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The Influence of Residual Calcium Hydroxide Intracanal Medicaments On The Bond Strength Of A Bioceramic

Sealer: In Vitro

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Abstract

The aim of the study was to compare and evaluate the influence of residual calcium hydroxide intracanal medicaments on the bond strength of an epoxy resin based sealer, AH Plus[®] (Dentsply Maillefer, Ballaigues) and a bioceramic sealer, SmartPasteBio (Smart-seal, DRFP Ltd., Stamford, UK).

Methodology: The root canals of 60 freshly extracted human single rooted mandibular premolars were prepared with the ProTaper System (Dentsply Maillefer, Ballaigues, Switzerland) up to a master apical file size of F3. Canals were obturated with AH Plus or SmartPaste Bio sealer using the single-cone technique either immediately (control group, n = 20) or after a 7-day intracanal calcium hydroxide [Ca(OH)₂] medicaments placement (Group 2 [Ca(OH)₂+distilled water]; group 3 [Ca(OH)₂+2%CHX]). In both groups, Ca(OH)₂ medicament removal was performed manually using Protaper F3 followed by passive ultrasonic irrigation with 2.5% NaOC1. A final flush of 17% EDTA was done followed by distilled water rinse. Post obturation specimens were stored for 30 days period before subjecting to push out bond strength testing. A 2-mm-thick middle section of each root was subjected to push-out testing and the maximum loads at failure were recorded in MPa.

Results: SmartPaste Bio showed similar bond strength to AH Plus in the control group (P >.05). In group 2, SmartPaste Bio showed higher bond strength values. While in Group 3, both the sealers showed similar bond strengths (P>.05).

Conclusion: When calcium hydroxide is used as a medicament, bioceramic sealer may be preferred over epoxy resin based sealer in terms of bond strength. However, with prior placement of combination of calcium hydroxide and 2% chlorhexidine, both sealers performed similarly with respect to dislodgement resistance.

Keywords: Calcium hydroxide, Chlorhexidine, AH Plus, Smart Paste Bio, Push out bond strength

Introduction

Over the past 70 to 80 years attempts have been made to improve on the nature of root canal obturation using newer materials and techniques to obtain a microbiologic barrier within the confines of a root canal system.¹ Although the gutta percha and endodontic sealer combination does not provide a complete bacteria-tight seal, this approach has remained the most widely accepted method of obturating the root canal system.² Ideally the sealer produces a bond between dentinal wall and core material.

Epoxy resin based sealer such as AH Plus (Dentsply International/ Maillefer) is widely used and has shown higher bond strengths to dentin than other sealers.^{3,4} Recently, Bioceramic based sealers have also shown good bonding to root canal dentin even under various conditions of dentin moisture; with added advantages of sealability, cvtocompatibilty.³⁻⁶ antibacterial activity and SmartpasteBio (Smart-seal, DRFP Ltd., Stamford, UK) is a bioceramic based sealer, described by the manufacturer as a premixed, injectable, hydrophillic cement paste, which may be used as a root filling material or sealer. The cement absorbs water from within the canal to initiate and complete its setting reaction and once set produces a radiopaque biocompatible cement. It produces calcium hydroxide and hydroxyapatite as by-products of the setting reaction.^{3,5-8}

Intracanal medicaments have been recommended during endodontic therapy to improve disinfection.⁹ Calcium hydroxide intracanal medicament has a wide range of antimicrobial activity against endodontic pathogens. Recently, Chlorhexidine has also been used as an intracanal dressing between the appointments in the gel form or as a mixture with calcium hydroxide.¹⁰ The combinations of calcium hydroxide and chlorhexidine show antimicrobial activity against obligate anaerobes, augmenting the antibacterial effect of either medicament on certain species.^{11,12}

Several methods have been employed to remove intracanal calcium hydroxide in an attempt to completely remove the medicament from the canals, however, none, yet, have been successful.¹³⁻¹⁸ Studies on interaction of residual calcium hydroxide with permanent filling materials with possible apical leakage of the obturated root canal system are reported.^{19,20} However, residual calcium hydroxide has shown a positive effect or did not affect bonding and sealing of endodontic materials in other investigations.²¹⁻²⁶ There is limited information available on the effect of calcium hydroxide intracanal medications on the bonding properties of bioceramic sealers.²⁴ Hence, the purpose of this study was to evaluate and compare the effect of residual calcium hydroxide medicaments on the bond strength of a bioceramic sealer with an epoxy resin based sealer.

