with the addition of calcium chloride.

Recently, Vargas et al. studied the retentive strength of MTA with that of a glass ionomer cement and zinc phosphate. MTA was found to be less retentive when compared to the other cement. However, this study did not use vibration to seat the posts.(13) Yu Z and associates found the use of ultrasonics to be effective in reducing cement film thickness.(14) Portland cement, and similar cements such as MTA, may require vibration to fully seat the post. MTA or accelerated Portland cement may both prove to have similar retentive properties when compared to traditional cements and seated with ultrasonic vibration. There, as of yet, has been no study evaluating the use of either MTA or accelerated Portland cement as a luting agent for posts when seated in such a manner.

Significance of the study

Perforations that occur during post placement are difficult to repair. Some of these perforations can not be surgically treated. The current practice is to place a moist pellet and temporary over the MTA for 72 hours to allow for setting.(15) A faster setting material would protect the site and eliminate this second visit. Thus, a bone reparative material, such as MTA, that could repair the perforation and simultaneously seat the post would be ideal.

Statement of the problem

Does mineral trioxide aggregate or accelerated Portland cement have significant strength after setting to be used as a post cement?

Hypothesis

There is no statistical difference in the force required to dislodge a stainless steel ParaPost from the following cements: MTA, Portland Type I Cement with 10% calcium chloride, Portland Type I Cement with 15% calcium chloride, Portland Type I Cement with IPANEX Rapid and zinc phosphate cement.

Definition of terms

- **Admixture**-a material that is used as an ingredient in concrete and is added to the batch immediately before or during mixing.
- **Calcium chloride**-a soluble salt admixture which accelerates the set time of Portland cement.
- **Dental cements**-dental materials that are classified according to their use. These materials are used as luting agents, insulators, temporary and permanent restorations, root canal fillings and pulp caps.
- **Gillmore Needles**- two needles with standardized needle head and weight used to determine initial and final set times. Initial set needle with 1/12 inch head and ¼ lb load. Final set needle with 1/24 inch head and 1lb load.
- **Gypsum**- (calcium sulfate) a material which reacts with calcium chloride to accelerate set of cements. MTA is 5% gypsum.
- **Instron Machine**- a testing machine designed to measure the mechanical properties such as the amount of tensile force required to remove posts from tooth roots.
- **IPANEX Rapid**- proprietary liquid admixture which accelerates the set of Portland cement.

- **Mineral trioxide aggregate(MTA)** -is a derivative of Portland cement made of fine hydrophilic particles whose main components are tricalcium silicate, dicalcium silicate, tricalcium aluminate , calcium sulfate dihydrate, tetracalcium aluminoferrite and bismuth oxide.
- **Obturation** - the phase of endodontic treatment in which the root canal system is filled. This forms a fluid tight seal through out the root.
- **PATCH 29 (CGM Inc., Bensalem, PA)** - Portland cement mixture made from four parts Portland cement to one part calcium aluminate.
- **ParaPost drills (Whaledent Int. New York, NY)** – Instrument used for creating a corresponding post space for a ParaPost dowel.
- **Perforation** - a pathologic or iatrogenic communication between the root canal space and the periodontal ligament.
- **Portland cement** -an inorganic cement fired and then made of ground clay and lime which reacts with water to produce calcium aluminates and calcium silicates.
- **Post Space** - a space within an endodontically treated tooth to allow for a post.
- **Potassium aluminate** -an admixture which accelerates the set of Portland cement.
- **Retention** - the ability to withstand an upward vertical force exerted by the Instron testing machine.
- **Retrofill material** - a root end filling material placed surgically to close the apex of a root.
- **Stainless steel ParaPost (Whaledent Int. New York, NY)** - Parallel-sided, prefabricated, serrated, vented post system made of stainless steel.
- **Zinc Phosphate (ZnPO_4) cement** - a dental cement consisting of a ZnO powder and aqueous phosphoric acid that when mixed form an insoluble salt.

Assumptions

The following may be assumed in this study:

1. Gillmore needles measure approximate set time.
2. MTA and accelerated Portland cements are biocompatible.
3. The Instron machine accurately demonstrates post dislodgement.

Limitations

1. Set time is affected by the amount of mixing and the humidity in the air.
2. There is a small amount of error associated with measurements.
3. There are natural biologic differences associated with the dentition of different teeth.
4. Operator variability in tooth preparation.

Delimitations

1. The set time of Portland cement admixtures was determined by Gillmore Needles.
2. 40 single rooted teeth with straight canals were used.
3. A 9mm post space
4. Corresponding stainless steel parallel-sided, vented, abraded, prefabricated Paraposts were used.
5. The follow cements were used as experimental cements: MTA, ProRoot white formula, Portland Type I Cement with 10% calcium chloride, Portland Type I Cement with 15% calcium chloride and Portland Type I Cement with IPANEX Rapid. Zinc Phosphate cement was used as a standard for comparison.
6. A special mounting apparatus was made to align posts.

7. Instron Mechanical Testing Machine cross head was set to move at a constant rate of .025 inches/min.

CHAPTER II

LITERATURE REVIEW

The studies in post retention have been generally confined to length, surface configuration, shape, cement type and cement thickness. In a classic study by Standlee et al.(16) it was shown that retention was greater with posts that were long, rather than short, rough rather than smooth and parallel rather than tapered.

Post retention is influenced by length. As the post is made longer, forces are spread over a greater surface of the tooth. This enables the tooth to withstand horizontal forces.(17) Johnson and Sakumuras found that an increase in length from 7mm to 11mm increased retention by 30%.(18) It has been shown that the apical seal of endodontically treated teeth remains with 4 mm of gutta percha.(19) This suggests that the dentist should make the post as long as possible.

