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This is to certify that the thesis prepared by Paul D. Clark entitled AN INVITRO
LEAKAGE AND VISCOSITY ANALYSIS OF ROOT END FILLING MATERIALS
has been approved by his committee as satisfactory completion of the thesis requirement
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AN INVITRO LEAKAGE AND VISCOSITY ANALYSIS OF ROOT END FILLING
MATERIALS

A thesis submitted in partial fulfillment of the requirements for the degree of Master of
Science in Dentistry at Virginia Commonwealth University.

by

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Abstract

AN INVITRO LEAKAGE AND VISCOSITY ANALYSIS OF ROOT END FILLING MATERIALS

By Paul D. Clark, D.D.S.

A Thesis submitted in partial fulfillment of the requirements for the degree of Masters of
Science in Dentistry at Virginia Commonwealth University.

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The purpose of this study was to determine if the viscosity of MTA Angelus Fluid is lower than that of Pro Root MTA and MTA Angelus; and to compare the viscosity, leakage and particle size of these materials to determine whether a relationship exists between these properties. The viscosity of each material was measured and compared with the Student's t test. MTA Angelus Fluid's viscosity was significantly lower than the other two materials tested. Microleakage of root end fillings was assessed in a passive diffusion model. Leakage groups were compared with a one-way ANOVA ($p < 0.05$). No significant

difference was found. Particle size and shape were evaluated with the SEM. MTA Angelus Fluid has a lower viscosity than the other materials tested. There was no significant difference in the sealing ability of the three materials tested and there was no apparent variation in each material's particle size or shape.

Introduction

Mineral trioxide aggregate (MTA) is widely used in dentistry for root end fillings, perforation repairs, and pulp capping. It has superior biocompatibility when compared to the traditional materials used in root end filling and repair (1). It has a long setting time (2) and difficult handling characteristics (3, 4). It was first marketed as ProRoot MTA[®] by Dentsply Tulsa Dental (Tulsa, OK).

The Brazilian company, Angelus Dental Solutions,(Odonto-Logika, Ind. Prod. Odont. Ltda, Londrina, Parana, Brazil) selected a Portland cement and added bismuth oxide to provide radiopacity similar to that of ProRoot MTA. This product is commercially available as MTA-Angelus[®] (5).

Recognizing the advantages and disadvantages of MTA and Portland cement, researchers in Singapore embarked on a project to create a root repair material that combines the superior biocompatibility of MTA with the handling characteristics of materials such as Super EBA. They speculated that increasing the viscosity would enhance the sealing ability and make handling easier. This new highly viscous material is known as Viscosity Enhanced Root Repair Material (VERRM) (6).

VERRM was formulated using Portland cement as the base material. Bismuth oxide and other compounds were added to increase the radiopacity and viscosity. In 2005,

Chng et al. found no significant difference in the physical and sealing properties of VERRM and white MTA. (6).

It is possible that by reducing the viscosity of MTA it may exhibit better handling characteristics allowing for better dentinal adaptation and decreased leakage. MTA Angelus Fluid, an endodontic root-end filling material newly developed by Angelus, reportedly possesses a lower viscosity which we hypothesize may improve the handling properties of the material, allow for better adaptation to the dentinal walls and ultimately decrease marginal leakage.

The purpose of this study was to determine if the viscosity of MTA Angelus Fluid is lower than that of Pro Root MTA and MTA Angelus; and to compare the viscosity, leakage and particle size of these materials to determine whether a relationship exists between these properties.