Materials & Methods

Specimen Preparation

Sixty single rooted human mandibular premolars of similar sizes were selected. Conventional access cavities were prepared. The working length was set to the length 1mm shorter than that of #10 K-file inserted upto the apical foramen of the root canal. Instrumentation of all canals were carried out using a crown down technique with ProTaper Rotary System (Dentsply Maillefer, Ballaigues, Switzerland) from S1 through F3 (master apical file). Conventional syringe irrigation was done using 2mL 2.5% sodium hypochlorite (NaOCl) between each rotary file. After completion of instrumentation, irrigation was performed with 5mL 2.5% NaOCl and a final flush with 5ml of 17% Ethylenediamine tetra-acetic acid (EDTA) for 1 minute was done to remove the smear layer. The prepared canals were rinsed with 5ml of distilled water and dried with absorbent paper points.

The teeth were randomly assigned to 3 groups (n=20) as follows:

1. **Group 1 (The control group):** no prior placement of calcium hydroxide [Ca(OH)₂] intracanal medicament before obturation.

2. Group 2 (Ca(OH)₂+distilled water): A paste of

calcium hydroxide was obtained by mixing $Ca(OH)_2$ powder with distilled water (1g $Ca(OH)_2$ powder to 1mL of distilled water).

3. Group 3 (Ca(OH)₂+2% CHX solution): Calcium hydroxide powder was mixed with 2% chlorhexidine gluconate solution in similar proportion as group 2 (1g Ca(OH)₂ powder to 1mL of CHX solution).

The pastes of Ca(OH)₂ with distilled water (Group 2) or Ca(OH)₂ with 2% CHX solution (Group 3) were placed inside the prepared root canal using hand pluggers until the material was just visible at the apical foramen. The quality of the intracanal medicament filling of each specimen was radiographically assessed. Excess material in the access cavity was removed with a sterile cotton pellet. A fresh cotton pellet was then inserted with an overlying seal of a non eugenol temporary filling material, Cavit G ($3M^{TM} ESPE^{TM}$, St. Paul, MN), with a depth of 2mm. The specimens were stored in an incubator for a week at 37^{0} C and 100% relative humidity.

After the 7 days period, the temporary filling material was removed with an excavator. The intracanal dressing was removed by manual use of master apical file, ProTaper F3 and irrigation using 5mL 2.5% NaOCl followed by passive ultrasonic irrigation (PUI) of a total volume of 6mL 2.5% NaOCl using a size 20 ultrasonic file (Irisafe, Acteon Satelec) mounted on a piezoelectric handpiece (Satelec) at a power setting of 6 and a total activation time of 1 minute (2mL activated for 20 seconds and the procedure was repeated 3 times). Final flush was done using 5ml of 17% EDTA, followed by 5mL distilled water. The specimens were dried with absorbent paper points.

Obturation of the specimens was done using single cone technique with matching-taper F3 gutta percha cones. Each group was subdivided into 2 subgroups (n=10) based

on the sealer used: AH Plus (Subgroup 1) and SmartPaste Bio (Subgroup 2).

Subgroup 1: AH Plus was mixed according to the manufacturer's instructions. The prefitted master cone was coated with AH Plus sealer and introduced into the canal to the working length with slight movements in apico-coronal direction to distribute the sealer.

Subgroup 2: SmartPaste Bio was injected to fill the apical third of the root using the delivery tip provided along with the sealer. The master cone was coated with sealer and introduced into the canal till the working length.

Coronal opening was sealed with Cavit G. Specimens were stored at 37^{0} C and 100% humidity for 30 days before push out bond strength testing.

Push Out Bond Strength Testing

Each specimen was transversely sectioned perpendicular to the long axis of the root using a safe sided diamond disk in a slow speed handpiece to obtain a section of $2\text{mm} \pm 0.1$ in thickness from the middle third of the root canal. Each section was coded and photographed from apical and coronal surfaces using a stereomicroscope at an original magnification of 40x. The diameter of the filling, both smaller diameter (apical surface) and larger diameter (coronal surface) was measured using an image analysis software (ImageJ, NIH, Bethesda, MD; Figure1). The radius was then calculated from the diameter measured.

