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ORIGINAL ARTICLE

An *in vitro* Comparison of the Effect of Three Different Endodontic Medicaments on the Bond Strength of a Resin-based Endodontic Sealer to the Root Canal Dentin

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ABSTRACT

Objectives: The aim of this study was to compare and evaluate the effect of calcium hydroxide [$Ca(OH)_2$], 2% chlorhexidine (CHX) gel, and propolis when used as intracanal medicaments on the bond strength of a resin-based sealer (AH Plus Jet; Dentsply DeTrey, Konstanz, Germany) to the root dentin.

Materials and methods: Sixty freshly extracted single-rooted human teeth were utilized for this study and the crowns were decoronated. The root canals were instrumented and randomized into four groups according to the medicament used: Group I: Control; group II: $Ca(OH)_2$; group III: CHX; group IV: Propolis. The specimens were stored for 10 days in 100% humidity at 37°C, and the intracanal dressings were removed by rinsing with 10 mL of 17% ethylenediaminetetraacetic acid followed by 10 mL of 2.5% sodium hypochlorite, then obturated with gutta-percha and AH Plus Jet sealer. A push-out test to measure the bond strength between the root canal dentin and the sealer was done. Two-way analysis of variance and Tukey's *post hoc* pairwise tests were used for statistical analysis.

Results: The use of intracanal medicaments had shown to significantly influence the push-out bond strengths of the resin sealer used ($p < 0.05$). At coronal and middle thirds, there was no significant difference in bond strengths among all the four groups ($p > 0.05$). At apical third, the mean bond strength value for propolis group was significantly superior when compared with the other three groups ($p < 0.05$). The CHX group showed higher bond strength values at apical third compared with $Ca(OH)_2$ and control groups, but it was not significant ($p > 0.05$).

Conclusion: Propolis showed superior push-out bond strength than CHX, $Ca(OH)_2$, and control groups at apical third, while no significant differences were observed among all the groups at coronal and middle thirds.

Keywords: AH Plus Jet, Bond strength, Calcium hydroxide, Chlorhexidine, Propolis, Push-out test.

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INTRODUCTION

The fundamental criteria required for the success of endodontic therapy includes correct diagnosis, thorough cleaning and shaping, and complete three-dimensional obturation of root canal space.[1] Although there has been a tremendous improvement in root canal instrumentation procedures, there is no evidence showing complete cleaning of the root canal system with the available instrumentation techniques, because of the ability of microbes to persist in the complex anatomy of root canal spaces.[2] Therefore, the infected root canal is subjected to combined chemomechanical treatment involving instrumentation in integration with extensive irrigation.

Viable microorganisms that remain even after root canal preparation and disinfection contribute significantly to the failure of root canal therapy.[3] The most common species isolated from the root canals with secondary apical periodontitis are *Candida albicans* and *Enterococcus faecalis.*[4] Therefore, the use of an intracanal dressing has been suggested by several studies as a valuable adjunct to chemomechanical preparation in disinfection of the root canal system.[5]

Calcium hydroxide [$Ca(OH)_2$] has been known to be widely used as an intracanal medicament since past times owing to its consistent antibacterial activity and minimal cytotoxicity. But its role in eliminating *C. albicans* and *E. faecalis* is uncertain.[6,7] For this reason, new compounds, such as chlorhexidine (CHX), antibiotics, and natural compounds like propolis have been suggested to be used as alternative intracanal medications.[8]

The search for an effective antimicrobial agent led to use of CHX within the root canals. The CHX when used as an intracanal medicament has shown commanding results even against *E. faecalis* and *C. albicans.*[9,10]

A natural product propolis has gained more appreciation in dentistry in recent times. Propolis (bee glue) is a flavanoid-rich resinous product of honeybees. It has been utilized in dentistry as an anticaries agent, a storage medium for avulsed tooth, a pulp capping agent, and as a sealant for dentinal hypersensitivity.[11-14] Other

authors have shown that propolis can be useful as a root canal dressing owing to its little toxicity and extensive antibacterial range.[13]

In addition to the antimicrobial properties, intracanal medications should be easy to remove from the root canal walls, the failure of which can lead to the impairment in the performance of endodontic sealer due to the obstruction of dentinal tubules by these intracanal medicaments.[15,16]

Thus the current *in vitro* study was assumed to evaluate the effect of three different endodontic medicaments – $Ca(OH)_2$, CHX, and propolis on the bond strength of AH Plus Jet resin sealer to root dentin using push-out test.