Materials and Methods

Viscosity Analysis

The viscosity of MTA Angelus, MTA Angelus Fluid, and Pro Root MTA was tested with a new technique developed at Virginia Commonwealth University. Each material tested was mixed according to manufacturer's instructions. After mixing, the material was gathered and condensed into the plastic scoop provided with the MTA Angelus to obtain a standardized sample size and shape (approximately 0.15 g) for each of the three root end filling materials tested. The material was then removed from the scoop and placed on a glass slab and a digital photograph was taken. A second glass slab was then placed on top of the material sample along with a twenty pound weight which rested in place for three minutes, assisting the flow of the material. When the three minute time period had elapsed, the weight and the second glass slab were removed and a final digital photograph was taken. Ten samples were prepared and tested for each material. The digital photographs taken of each material sample were evaluated with the UTHSCSA image tool[®] software program. The measurement tool in this program was calibrated and used to trace the initial and final areas of each material sample. The ruler in the photograph allowed for sample size calibration. The difference between the initial and final areas was interpreted as a quantitative representation of the material's flow capability. Because viscosity describes a fluid's internal resistance to flow, any alteration in viscosity

will also change the material's ability to flow. Area measurements were made to the nearest 0.01 mm. The mean final area between the three material groups was compared using ANCOVA, where the initial areas were used as covariates.

Leakage Analysis

A passive diffusion model with a non-buffered 1% methylene blue solution was used to evaluate the microleakage of the different root-end filling materials. Thirty four extracted human teeth were collected and stored in saline. The crowns were removed at the cementoenamel junction with a diamond saw. The teeth were instrumented with K-type hand files, Gates-Glidden burs 2-4, ProTaper files (Dentsply Tulsa Dental, Tulsa, OK) S1, S2, and F1, and NiTi rotary ProFiles (Dentsply Tulsa Dental, Tulsa, OK) to size 40/.06 taper 1mm from the apical foramen. Each tooth was instrumented with RC Prep and irrigated with a total volume of 20 ml 5.25% NaOCl and 20 ml 17% EDTA used alternately after each file. Canals were dried with paper points and obturated with gutta percha and AH-Plus root canal sealer using the continuous wave obturation technique. The access cavities were sealed with Vitrebond and the teeth were stored in a humidior for two weeks, allowing the sealer to set. Root ends were resected at 90 degrees to the long axis of the tooth 3 mm from the end of the root. Root end cavities of 4 mm were prepared with a #2 round bur in a slow speed handpiece. Once prepared, the teeth were randomly divided into 3 groups of 10. Root end fillings were performed with MTA Angelus, MTA Angelus Fluid, or Pro Root MTA. Root end filling materials were prepared according to the manufacturer's instructions. Two obturated teeth with retro-preparations received no retro-filling and served as positive controls. Two additional teeth with no root-end

resection were instrumented, obturated, and completely covered with nail varnish. These teeth served as negative controls. The teeth were then stored in a humidifier for 24 hours, allowing the root-end fillings to set. Next, two coats of nail polish were applied to the whole surface of each root except for the resected root surface. Then, the teeth were placed in 1% methylene blue at 37 degrees Celsius for 72 hours. The teeth were then decalcified in 5% nitric acid for 48 hours and dehydrated with 80% ethyl alcohol for 24 h, 90% ethyl alcohol for 24 h and 100% ethyl alcohol for 24 h. The teeth were submerged in methyl salicylate until transparent.

Digital photographs were made of each tooth sample and the samples were evaluated for leakage using CliniView software's (GE Healthcare; Tuusula, Finland) measurement tool. A ruler was included in the photograph allowing for length calibration and leakage measurements to the nearest 0.01 mm were obtained (Figure 5). Sample groups were compared with a one-way ANOVA (at $\alpha = .05$).

Particle Analysis

The particle size and shape of MTA Angelus, MTA Angelus Fluid, and Pro Root MTA were evaluated with the SEM. Individual samples were prepared for SEM analysis. Several areas of each material sample were visually examined under the SEM looking for variations in particle size, shape, and other properties and characteristics which may affect viscosity and flow properties. Areas were selected which were representative of each material and photographs were taken.

Results

In the viscosity evaluation, the average areas for the samples within each material group are shown in Table 1. Over all 15 samples, the mean initial value was 53.4 mm² (SD = 3.29).