The root canal filling only was subjected to loading via a universal testing machine at a crosshead speed of 1mm/min using a stainless steel plunger of 0.5mm in diameter (Figure 2). The load was applied on the apical surface of each section and in an apico-coronal direction, so as to push out the filling material towards the larger surface of the section, thus avoiding any limitation of material movement due to the taper of the root canal. Load was applied until bond failure occurred, which was manifested by sudden drop in load observed as sharp

decline on the load/time graph during compression testing and/or extrusion of the filling material. The maximum load before bond failure was recorded in newtons (N)

To express the bond strength in MPa, the load at bond failure was divided by the area of the bonded interface.

Push out bond strength (MPa) = Maximum Load (N)/ adhesion area to dentin (mm^2)

Where, MPa is the push out bond strength in megapascals and N is the maximum load before bond failure in newtons.

To calculate the adhesion area (A) for each section the following formula was used:

 $A = \pi (r_1 + r_2) \sqrt{(h^2 + (r_1 - r_2)^2)}$

Where, A is the surface area in mm^2 , π is the constant 3.14, r_1 is the larger radius (coronal surface), r_2 is the smaller radius (apical surface) and h is the thickness of the section.

Push out bond strength value for each specimen was tabulated and the data obtained was statistically analyzed. **Figure 1.** Measurement of diameter using ImageJ analysis software.



Figure 2. Schematic representation of push out bond testing using universal testing machine on 2mm thick mid root sections.



Method of Statistical Analysis

2-way analysis of variance (ANOVA) was used to detect the effect of the independent variables, material (AH Plus Bio) condition and Smart paste and (control, Ca(OH)₂+distilled water and $Ca(OH)_2+2\%$ CHX solution), and their interaction (material * condition) on push-out bond strength. One-way ANOVA was used to compare means among groups. Further post hoc Tukey tests were used to determine significant difference among the groups. The significance level was set at P < 0.05, confidence interval of 95%. Statistical analysis was performed using SPSS 16.0 software (SPSS Inc, Chicago, IL).

Results

Two-way ANOVA showed a statistically significant effect of sealer (P< .0001, Table 1), but not the medicament (P =0.210, Table 1), There was a statistically significant interaction between sealer*medicament (P<0.0001, Table 1).

Without prior Ca(OH)₂ medication (control), AH Plus showed similar push out bond strength to SmartPaste Bio (P=0.255, Table 2); With prior placement of Ca(OH)₂ mixed with distilled water, there was a statistically significant difference between the sealers (P< 0.001, Table 2). However, with prior placement of Ca(OH)₂ mixed with 2% chlorhexidine solution, there was no statistically significant difference between the sealers (P=0.265, Table 2). SmartPaste Bio showed an increase in mean push out bond strength with the prior placement of $Ca(OH)_2$ regardless of the different types used when compared to control group; a significant increase was noted only for calcium hydroxide mixed with distilled water group (Table 2). Statistically significant difference was not noted in bond strength values for AH Plus with prior placement of medicaments when compared to control group (Table 2).

Table 1: Two Way ANOVA for different Medicaments, Sealers and their Interaction

Two Way ANOVA										
Source	Type III Sum of	df	Mean Square	F	Sig.					
	Squares									
Corrected Model	21.038	5	4.208	10.153	.000					
Intercept	1580.779	1	1580.779	3814.612	.000					
Medicament	1.332	2	.666	1.607	.210					
Sealer	11.068	1	11.068	26.709	.000					
Medicament Sealer Interaction	8.638	2	4.319	10.422	.000					
Error	22.378	54	.414							
Total	1624.195	60								
Corrected Total	43.416	59								

Table 2: Mean, Standard Deviation, ANOVA and t-test values of Push Out Bond Strength values (MPa) of sealers to dentin pre treated with different calcium hydroxide medicaments