Surface configuration is a variable relating to post retention. Standlee et al. found an increase in retention with roughened or serrated posts.(20) Likewise, abrasive blasting of metal posts was found to have superior retentiveness. (21)

Another consideration in post system configuration is shape. The superior retentive abilities of parallel-sided posts over tapered posts have been demonstrated. Standlee compared the retentive capacity of prefabricated posts and found the tapered the least retentive.(22) However, recently in a study ascertaining the effect of endodontic taper on post retention, it was found that a .04 taper was more retentive than a .02 taper.(23)

It would seem logical that increase in diameter would increase resistance to dislodgement. However, studies on the effect of diameter on retention are variable. Many have recommended a post diameter that is one third the root diameter.(24) Krupp found that post length is the most important factor and that diameter was secondary.(25) Conversely, Standlee showed that varying the diameter had no effect on dowel retention.(26)

Cementation type has been studied in its relation to retention. Goldman et al. reported higher retentive values with resin cement than zinc phosphate.(27) Schwartz found just the converse.(28) Some studies have shown no correlation between type of cement used and post retention.(29;30) Colley et al. suggest that the type of cement is not as important as the film thickness.(31) Standlee agrees.(32) This study states that prefabricated parallel-sided posts were not well suited for teeth with conical or ribbon shaped canals. This mismatch could lead to increased cement thickness. It was this cement thickness that was the retention factor. Thus, long, parallel rough posts have been found to have superior retention. In contrast, the correlation between cement type, cement thickness and diameter with post retention is unclear.

Post placement can lead to root perforation. A new class of material called MTA, mineral trioxide aggregate, has been developed to help solve problems when communication occurs between the root structure and tissue. Mineral trioxide aggregate is widely used as a perforation repair and root end filling material. Low microleakage, biocompatibility and antimicrobial properties have been cited as desirable attributes for these materials. Torabinejad et al. found MTA leaked significantly less than amalgam and Super EBA when used as a root end filling material.(33) Lee and colleagues reported that in lateral perforations, MTA leaked less than IRM or amalgam.(34) Conversely, canals obturated with

MTA showed significantly more leakage when compared to those obturated with gutta percha.(35)

Another necessary property for a root perforation material is biocompatibility. Balto studied the attachment and morphology of human periodontal ligament fibroblasts.(36) Attachment of these cells to MTA was demonstrated with a scanning electron microscope. Cementum was reported to have formed over MTA in an *in vivo* study by Torabinejad.(37) An *in vitro* cytotoxic screening was conducted by the same author. Here two methods were used. In the agar overlay method, amalgam was found to be less toxic than MTA. However, this method prevents the direct contact of the test material. In the radio chromium release method, MTA was less toxic than amalgam.(38) MTA was shown to stimulate cytokine release and interleukin production.(39) Thus, MTA is speculated to promote hard tissue formation.

Initially, calcium and phosphate ions were speculated by Torabinejad to be responsible for MTA's biocompatibility with bone.(40) Later, in animal studies, Torabinajad reported the regeneration of new cementum over MTA. Here he sited the materials high pH and sealing ability as properties that may have activated the cementoblasts.(41) Recently, Fridland and Rosado found that calcium hydroxide was the main compound released by MTA in water.(42) Sarkar and associates studied the interactions of MTA with a synthetic tissue fluid composed of a neutral phosphate buffer solution. The precipitate they found was similar in composition to hydroxyapatite. It was concluded that calcium was the dominate ion released and that it reacted with phosphate to form hydroxyapatite.(43) The exact mechanism responsible for MTA's biocompatibility remains unclear.

Ideally dental materials would be antibacterial. Torabinejad et al. tested MTA, amalgam ZOE and Super EBA. While MTA did have an effect on facultative bacteria, there was no effect on strict anaerobes.(44) MTA's high pH may have an antibacterial effect.(45)

Underlying these unique properties, are the newly discovered uses of MTA. ProRoot MTA is registered with the FDA and indicated for apexification, pulp capping, internal resorption, repair of root perforations, and root-end filling. Torbinajad found that MTA was similar to calcium hydroxide in root end induction or apexification.(46) Aeinehchi et al. showed that MTA demonstrated less inflammation and necrosis when compared to traditional calcium hydroxide as a pulp capping agent.(47) MTA has been used successfully in a case study of repair of internal resorption.(48) MTA has been mostly used as a perforation repair material and retrofill material. Craig et al. in a long term study of MTA as a root perforation repair material found all 16 cases demonstrated radiographic resolution of lesions associated with perforations at the 12 to 45 month follow up visit.(49)

Thus, MTA has many advantages for use as a post cement when perforation occurs. However, long set time and low compression strength may be disadvantages.(50) Set time and compression strength of MTA vary in the literature. Initial work with MTA was done by Dr. Mahmoud Torabinejad. This study used the grey form of MTA. It showed an initial low compressive strength of 40 MPa that increased to 67 MPa in 21 days. The set time was two hours and 45 minutes.(51) MTA is available in a grey and newer white form. White ProRoot MTA was formulated because the grey had esthetic problems. The difference is primarily a result of the lower iron oxide in the white formula. The white differs from the grey by less than six percent.(52) Recent analysis of both the white and grey formulas of MTA gives compressive strengths of 68MPa and 58MPa respectively. However, much longer set times were noted. Here MTA grey formula was set in six hours and 30 minutes

while the white formula was set in six hours. The setting time in the mouth was much shorter, four hours.(53)

Sluyk et al. studied the effect of time and moisture on the setting and retention of MTA as a furcation perforation repair material.(54) While the setting and compressive strengths of these materials have been shown to result from water,(55) this study differed. The results of this study showed no difference in resistance to displacement with the use of a wet or dry cotton pellet. The difference was seen between the 24 hour set time and the 72 hour set time. The 72 hour set time had a greater resistance to dislodgement than the 24 hour set time.