An analysis of covariance was used to compare the three group means at the average initial area. There was a significant relationship between the initial and final areas (p-value = 0.0319, see Figure 1) and the groups were significantly different [F (2, 11) = 4.47, p-value = 0.0494]. The Student's t-test found the viscosity of MTA Angelus Fluid significantly lower than the other two materials. The adjusted final areas are shown in Table 2.

Leakage results were analyzed with a one-way ANOVA (at alpha < .05) and are shown in Table 3. The groups were not found to be significantly different [F (2, 27) = 1.97, p = 0.1597].

Lastly, no variations in particle size, shape, or other properties and characteristics which may affect viscosity and flow properties were visually apparent in the material samples examined under the SEM (Figures 2, 3, and 4).

Discussion

This study showed that MTA Angelus Fluid seals as well as both MTA Angelus and ProRoot MTA. However, MTA Angelus Fluid was shown to have more fluid handling characteristics and a lower viscosity in our testing making it easier to use.

In an effort to explain this lower viscosity, multiple areas of each sample were microscopically examined looking for variations in particle size, shape, and other properties and characteristics which may affect viscosity and flow properties. No significant differences were visually apparent in the materials. Angelus (Angelus, Londrina, PR, Brazil) was contacted and stated that proprietary plasticizers were added to the formula in order to improve its flow characteristics. (7)

Plasticizers are chemical admixtures that can be added to concrete mixtures to improve workability. In order to produce stronger concrete, less water is added, which makes the concrete mixture very unworkable and difficult to mix, necessitating the use of plasticizers and superplasticizers. (8) Recently, the work of Bortoluzzi et al. (9) showed the addition of CaCl_2 to MTA enhanced the physicochemical properties of the material, required less water during mixing, and made it easier to handle clinically.

Moon et al (10) discuss flowable resin composites, their recommendations for clinical use, and their various formulations and viscosities. His study compared the variation in viscosity of flowable resin composites using the ADA Flow Test. Because the physical properties of MTA are different than those of resin composites, the ADA flow test

could not be used to assess the viscosity of those materials tested in this study. Therefore the methodology for testing viscosity as desired herein was developed.

Dye penetration, which is the most common technique used to assess the quality of the apical seal of root end filling materials and to determine internal tooth morphology, was used in this study. Other methods include radioisotope labeling, fluid filtration, bacterial leakage, and the electrochemical method. Because none of the methods for evaluating apical leakage reproduces all the complex mechanisms that lead to a periapical infection, all of these techniques have value and limitations (11). In a series of pilot studies conducted in conjunction with this research study, consistent leakage results were obtained with methylene blue; while inconsistent results were achieved with India ink. While it is true that Wu & Wesselink (12) say that dye penetration, particularly with small dye particles such as methylene blue, overstates leakage, it is also true that bacteria can live in dentinal tubules for some time after obturation (13) and may only require a source of nutrients such as simple sugars that are also very small to survive.

Camps and Pashley (14) stated that evaluation with dye penetration and sectioning relies on randomly sectioning the root into two pieces without knowing if the section is representative of the greatest extent of dye penetration. Various sectioning techniques have been used to determine the extent of the dye penetration, but a non-destructive technique such as chemical clearing offers the most accurate 3-dimensional method of assessing the extent of dye leakage. Understanding the extent to which a root-end filling material approaches the ideal “hermetic sealing” of the root canal is important because

clinical success rates may be improved for materials demonstrating superior resistance to leakage.

Conclusion

In conclusion, MTA Angelus Fluid has a lower viscosity than the two other materials tested. There was no significant difference in the sealing ability of the three materials tested in this study and there was no variation visually apparent in the material's particle size or shape under the SEM. There is limited information available relating MTA's viscosity and sealing ability. Further development and study of MTA is indicated to develop a biocompatible root end filling material with superior handling characteristics and excellent sealing properties.