Push Out Bond Strength	AH Plus			Smart Pas			
Statistical Analysis							t-test
	Ν	Mean	SD	Ν	Mean	SD	
Control	10	4.83	.37	10	5.02	.38	<i>P= 0.255</i>
Ca(OH) ₂ + Distilled Water	10	4.25	.38	10	6.17	.68	P < 0.001
Ca(OH) ₂ + 2% Chlorhexidine	10	5.04	1.00	10	5.50	.77	<i>P</i> = 0.265
ANOVA	P = 0.032			P = 0.002			

Discussion

Among a plethora of root canal filling materials and techniques available today, the use of a sealer in conjunction with a thermoplastic core material is the most widely used combination during root canal filling.²⁷ The combined use of gutta-percha and zinc oxide eugenol or epoxy resin-based root canal sealers have given predictable clinical outcomes.²⁸ However, the quest for alternative sealers with better seal and dislocation resistance continues. More recently, bioceramic sealers introduced which exhibit were outstanding biocompatibility, excellent sealing and osseoconductive properties.⁷ However, whether the residues of different intracanal medicaments would affect the dislocation resistance of the bioceramic sealers required investigation. This study compared and evaluated the effect of different calcium hydroxide medicaments on the bond strength of an epoxy resin based sealer and a bioceramic sealer.

Several studies have evaluated different techniques for intracanal $Ca(OH)_2$ medicaments. removal of Lambrianidis et al. reported that amount of calcium hydroxide powder in the paste does not affect the removal but the vehicle used for making the paste can affect the retrieval.²⁹ Conventionally, rotary NiTi-instruments and apical 'patency file' have been suggested to remove calcium hydroxide.^{3,30} Studies showed better Ca(OH)₂ removal when using certain irrigants like NaOCl and chelating agents like citric acid and EDTA. PUI has been reported to improve Ca(OH)₂ removal when used along with the conventional methods.^{16,17,31} Nevertheless, no technique has been able to completely remove Ca(OH)₂ from the canal.^{13-17,29} In the present study, the medicaments were removed by combining the manual use of master apical file and passive ultrasonic irrigation.

Several methods have been employed to evaluate the adhesive strength of root canal filling materials that

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include shear bond strength, microtensile bond strength,³² and push out bond strength testing.³³ The push out test is a reliable technique to measure the bond strength of root canal filling materials to root dentin. It provides a realistic assessment of bond strength, even at low levels.¹

SmartPaste Bio showed similar dislodgement resistance to AH Plus in the control group i.e, without medicament. The results are in corroboration with other studies where bioceramic sealer has shown similar bond strengths to AH Plus sealer.^{3,34,35} Bioceramic sealers release calcium and hydroxyl ions which results in the formation of an apatite layer when it comes in contact with phosphate containing fluids for 2 months.³⁶ Formation of this interfacial layer develops a chemical bond between calcium silicate-based materials and dentinal walls which may be partly attributed to its high dislodgement resistence.

Ersahan and Aydin conducted an in-vitro study to evaluate the push-out bond strength of iRoot SP with AH Plus, Sealapex and EndoREZ Bond. They concluded that iRoot SP and AH Plus performed similarly and better than EndoREZ and Sealapex in terms of bond strength.³ Similarly, Sagsen et al. assessed the push-out bond strength of two new calcium silicate-based endodontic sealers i.e, iRoot SP and MTA Fillapex, in the root canals of extracted teeth. They found that the push-out bond strengths of AH Plus and iRoot SP were significantly superior to that of MTA Fillapex, but no significant difference between AH Plus and iRoot SP was found.³⁵ More recently, in accordance with the previous studies, Shokouhinejad et al. also found that the bond strength of the new bioceramic sealer, EndoSequence BC Sealer, was equal to that of AH Plus with or without the smear layer.³⁴ However, variability seems to exist in the bond strength of bioceramic sealers.^{6,24} Nagas et al. conducted an in-vitro study to evaluate the effects of intraradicular moisture conditions on the push-out bond strength of iRoot SP. AH