MTA is composed of 75% Portland cement and has similar chemical properties.(56) The main difference between Portland cement and MTA is the addition of bismuth oxide, which makes the aggregate radiopaque.(57) Portland cement is the binding ingredient in ordinary concrete mixes and is classified as a hydraulic cement.(55) The essential composition of Portland cement is as follows: 20% silica, 10% alumina plus ferric oxide, 5% other compounds and 65% lime. Lime is composed of calcium and magnesium oxides. Portland cement is produced by grinding clay and these lime-bearing minerals in the correct proportions and then heating the mixture to 1400° C. This process called *calcination* produces physical and chemical changes in the raw materials. The resulting “clinker” is ground to a fine powder and a small amount of gypsum is added to retard the setting process. The properties of Portland cement, in large part, are due to its composition.(58)

The set of Portland cement results from a complicated hydration reaction. Two principle constituents are tricalcium silicate (3 CaO-SiO₂) and dicalcium silicate (2 CaO-SiO₂). An example of one such reaction is:

Here x is dependant on how much water is available. These end products are in the form of complex gels or crystalline substances.(58) Portland cement sets in a series of stages; first, there is dispersion of clinker grain in water. Then hydration products eat into and grow out from the surface of each grain. Next, setting occurs when the different clinker grains join. Finally, hardening occurs with further development of the gel. Crystalline particles are disseminated throughout. The forces that bind the colloidal particles together in the gel are speculated to be hydrogen bonds, Vander Waals forces, ionic attractions and covalent bonds such as Si-O-Si bonds.(55) Part of the water will be consumed by the reaction. The other part of the water will be trapped in the pores. Evaporation can occur during or even after setting. This water that is lost will refill in an “osmotic effect”. During setting the continuity of the capillary system is broken.(59) The hydration of the powder produces tricalcium silicate, tricalcium phosphate, tricalcium oxide and others.(55)

Admixtures have been used to accelerate the set of Portland cement. One solution to the slow set of Portland cement has been the addition of calcium chloride. Calcium chloride has been reported to produce a 50% reduction in both the initial and final set of Portland cements.(60) In a series of exothermic reactions, the salt penetrates the pores resulting in faster silicate hydration. In the earlier literature, the action of calcium chloride was speculated to be one of a catalyst.(61) Now it is thought that calcium chloride may combine with cement minerals.(60) While the exact mechanism is still unclear, the reaction of calcium chloride has been shown to work by increasing the hydration of the calcium silicates. The silicates are thought to crystallize more rapidly. This results in a faster set.(61) Calcium chloride is known to change the strength, chemical composition, surface area, and pore characteristic of Portland cement.(60) This admixture has the reported advantage of increasing compressive strength and decreasing permeability.(60) One disadvantage of

calcium chloride admixtures is that final cement can be corrosive. Another admixture which accelerates the set of Portland cement is the addition of calcium or potassium aluminates. These act directly on cement hydration. The disadvantage of this admixture is a decrease in final compressive strength.(60) Thus, an admixture may not only decrease set time but also change the compressive strength. Decreasing set time may allow MTA, a Portland derivative, to be used as cement for a post.

The purpose of this study is twofold. The first is to evaluate the set time decrease associated with the addition of accelerating agents to Portland cement. The second is to determine if MTA or the accelerated Portland cements can be used as post cements.

CHAPTER III

MATERIALS AND METHODS

Sample Description

A trial study was done with admixtures which accelerated the set of Portland cement. Those admixtures which significantly decreased the set time of Portland cement and contained materials which were non toxic were chosen for the post study. Forty, single rooted teeth were sectioned off at the CEJ and conventionally treated with endodontic therapy and prepared to receive a post. From here post spaces were made and posts seated with the cements. The roots were then stabilized in acrylic resin in a manner which ensured that the posts were parallel. Instron mechanical testing was used to determine the force needed for post dislodgement.

Different types of admixtures which were reported to accelerate the set of Portland cement were studied. 50 gram samples of Portland Type I Cement, Portland Type I Cement with 15% calcium chloride by weight, Portland Type I Cement with 10% calcium chloride by weight, Portland Type I Cement with IPANEX Rapid, a liquid admixture, and Patch 928, a Portland cement and crystalline silica mix were tested for set time. Appendix B show how these materials were mixed. Those samples which had adequate set times and properties were selected as cements for the post retention evaluation.

Recently extracted teeth stored in 35% peroxide, 35% alcohol, 10% glycerin and 20% water were used for post preparation. A total of 40 single rooted teeth were selected. Those

teeth, when viewed with 2.6 microscopic lenses, with visible fractures or damage on extraction were eliminated. Any teeth with abnormal morphology in the canal structure such as calcified or ribbon shaped canal were omitted. An exemption from the Institutional Review Board for the Protection of Human Research subjects was obtained because the teeth could not identified to a patient. (Appendix A)

Set Time Methodology

The set time of Portland cement admixtures was evaluated prior to post retention determination. Thus, the first part of this study determined the time of set for various admixtures; Portland Type I Cement, Portland Type I Cement with 10% and 15% calcium chloride by weight, Portland Type I Cement with IPANEX Rapid, a proprietary liquid admixture, and Patch 928, a Portland cement and crystalline silica mix. The standard method of testing the time of set for hydraulic cement, A.S.T.M. designation C191-44, was used. Four, 50 gram samples of each of the four cements were mixed. Calcium chloride was ground to a fine powder. Room temperature water was measured with a Monoject syringe. The cements were vigorously hand mixed for one minute. Immediately after mixing the cement, the sample was placed on a glass slab and molded to a depth of 5-6mm. A digital timer and Gillmore needles were used to determine set. The needles were held 2-4mm above the mold. The *initial set* was said to have occurred when the initial needle ceased to visibly sink into the mold after being released. The *final set* was said to have occurred when the final needle ceased to visibly sink into the mold after being released.

Figure 1. Gillmore needles

The needle could mark the surface as long as it did not indent the surface. Testing was done until markings were uniform. Care was used to keep the needles clean. The time of set was tested from initial mix until *final set* was achieved.