Table 1: Average Initial and Final areas of Viscosity Samples

Description of samples					
Group	n	Mean	SD	95% CI	
Initial area (mm2)					
1 MTA A	5	53.92	2.39	50.95	56.88
2 MTA F	5	53.16	3.16	49.23	57.09
3 MTA	5	53.23	4.65	47.45	59.00
Final area (mm2)					
1 MTA A	5	183.26	23.65	153.89	212.63
2 MTA F	5	210.66	24.16	180.66	240.66
3 MTA	5	176.10	27.84	141.54	210.67

Table 2: Adjusted Final Areas of Viscosity Samples

Adjusted (LS) means				
Group	LS Mean	SE	95% CI	
1 MTA A	181.20	9.53	160.23	202.17
2 MTA F	211.83	9.50	190.92	232.75
3 MTA	176.99	9.50	156.09	197.90

Table 3: Leakage Results of Root End Filling Materials

Materials	n	Average Leakage (mm)		
		Mean	SD	
MTA (Angeles)	10	3.09	0.79	
MTA (fluid)	10	2.46	0.85	0.7284963904
MTA (std)	10	3.45	1.59	2.5359925504
neg	2	0.00	0.00	1.277593234
pos	2	12.09	0.32	

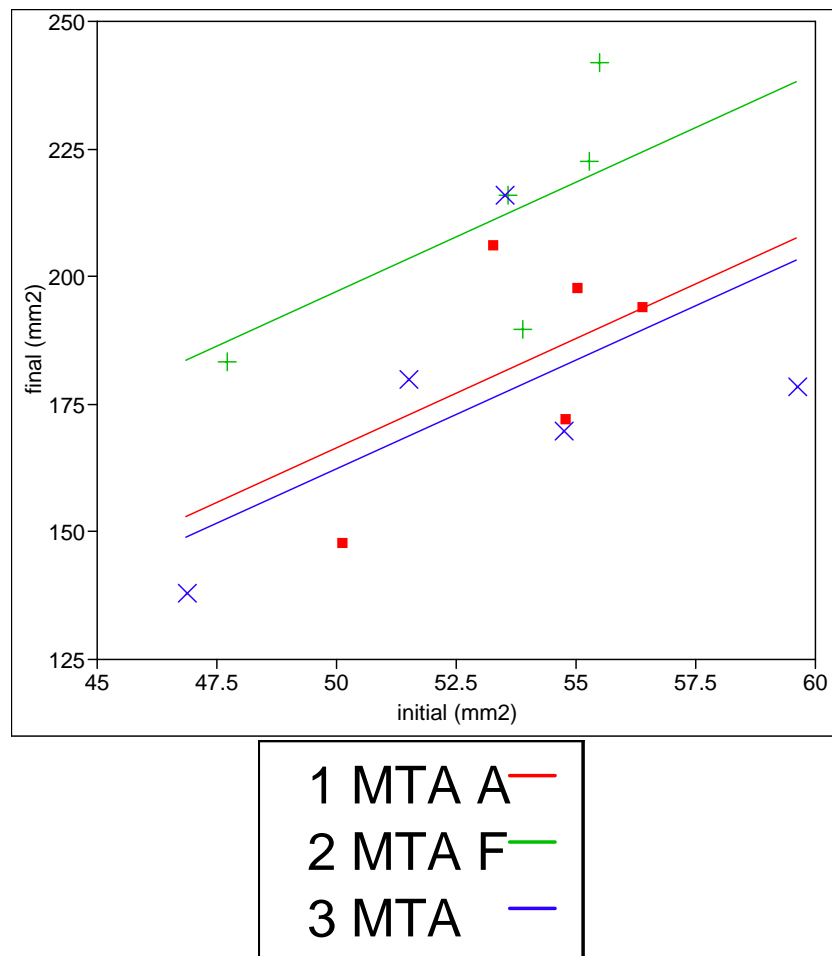
Figure 1: Initial and Final Area Comparison of Viscosity Samples