MATERIALS AND METHODS

The study was performed in the Department of Conservative Dentistry and Endodontics at College of Dental Sciences, Davangere, India. The present study used 60 freshly extracted single-rooted human teeth with no detectable cracks or fractures, abnormal curvatures, and internal or external resorption of roots. They were stored in physiologic saline solution until use. The teeth were decoronated with the use of diamond disk under water coolant, and root lengths were standardized approximately to 14 to 16 mm.

Size 15 K-file was inserted into the canal till the tip was envisioned at the apical foramen. Working length was then established by reducing 1 mm from the length of the file. Root canal preparation was done using ProTaper Next (Dentsply Maillefer) rotary files up to a master apical file size of #X2 with a torque and speed-controlled electric motor. Irrigation was done using 3 mL of 2.5% sodium hypochlorite (NaOCl) between each successive instrumentation. Finally, the root canals were irrigated with 5 mL of 17% ethylenediaminetetraacetic acid (EDTA) for 3 minutes and 5 mL of distilled water was used as the final rinse. The root canals were dried using sterile absorbent paper points (Dentsply Maillefer). Subsequently, the teeth were divided into four experimental groups (n = 15) as follows:

Group I: without intracanal medicament (Control)

Group II: $Ca(OH)_2$/distilled water paste. The $Ca(OH)_2$ paste in this group was prepared by mixing $Ca(OH)_2$ powder (Kalsin; Spot Dis Deposu AS, Izmir, Turkey) with distilled water (1:1.5, powder to liquid ratio) until a creamy consistency was reached.

Group III: 2% CHX gel (Endogel)

Group IV: Propolis powder/glycerine paste. Propolis paste was prepared by manipulating propolis powder using glycerine until a creamy consistency was reached.

The prepared pastes were placed into the root canals using a size #30 Lentulo spiral. The access openings of the root canals were sealed with a small cotton pellet and temporary filling material (Cavit G, 3M ESPE, Seefeld, Germany) to prevent leakage. In the control group, access cavities were directly sealed with temporary filling material without intracanal medicament. The specimens were stored at 37°C in 100% humidity for 3 weeks to simulate clinical conditions.[17,18]

After 3 weeks, the medicaments were rinsed off by using 10 mL 17% EDTA followed by 10 mL 2.5% NaOCl and final irrigation was done with 5 mL distilled water.[17,19] The root canals were dried using paper points and the canal walls were then coated with AH Plus Jet sealer and a single gutta-percha cone (X2 ProTaper Next, Dentsply Maillefer) was inserted into the root canals of all the specimens. Mesiodistal and buccolingual radiographs were taken to confirm complete filling. After root filling, the coronal access cavities were sealed with a temporary filling material, and the specimens were stored at 100% humidity at 37°C for 1 week to completely set.

Push-out Test

For push-out test analysis, specimens were sectioned perpendicular to the longitudinal axis of the root using a diamond disk at a slow speed under water coolant. Three slices with thickness of 2 ± 0.1 mm were obtained from each tooth (n = 30 for each group) at the extent of 3, 8, and 12 mm from the coronal surface. Thickness of each slice was carefully observed using a digital caliper to eliminate the influence of specimen thickness variation. The push-out test was performed on each specimen with a universal testing machine at a crosshead speed of 1 mm/min using 0.6-, 0.7-, and 0.8-mm diameter cylindrical pluggers, matching with the diameter of each canal third. The maximum load applied to the filling material before failure was recorded in Newton and converted to megapascals.

The force applied to dislodge the filling material (in kN) was converted to shear stress (in MPa) using the formula:

Push-out bond strength (MPa) = maximum load (N)/sealer adhesion area (SL) (mm^2) (calculated for both the upper and lower surfaces of each slice) SL was calculated using the below formula:

$$SL = p(R + r)g, \qquad g = \sqrt{(r - R) + h2}$$

where SL=sealer adhesion area, p = 3.14, R = mean radius of coronal aspect of canal (mm), r = mean radius of apical aspect of canal (mm), g = height relative to the tapered inverted cone (mm), and h = thickness of root section (mm).[20]

Stereomicroscopic Analysis for Failure Mode

After the test procedure, each specimen was visualized to evaluate the failure type under a stereomicroscope

(Olympus, 32× magnification). Three types of failures were grouped: Adhesive failure (between the sealer and root dentin), cohesive failure (within the sealer or root dentin), and mixed (a combination of cohesive and adhesive).[21]

Statistical analysis using two-way analysis of variance was done to evaluate the significance of effect of medicaments on the push-out bond strengths of resin sealer. Tukey's *post hoc* test was used to exactly determine the difference between the medicaments at each root canal third. The data for failure mode were statistically analyzed using chi-square test. Statistical significance was defined at $p < 0.05$.