Root Preparation Methodology

Roots were prepared by sectioning extracted single rooted teeth horizontally at the cementoenamel junction using a fissure bur and a high speed hand piece. The root surface was notched with the same bur to allow for retention when finally stabilized in acrylic resin. A number 10 Sureflex ISO hand file (Dentsply, Tulsa Dental, Johnson City, TN) was placed in the canal until it appeared through the foramen. One millimeter was then subtracted from this length to obtain working length. Sureflex ISO hand files number 10 through 20 were used for initial cleaning of the canal and pulp removal. The apical portion of the canal was enlarged with a step back series of number 2, 3, 4 Gates Glidden burs. (Dentsply, Milford, DE) Instrumentation was completed with .06 ProFile Ni-Ti rotary files. The files were used in a crown down fashion until working length was achieved. The canals were irrigated with 5.25% sodium hypochlorite between filing and as a final rinse.

After final instrumentation, the roots were dried with paper points. The roots were then obturated with Roth Root Canal Cement (Type 801 Elite grads, Roth International, Chicago, IL) and gutta percha from the Obtura II. (Obtura Spartan, Fenton, MO) The heated gutta percha was vertically compacted with Schilder pluggers (Obtura Spartan, Fenton, MO) until the canals were filled level to the occlusal table. Three to four millimeters of gutta percha was then removed with a round bur and slow speed hand piece. An IRM temporary (Caulk Division, Milford, DE) was placed to seal the canals. The specimens were then stored in 100% humidity.

Post Space Methodology

ParaPost® XP stainless steel posts were abrasively blasted using a sand blaster (Integral Systems, Inc. Culver, City, CA) and rinsed with water prior to use. Radiographs

were taken of all the obturated roots. By visual approximation, dowels were chosen. From these dowels, ParaPost drills were selected. These corresponded to the following ParaPost drill sizes; yellow with 1.00 mm in diameter, blue with 1.14mm in diameter and red with 1.25mm in diameter. Those with larger or smaller root canal systems were discarded. Gates Glidden drills were used to remove gutta percha to a 9mm length from the occlusal table. Dowel preparation was made with the corresponding dowel drill. If the ParaPost drill was short of the 9 mm depth, the next larger ParaPost drill was used until the 9mm depth was achieved. A stopper was placed on the corresponding dowel at 9mm. The dowel was inserted into the canal space to ensure proper adaptation. Those lacking conformity with the post space were rejected. At times it was necessary to reinsert the last ParaPost drill used into the post space to ensure proper depth. Any tooth debris from the canal was removed by irrigation with water. The canal space was dried with paper points. Follow up radiographs were taken to ensure that all gutta percha was removed from the canal space. The ParaPost that corresponded to the last drill was selected for cementation.

Cementation Methodology

Parapost head tabs were sectioned off from the posts and smoothed. Four cements were used: Hy-Bond Zinc Phosphate Cement (Shofu Dental Corp., San Marcos, CA), ProRoot MTA white formula (Dentsply, Tulsa Dental, Johnson City, TN), Portland cement with 10% CaCl_2 , and Portland cement with 15% CaCl_2 . Hy-Bond Zinc Phosphate cement and ProRoot MTA were mixed according to manufacturers' instructions. The accelerated Portland cements were mixed 5 grams of cement to 1.5 grams of distilled water. Post spaces were dried with absorbent paper points. ParaPosts were inserted with finger pressure and ultrasonic vibration. (Sybon Mini Endo, Sybron Dental Specialties, Glendora, CA) Finger

pressure was used on the top of the post while an ultrasonic tip was placed on the side. The posts were vibrated into place. Excess cement was removed flush with the top of the root. After cementation, the samples were wrapped in a moist paper towel and sealed in an air tight container during setting time.

Figure 2. Mineral Trioxide Aggregate

Figure 3. Portland Cement

Stabilization Methodology

A surveyor and special mounting apparatus were used during mounting procedures to ensure that the posts were parallel. A Masserann Micro Kit (Union Broach Corp., Long Island city, NY) was used. The yellow, blue and red ParaPosts were fitted into the 14, 15 and 16 size hollow trepans from the kit. The trepans were inserted into a surveyor to ensure the same alignment for all posts. Fasttray acrylic resin (Bosworth Company, Skokie, IL) was mixed and placed into the one inch copper ring. The cemented post within the root was then inserted into the trepan. This assembly was then lowered into the acrylic. The assembly was held in place until the acrylic hardened around the root. The specimens were wrapped in a moist paper towel and sealed in an air tight container until time for tensile testing.

Figure 4. Special Mounting Apparatus

Dislodgement Methodology

Force required to dislodge the posts was determined on an Instron Mechanical Testing Machine. (Instron Corp., Canton, MA) A special mounting jig was made to mount the copper ring which held the roots placed in acrylic. The Instron Universal Testing Machine was set with a constant rate of .025 inches/ min. Failure was reported to be the maximum load reached prior to post displacement. This was recorded for each sample. A total of 40 samples were tested. 20 samples were tested at 72 hours. Five samples each of ProRoot MTA, Portland Type I Cement with 10% calcium chloride and Portland Type I Cement with 15% calcium chloride were tested against five samples of zinc phosphate. 20 samples were tested at 28 days. Ten samples of zinc phosphate were tested against ten samples of MTA. Samples were examined for post slippage as well as acrylic or root displacement with a Zeiss intraoral microscope after Instron testing.