Figure 2: SEM photograph of ProRoot MTA

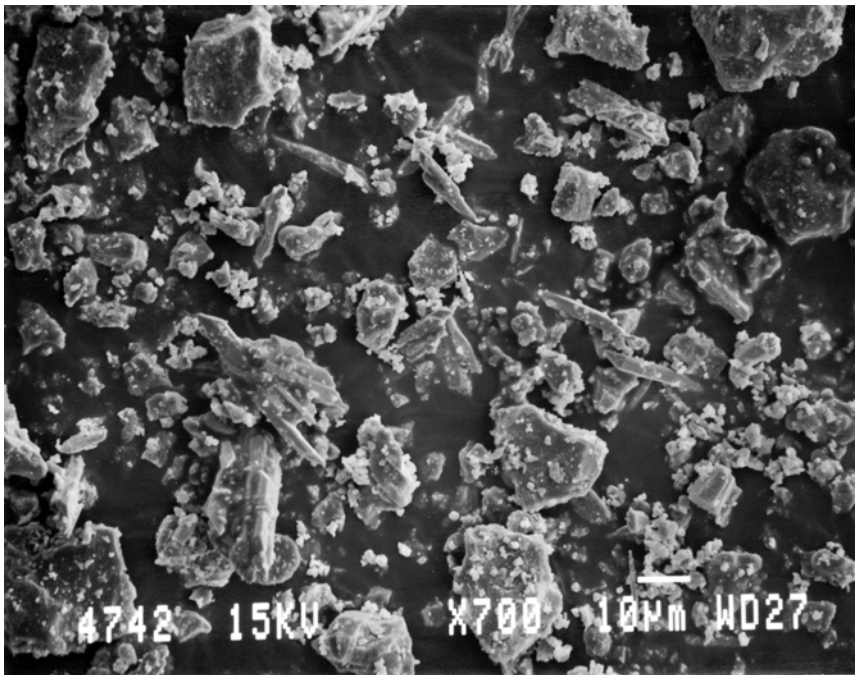


Figure 3: SEM photograph of MTA Angelus

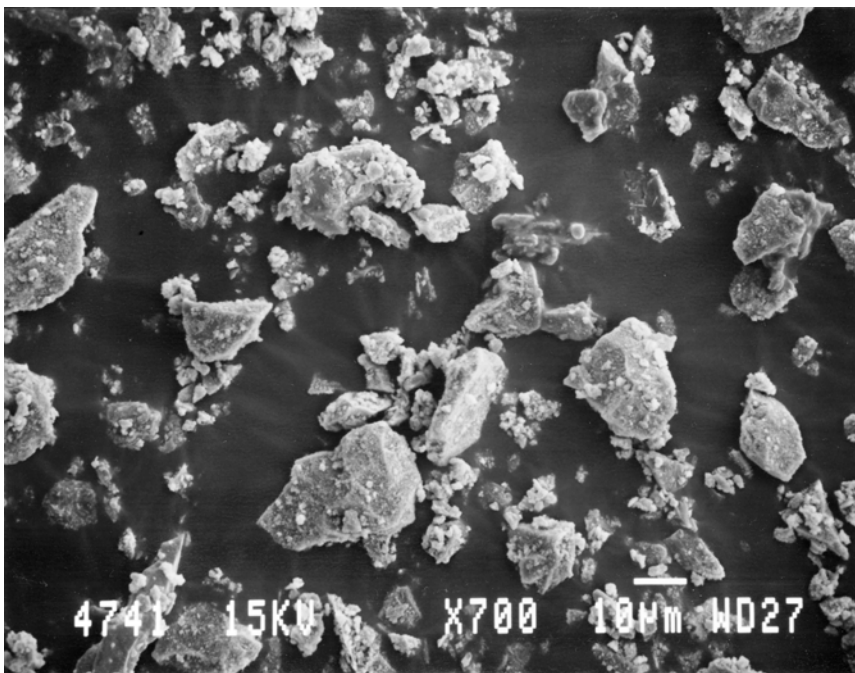


Figure 4: SEM photograph of MTA Angelus Fluid