Plus, Epiphany, and MTA Fillapex. They concluded that iRoot SP presented the highest bond strength compared with the other tested sealers, irrespective of the canal moisture conditions.⁶ These discrepancies among the results may be explained on the basis of differences in experimental designs like obturation technique, storage period and/or sealer brands and compositions used.²⁴ In the present study, a matching taper single cone technique was used because of its popularity and also to facilitate comparison with other studies. Higher bond strength of iRoot SP when compared to AH Plus in Nagas et al.'s study may be attributed to the obturation technique, continuous wave obturation with System B, used in their study.⁶ The difference in results may also be partly explained based on the storage period of specimens after obturation and before bond testing. The specimens were stored for 30 days in the present study unlike the 7-day period in their study. It has been recently documented that calcium silicate-based materials has an adverse effect on the integrity of the dentin collagen matrix with prolonged contact. Ca(OH)₂ released from these materials can penetrate the intrafibrillar network of the mineralized collagen fibrils and alter the 3-dimensional conformation of tropocollagen.³⁷ Literature has also recorded lower bond strength of bioceramic sealers. Amin et al. showed that iRoot SP had significantly lower bond strength when compared to AH Plus.²⁴ Despite similarity in methodology, variation in bond strengths was found. Higher bond strength for bioceramic sealer recorded here may be attributed to the difference in the sealer brand used in the study. The high bond strength of SmartPaste Bio may be explained by its calcium silicate composition, which uses the moisture naturally present in dentinal tubules to initiate and complete the setting reaction so that no shrinkage occurs during setting.³

SmartPaste Bio recorded the highest bond strength with prior placement of calcium hydroxide mixed with distilled water compared to all other groups. Regardless of the type of calcium hydroxide medicament, SmartPaste Bio showed an increase in bond strength. These findings support previous studies in which Ca(OH)₂ remnants improved the dislocation resistance of calcium silicate based sealers.²⁴ Residual calcium hydroxide also improved the marginal adaptation of MTA^{26} . It is presumed that Ca(OH)₂ residues might chemically interact with root canal sealer or they might increase the frictional resistance and/or micromechanical retention of SmartPaste Bio sealer in this study.²⁴ Currently, there is no literature reported regarding the influence of a mixed intracanal medication containing calcium hydroxide powder and 2% chlorhexidine solution on bonding ability of bioceramic sealers. In the present study, the remnants of combination of calcium hydroxide and 2% chlorhexidine did not improve the bond strength of bioceramic sealer significantly.

In the current study, residual calcium hydroxide medicaments did not affect the bonding behavior of AH Plus significantly, which is in agreement with other studies.³⁸⁻⁴¹ The slight decrease in values for Ca(OH)₂+distilled water group suggest that the Ca(OH)₂ residues left in the root canal space could have interfered with the adhesion of AH Plus sealer. Calt and Serper demonstrated that residual Ca(OH)₂ prevents penetration of sealer into dentinal tubules.⁴² Ca(OH)₂+ 2% Chlorhexidine had a positive effect on the bonding behavior of AH Plus sealer, but the difference was not statistically significant when compared to the control group. These results are in agreement with previous studies conducted to evaluate the influence of a mixed intracanal medication containing calcium hydroxide powder and 2% chlorhexidine solution on bonding ability

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of AH Plus sealer.38,40

Recently, newer irrigation systems like continuous passive ultrasonic irrigation, Self Adjusting File and EndoVac have been evaluated for their efficacy in removing calcium hydroxide from simulated irregularities in the apical part of root canal and found that Continuous PUI and SAF were more effective than EndoVac.⁴³ However, in the present study, an intermittent flush method of PUI was used for the removal of calcium hydroxide from the root canals.

The extrapolation of the results from work that is purely in-vitro in nature must always be made with caution. The ultimate clinical decision-making should consider the patient related variables to maximize the long-term prognosis of endodontically treated teeth. In the light of the current study's results, further research should be carried out, seeking for a better technique for removal of calcium hydroxide or to investigate the effect of residual calcium hydroxide on the physical properties of different bioceramic sealers under various experimental conditions. Within the limitations of the study, it was inferred that when calcium hydroxide is used as a medicament, bioceramic sealer may be preferred over epoxy resin based sealer in terms of bond strength. However, with prior placement of combination of calcium hydroxide and 2% chlorhexidine, both sealers performed similarly with respect to dislodgement resistance.

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