Figure 5. Instron Universal Testing Machine with special mounting jig

Equipment and Materials

- Portland Type I cement
- Pro Root MTA (Dentsply, Tulsa Dental, Johnson City,TN)
- Calcium Chloride (Fisher Scientific, Fair Lawn, NJ)
- Gillmore needles (ASTM International,West Conshohocken,PA)
- Monoject syringe (Monoject Medical, St. Loius, MO)
- OHaus scale (OHous Corp., Florham Park, NJ)
- Digital timer
- Extracted single-rooted human teeth
- Standard school of dentistry endodontic set up
- Obtura II warm vertical obturation system (Obtura Spartan, Trenton, MO)
- 1/2 inch copper tubing (NIBCO, Elkhart,IN)
- Fastray (Bosworth Company, Skokie,IL)
- Instron Machine (Instron Corp., Canton, MA)
- IPANEX (IPA Systems Inc., Philadelphia, PA)
- ParaPost Drills (Coltene/Whaledent, Cuyhahoga Falls, OH)
- ParaPost XP Posts, stainless steel (Coltene/Whaledent,Cuyhahoga Falls, OH)
- ProFile ISO .06 rotary files (Dentsply, Milford, DE)
- Sure flex ISO files (Dentsply, Milford, DE)
- Roth Root Canal Cement (Type 801 Elite grads, Roth International, Chicago, IL)
- Capillary tips .014 (Ultra Dent Products Inc., South Gordon, Utah)
- IRM intermediate restorative material (Caulk Division, Milford,DE)
- Gates Glidden burs (Dentsply, Milford, DE)
- 5.25% NaOCl (The Clorax Company, Oakland, CA)

- FlexR files (Union Broach, Mayco York, PA)
- Absorbent points (Dentsply, Milford, DE)
- Glass slabs (Buffalo Dental, Sgosset, NY)
- Hy-Bond Zinc Phosphate Cement (Shofu Dental Corp., San Marcos, CA)
- Sybron Mini Endo (Sybron Dental Specialties, Glendora, CA)
- Messerann Kit (Union Broach Corp., Long Island city, NY)
- Zeiss Intraoral microscope (Carl Zeiss International, Oberkochen, Germany)
- Dental Surveyor (Ney Dental Inc., Bloomfield,CO)

Data Collection/Instrumentation

Table 1. Initial and final mean set time results per cement type

Cement	Ave. Initial set	Ave. Final set
Portland cement	24 minutes	4 hours 31 minutes
Portland cement with 10% CaCl ₂	7 minutes	46 minutes
Portland cement with 15% CaCl ₂	8minutes	47 minutes
IPANEX	30 seconds	3 minutes
PATCH 928	1 hour and 6 minutes	4 hours

Table 2. The mean strength and standard deviations in newtons for post dislodgement for all cements at 72 hours and 28 days.

Time	Cement	Mean (retentive force in newtons)
28 days	MTA	229±71
28 days	ZnPO ₄	318±116
72 hours	10% CaCl ₂	215±100
72 hours	15% CaCl ₂	150±65
72 hours	MTA	243±76
72 hours	ZnPO ₄	367±147

Table 3. Microscopic evaluation of cement dislodgement

	Both	Post from Cement	Cement from tooth
72 hours Portland cement with 10% CaCl ₂	0	4	1
72 hours Portland cement with 15% CaCl ₂	1	2	2
72 hours Zinc phosphate	1	0	4
72 hours Mineral trioxide aggregate	0	4	1
28 day Zinc phosphate	1	2	7
28 day Mineral trioxide aggregate	3	3	4

Figure 6. The mean, maximum and minimum force in Newtons for post dislodgement in the 28 day

sample group.

Figure 7. The mean , maximum and minimum force in Newtons for post dislodgement in the 72 hour group.

CHAPTER IV

RESULTS AND DISCUSSION

Set Time Results

Some areas of the molds set faster than other areas. This was seen as some areas not marking and other areas marking. Therefore, the reported set times were only an approximation used to determine what cements were to be used for the post retention aspect of the study. The use of MTA was cost prohibitive and Portland cement was used as a substitute.

Portland cement had a mean final set time of 4 hours and thirty-one minutes (Table 1). PATCH 928, a blend of Portland cement and 30-60% proprietary silicate mixture, produced an initial set time of 1 hour and 6 minutes. This, along with a grainy texture, eliminated the cement from further testing. It was found that calcium chloride and IPANEX significantly reduced the set time of Portland cement. The addition of IPANEX was found to be extremely alkaline and not suitable for the post study. Adding either 10 % or 15% calcium chloride to Portland cement resulted in an exothermic reaction. Appendix B gives the raw data for each group.

72 Hour Post Retention Results

Table 2. shows mean failure loads and standard deviations for all groups in the 72 hour group. Figure 7 depicts this. A one-way analysis of variance and a Students' t test were

used to establish the statistical significance among the means of the groups. The Students't test was used due to the large and unequal standard deviation. One way ANOVA analysis showed those posts seated with zinc phosphate to have higher resistance values than the experimental groups ($p < .05$). When the experimental cements were compared to each other, there was no significant difference in resistance to post dislodgement. Experimental samples pulled at a lower force than zinc phosphate. Appendix C gives all raw data for the 72 hour and 28 day post retention tests.

28 Day Post Retention Results

Figure 6 represents the dislodgement forces in the 28 day group. A two-way ANOVA found the force required to dislodge the posts not significant in relation to time (Appendix E.) The higher retentive forces for zinc phosphate versus the experimental forces can be seen in Figure 6 and 7.

Analyzing mean values for the dislodgement force for zinc phosphate and MTA cements gives the following: ZnPO₄ with 318.0 ± 116 newtons and MTA with 229 ± 71 newtons (Table 2.). The retention force of MTA was 28% less than zinc phosphate in the 28 day study.

Manner of Dislodgement

An optical microscope (Carl Zeiss International) was used to view the method of cement dislodgement. Samples were studied under the microscope (x40) to determine if separation occurred from the post or from tooth structure. Samples that demonstrated both dislodgement from the post and the tooth were recorded as Both. Table 3 shows the raw data. Dislodgement from the tooth with cement remaining attached to the post occurred in 73 %

for zinc phosphate and 32 % for the experimental cements. Separation of the post from the cement occurred in 13 % for zinc phosphate and 52 % for the experimental cements.

Discussion

Due to its hydrophilic nature and biocompatibility, MTA is the material of choice when a perforation occurs. The drawback associated with this material is slow set time and initial low compression strength.