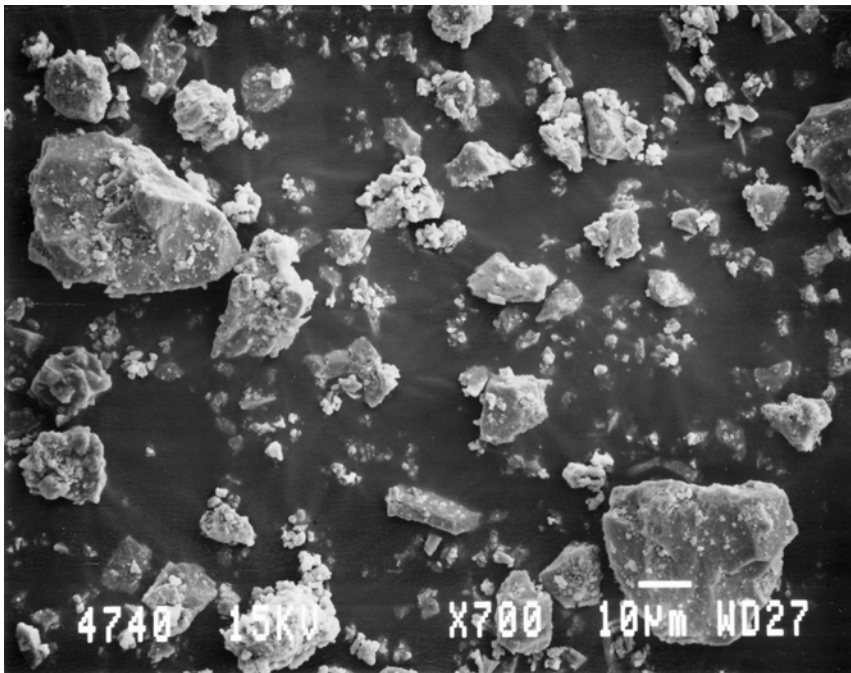
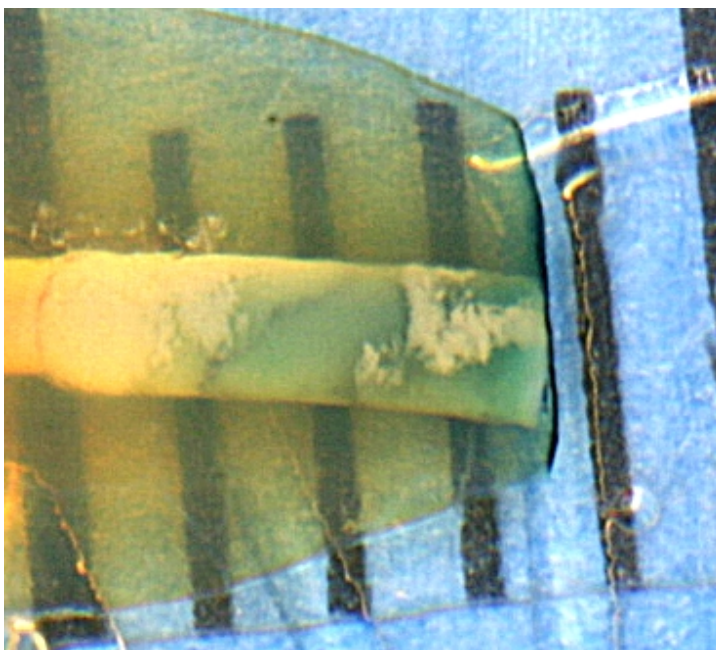


Figure 5: Root End Filling Dye Leakage Sample



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