Admixtures chosen for this study were those that decreased Portland cement set time. These were found through an internet search and information from various cement suppliers. The standard method for testing hydraulic cements was used. This method used larger samples which allowed for better evaluation of the mix texture and future comparison with other cement admixtures. Set time is affected by many factors; temperature, humidity, entrapped air, pH and the amount of mixing.(55) Some areas of the molds set faster than other areas. Thus, the reported set times are only an approximation. Portland cement had a final set time of four hours and 31 minutes. This was found to be similar to the reported set time of three to four hours for MTA.(62) Those admixtures which had poor texture or had the possibility of being toxic were eliminated. PATCH 928 and IPANEX fell into this category.

The addition of calcium chloride as an admixture to Portland cement is well known in industry. Calcium chloride was chosen for the post study because it decreased both initial and final set times. It can be seen from Table. 1 that the addition of either 10% or 15% calcium chloride to Portland cement decreased the initial set time from 24 minutes to eight minutes and the final set time from four hours and 30 minutes to 47 minutes. The addition of this salt produced an exothermic reaction. 15% calcium chloride when compared to 10% calcium

chloride had a faster rate of heat production. In general, the addition of calcium chloride decreased the setting time by more than 50%.

Zinc phosphate was chosen as the standard because it has long been the cement to which other cements are compared. Zinc phosphate has a long history of *in vivo* success.(63-65) The white formula of MTA was chosen over the grey formula because it is reported to develop a quicker and higher final compressive strength.(66) Para Post XP stainless steel posts were chosen because they are serrated and parallel-sided. This has been shown to increase resistance to dislodgement.(67) Abrasive blasting of posts was performed because this has also been shown to increase retention.(68)

The cementation methodology used employed both finger pressure and ultrasonic vibration. Recently Vargas et al. compared the retentive strength of MTA to that of zinc phosphate and glass-ionomer cements at 72 hours. Vargas used posts seated with finger pressure only. The post were also seated to a 9mm post space. It has been speculated that the use of ultrasonic vibration would allow for easier flow and a more uniform cement layer.(69) This could explain the increased retentive force for MTA in the present study when compared to that found by Vargas et al. The present study produced a mean retentive force of 243 ± 79 newtons in 72 days for MTA while Vargas et al produced a mean force of 182 ± 121 newtons.

The stabilization assembly made to ensure that the posts were all parallel was similar to that employed by Schwartz.(70) The present investigation, however, used hollow trepans from a Masserann kit (Union Broach Corp.) for post stabilization. Once stabilized, the Instron Universal Testing machine tested the tensile force in an axial direction. The retention force for each sample was recorded and charted by the machine. This method employed

examination of retention from a direct pull only. However, the information obtained was sufficient to establish comparative values.

At 72 hours, zinc phosphate was shown to be significantly more retentive than the experimental cements. Appendix D gives the statistical examination. Because of this, and the fact that there was no significant difference between groups, only MTA was chosen for the 28 day study. 28 days was chosen because this is the time reported for maximum strength of Portland cement.(60) In the 28 day group, zinc phosphate remained more retentive when compared to MTA. A two-way ANOVA showed significance between cements but not time.(Appendix D) Therefore, there was no significant increase in retention when comparing the 72 hour samples with the 28 day samples for either MTA or zinc phosphate. The hardening process of MTA is one of hydration, in which water actually participates in the reaction. Water may not have reached the entire cement depth due to the presence of the post and the depth of the prep. The depth of set was not determined in the present study. Sluyk et al. found a significant difference in resistance to displacement between the 24 hour sample and the 72 hour sample. The 72 hour sample resisted displacement significantly more than the 24 hour sample.(71) This was a punch out study with a shallow preparation and may not be comparable to the present study. Appendix D and E gives the statistical examination and Appendix C gives raw data for both the 72 hour and 28 day groups.

When evaluating the method of post dislodgement, zinc phosphate cement was more likely to dislodge from the root surface rather than the post. See Table 3. Colley et al. confirms this result. He reports that zinc phosphate was “sticky” before it set but that it was not adhesive to dentine after setting. It was the “lock” that caused adhesion.(72) Conversely, MTA was more likely to dislodge at the post-cement interface with cement remaining along

the dentine wall. Sluyk et al. found similar results when testing the retention of MTA as a furcation repair material.(73)

There is no material that is used for both repairing a perforation and seating a post. Furthermore, exact cement strength needed to retain a post is unknown. However, if a tooth is perforated in an area that would be hard to reach, vibrating MTA down the canal with a post could be an option. Sufficient tooth structure would be needed for restorative reasons. Also, the post would need to be stabilized during setting.

CHAPTER V

SUMMARY AND CONCLUSIONS

Summary

A post is required for the retention when significant coronal tooth structure is missing. Post placement can lead to perforations which can compromise long term prognosis. Mineral trioxide aggregate is a new material with successful perforation repair properties but which requires a long setting time. The purpose of this study was two fold. First, was to determine the set time decrease with various admixtures. The second aspect of this study was to evaluate the use of accelerated Portland cement and MTA as cementing agents for posts.

Portland cement without the addition of an accelerating agent was found to have a similar set time to MTA. This was from three to four hours. It can be seen from Table 1. that the addition of calcium chloride, either 10% or 15% by weight, to Portland cement reduce the set time. Initial set time was decreased from approximately 24 minutes to eight minutes. Final set time was reduced from approximately four hours and 30 minutes to 47 minutes. From the current study it can be speculated that the addition of calcium chloride to MTA would decrease set time. The current practice to place a moist pellet over MTA for 72 hours to allow for set could possibly be eliminated with an accelerated form of MTA. Further investigation is needed.

Conclusions

While the addition of 10% and 15% calcium chloride decreased the set time of Portland cement, it did not increase resistance to post dislodgement. The present study found the experimental cements; MTA, Portland cement with 10% calcium chloride, Portland cement with 15% calcium chloride, to be less retentive when compared to zinc phosphate. MTA was found to be 28% weaker than zinc phosphate.

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APPENDIX A
IRB EXEMPTION FORM

Application for Exemption

Approval from the IRB staff must be received prior to beginning the research described below. Please **type** all responses and submit this form with original signatures. All investigators must complete Ethics Training before an approval will be granted.

1. Title of study: The use of mineral trioxide aggregate and accelerated Portland cement as cements for posts.

2. Investigators (list all investigators, principal investigator first; attach additional sheets if necessary):

Name	Signature	Dept/College	Address	Tel No
Pamela Harrington	<i>Pamela Harrington</i>	Endodontics/School of Dentistry	R.C. Byrd HSC	304 293-0627
M. Richards	<i>M. Richards</i>	Orthodontics	PO BOX 9450	
M. Bagby	<i>M. Bagby</i>	Bio Materials	Morgantown, WV	
Name or initials			Ethics Training ¹	HIPAA Training ²
<i>(PH)</i>		YES if training has been completed	yes	yes
<i>M.B.</i>		¹ Ethics training: http://www.wvu.edu/~rc/irb/ethi_tra.htm	yes	yes
<i>M. Richards</i>		² HIPAA info: http://www.wvu.edu/~rc/irb/index.htm	yes	yes

3. Estimated period of project or of human subject involvement: Starting date: 09-15-04 Ending date: 09-15-05

4. Reason for conducting research ☐ Professional ☐ Dissertation ☒ Thesis
☐ Class Assignment ☐ Other specify

5. Source of funding (if applicable) WVU School of Dentistry Seed Money Grants

6. Number of projected subjects 50-100 Number of projected records or data files 50-100

7. This research involves (check all that apply—see attached "Exempt Research" page):

- ☒ a Collection or study of existing data, documents, records or specimens, recorded without identifiers
☐ b Normal educational practices conducted in established or commonly accepted educational settings
☐ c Educational tests (cognitive, diagnostic, aptitude, achievement)
☐ d Observation of public behavior
☐ e Surveys, interviews or hand-outs for subjects over 18
 (use age ranges, not actual age, for demographic information):
☐ Mail ☐ Telephone ☐ Person-to-person
☐ f Any possibility of identifying a subject (discuss in cover letter)
☐ g The possibility that the subject's responses or conduct (if they became public) may place the subject at risk of criminal or civil liability or be damaging to the subject's financial standing or employability
☐ h Sensitive aspects of personal behavior (for example: illegal conduct, drug use, sexual behavior or use of alcohol)
☐ i Investigator's participation in activities being observed
☐ j Only surveys or interviews of elected or appointed public officials or candidates for public office
☐ k Audiotaping
☐ l Children under age 18 (see Chapter II of the Guidelines)
 Note: Interviews and surveys with children are never exempt.
☐ m Food tasting and evaluation
☐ n Research and demonstration projects
☐ o Access to protected health information (PHI) (See HIPAA requirements: <http://www.wvu.edu/~rc/irb/index.htm>)

8. Goal of research
The goal of this research is to evaluate the use of mineral trioxide aggregate and accelerated Portland cement as cements for posts.

9. Explanation of procedures involved in research

Portland cement will be accelerated using different admixtures. The set of these cements will be evaluated. Endodontic therapy will be conducted on extracted teeth from a jar containing previously extracted teeth with no identifiers. Post spaces will be prepared and posts will be seated with the different cements. The posts will then be subjected to dislodgement.

10. Explanation of known risks to human subjects

NA

11. Explanation of how records will be kept

The set times of the different cements will be recorded. The amount of force needed to dislodge the posts will be recorded. A paired t test will be used for statistical treatment. Records will be kept in a locked box.

A cover letter addressed to respondents must accompany any survey or questionnaire. The cover letter **must** be on *the investigator's WVU departmental letterhead* and must include the following:

- 1 a statement that the project is research being conducted in partial fulfillment of the requirements for a course, master's thesis, dissertation, etc.
- 2 purpose of study
- 3 a statement that subjects' responses will be kept anonymous or confidential (explain extent of confidentiality if subjects' names are requested)
- 4 if audiotaping, a statement that subject is being audiotaped (explain how tapes will be stored or disposed of during and after the study)
- 5 a statement that subjects do not have to answer every question
- 6 a statement that subject's class standing, grades, or job status (or status on an athletic team, if applicable) will not be affected by refusal to participate or by withdrawal from the study
- 7 a statement that participation is voluntary

Attachments:

- ☐ questionnaire/survey to be used
- ☐ telephone text (including introductory remarks as in a cover letter—see above)
- ☐ cover letter
- ☐ permission from external institution, on their letterhead (if applicable)

I have reviewed the above information and recommend this study for exemption.

Dean or Director 9-15-09

Dean or Director

Department Chair

Department Chair

Faculty Advisor

APPENDIX B
SET TIME DATA

CEMENT MIXTURES FOR SET TIME

CEMENT	DRY	LIQUID
Portland Cement (P.C.)	50 g P.C.	15 ml H ₂ O
P.C. +10% CaCl ₂	45g P.C.+ 5g CaCl ₂	15 ml H ₂ O
P.C.+15% CaCl ₂	42g P.C.+8g CaCl ₂	15 ml H ₂ O
IPANEX*	50g P.C.	50 ml IPANEX
PATCH**	50g PATCH	10 ml H ₂ O

* Material was extremely alkaline and clumpy

**Material was grainy and contained silicates.

Portland cement with 10% Calcium Chloride

Initial set	Final set
8 minutes	47 minutes
6 minutes	42 minutes
8 minutes	45 minutes
6 minutes	51 minutes

Portland cement with 15% Calcium Chloride

Initial set	Final set
8 minutes	55 minutes
8 minutes	43 minutes
7 minutes	40 minutes
8 minutes	50 minutes

Portland cement

Initial set	Final set
25 minutes	4 hours 35 minutes
26 minutes	4 hours 52 minutes
15 minutes	4 hours
30 minutes	3 hours 50 minutes

APPENDIX C

RETENTION DATA IN NEWTONS FOR THE 72 HOUR AND

28 DAY GROUPS

Data for the 28 day group

ZnPO ₄	MTA
254.53	350.14
241.02	135.32
179.09	236.42
503.01	265.87
456.19	162.63
212.79	162.93
293.25	179.84
382.46	223.66
224.90	248.22
432.88	321.29

Data for the 72 hour group

MTA 10%CaCl₂ 15% CaCl₂ ZnPO₄

352.29	239.91	161.36	228.77
249.65	271.34	74.31	567.23
137.92	342.70	195.88	340.08
249.65	118.79	226.40	650.40
221.39	111.21	93.41	462.56

APPENDIX D

ONE WAY ANALYSIS FOR 72 HOUR GROUP

Oneway Analysis of Column 2 By Column 1

Tests that the Variances are Equal

Level	Count	Std Dev	MeanAbsDif to Mean	MeanAbsDif to Median
MTA	5	79.2448	51.1400	49.9260
MTA10	5	100.1433	81.4320	83.0940
MTA15	5	65.1959	53.1296	57.8160
Zinc	6	175.2807	146.0933	146.0933

Test	F Ratio	DFNum	DFDen	Prob > F
O'Brien[.5]	3.2712	3	17	0.0468
Brown-Forsythe	3.5940	3	17	0.0354
Levene	3.9711	3	17	0.0258
Bartlett	1.5849	3	.	0.1907

Warning: Small sample sizes. Use Caution.

Welch Anova testing Means Equal, allowing Std Devs Not Equal

F Ratio	DFNum	DFDen	Prob > F
3.8601	3	9.2922	0.0485

Oneway Analysis of Column 2 By Column 1

Oneway Anova

Summary of Fit

Rsquare	0.47436
Adj Rsquare	0.3816
Root Mean Square Error	117.7865
Mean of Response	263.6681
Observations (or Sum Wgts)	21

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Column 1	3	212842.78	70947.6	5.1138	0.0105
Error	17	235852.23	13873.7		
C. Total	20	448695.02			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
MTA	5	243.580	52.676	132.44	354.72
MTA10	5	216.790	52.676	105.65	327.93
MTA15	5	150.272	52.676	39.14	261.41
Zinc	6	413.970	48.086	312.52	515.42

Std Error uses a pooled estimate of error variance

Oneway Analysis of Column 2 By Column 1

Means Comparisons

Comparisons for each pair using Student's t

	t	Alpha
	2.10982	0.05
Abs(Dif)-LSD		
	Zinc	MTA MTA10 MTA15
Zinc	-143.48	19.91 46.70 113.22
MTA	19.91	-157.17 -130.38 -63.86
MTA10	46.70	-130.38 -157.17 -90.65
MTA15	113.22	-63.86 -90.65 -157.17

Positive values show pairs of means that are significantly different.

Level	Mean
Zinc A	413.97000
MTA B	243.58000
MTA10 B	216.79000
MTA15 B	150.27200

Levels not connected by same letter are significantly different

Level	- Level	Difference	Lower CL	Upper CL	p-Value
Zinc	MTA15	263.6980	113.219	414.1769	0.0017886
Zinc	MTA10	197.1800	46.701	347.6589	0.0132559
Zinc	MTA	170.3900	19.911	320.8689	0.0287599
MTA	MTA15	93.3080	-63.862	250.4781	0.2273286
MTA10	MTA15	66.5180	-90.652	223.6881	0.3843649
MTA	MTA10	26.7900	-130.380	183.9601	0.7235577

APPENDIX E

TWO WAY ANALYSIS FOR 72 HOUR AND 28 DAY GROUPS

Effect Test

Sum of Squares	F Ratio	DF	Prob>F
6745.8407	0.6334	1	0.4333

Least Squares Means

Level	Least Sq Mean	Std Error	Mean
28 Days	273.3220000	23.07596668	273.322
72 Hours	305.1320000	32.63434504	305.132

Effect Test

Sum of Squares	F Ratio	DF	Prob>F
1895.5136	0.1780	1	0.6766

Least Squares Means

Level	Least Sq Mean	Std Error
MTA, 28 Days	228.6320000	32.63434504
MTA, 72 Hours	243.5800000	46.15193336
ZnPO4, 28 Days	318.0120000	32.63434504
ZnPO4, 72 Hours	366.6840000	46.15193336

Whole-Model Test

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	3	84576.25	28192.1	2.6471
Error	26	276900.12	10650.0	Prob>F
C Total	29	361476.37		0.0701

Cement

Effect Test

Sum of Squares	F Ratio	DF	Prob>F
75249.084	7.0656	1	0.0133

Least Squares Means

Level	Least Sq Mean	Std Error	Mean
MTA	236.1060000	28.26217184	233.615
ZnPO4	342.3480000	28.26217184	334.236

Response: Strength

Summary of Fit

RSquare	0.233974
RSquare Adj	0.145587
Root Mean Square Error	103.1989
Mean of Response	283.9253
Observations (or Sum Wgts)	30

Parameter Estimates

Effect Test

Source	Nparm	DF	Sum of Squares	F Ratio	Prob>F
Cement	1	1	75249.084	7.0656	0.0133
Time	1	1	6745.841	0.6334	0.4333
Cement*Time	1	1	1895.514	0.1780	0.6766

CURRICULUM VITAE

Pamela P Harrington DDS, MS

Residency

2003-2005

MS Endodontics

West Virginia University

Morgantown, WV

Education

1987-1991

DDS Dentistry

University of Minnesota School of Dentistry

Minneapolis, MN

1980-1985

University of Minnesota Duluth, MN

Work experience

1993-2003

Staff Dentist

VA Medical Center

Sioux Falls, SD

Memberships

2003-present Professional Conduct and Ethics Committee American Association of Endodontists

1999-2003 Disciplinary Appeals Board Examiner, Department of Veterans

Publications

Shared Responsibility for Oral Health in Long-Term Care Facilities, Special Care in Dentistry, Sept.-Oct., 1990

Awards

Chapter Appreciation Award, Disabled American Veterans