RESULTS

Push-out Strength

The push-out bond strength results of each group at three different root canal thirds are shown in Table 1. At each root canal region, statistical comparisons were conducted among the four experimental groups.

At apical third region, there was significant difference between propolis group and the remaining three experimental groups ($p < 0.05$), with the highest bond strength values shown by propolis group and the next higher values seen in the order of: Control, CHX, and $Ca(OH)_2$ groups. At coronal and middle third regions, there were no significant differences in bond strengths among the four groups ($p > 0.05$).

Table 1: Mean (MPa) and standard deviation of push-out bond strength values for the four experimental groups at different root canal thirds

		Mean (MPa)	*Standard deviation*	*p-value*
Apical	Group IV: Propolis paste	6.932	(3.964)	0.022
	Group III: CHX gel	4.306	(2.312)	
	Group II: $Ca(OH)_2$ paste	3.675	(2.112)	
	Group I: Control	2.492	(1.456)	
Middle	Group IV: Propolis paste	3.702	(2.630)	0.534
	Group III: CHX gel	3.620	(2.124)	
	Group II: $Ca(OH)_2$ paste	3.503	(2.054)	
	Group I: Control	3.786	(3.042)	
Coronal	Group IV: Propolis paste	2.024	(1.860)	0.520
	Group II: CHX gel	2.030	(1.206)	
	Group III: $Ca(OH)_2$ paste	2.676	(1.234)	
	Group I: Control	2.548	(1.082)	

Failure Modes

The failure mode results of the three experimental groups according to the root canal region are shown in Table 2. Adhesive failure was the least noticed failure mode among all the three groups. Cohesive and mixed failure modes accounted for most of the failures that had been noticed.

DISCUSSION

Intracanal medicaments are commonly recommended to eliminate remaining microbes in the root canal, dentinal tubules, accessory canals, canal irregularities and to reduce periapical inflammation, encourage periapical healing, eliminate apical exudates, control inflammatory root resorption, and avert contamination of the canal between appointments.[22]

Calcium hydroxide is considered to be one of the mainstay root canal medicaments. But it has shown to be unsuccessful at eliminating *E. faecalis* and *C. albicans*, due to the ability of these microbes to survive in high-alkaline pH environment provided by $Ca(OH)_2$.[23] So, exploration for possible intracanal medicaments has continued.

Recently, attention has been centered on the effectiveness of CHX as an intracanal medicament. Chlorhexidine has been widely used as a medicament in the treatment of infected root canals owing to its extensive range of antimicrobial activity, substantivity, low toxicity, and water solubility.[24,25] It has been shown to be efficacious against *E. faecalis* and *C. albicans*.[26,27]

Propolis, a centuries-old natural antibiotic, has also been tried as an intracanal medicament due to its superior antimicrobial activity against a wide range of bacterial species including *E. faecalis* and *C. albicans*.[28,29] Therefore, propolis could be used as an alternative intracanal medicament in cases of persistent endodontic infections. However, understanding the effect of these materials on bond strength of resin-based sealers to root dentin is also considered to be important before recommending to use as intracanal medicaments.

Several authors have evaluated the difficulty of removing intracanal medicaments from root canal walls, especially in their apical part, that could adversely affect dentinal bond strength and compromise endodontic sealing.[30] In the present study, canals were flushed with

Table 2: Failure mode distribution of groups I to III (%) according to root canal region

	Group IV: Propolis			*Group III: CHX*			*Group II: $Ca(OH)_2$*			*Group I: Control*		
	C	*M*	*A*	*C*	*M*	*A*	*C*	*M*	*A*	*C*	*M*	*A*
Adhesive	0	0	0	0	0	10	0	0	20	0	0	20
Cohesive	60	50	45	50	50	30	70	60	15	50	60	10
Mixed	50	40	50	40	50	40	40	50	70	40	45	60

C: Coronal third; M: Middle third; A: Apical third

10 mL of 17% EDTA followed by 10 mL of 2.5% NaOCl, and 5 mL distilled water as final irrigant to remove intracanal medicaments which was based on previous studies.[17,19] The results showed better removal of medicaments as compared with the study done by Victorino et al[8] that evaluated the removal efficiency of the medicaments using 3 mL of 1% NaOCl and 3 mL of 17% EDTA and saline as final irrigation solutions. According to that study, no significant difference in root canal cleanliness for the removal of propolis or $Ca(OH)_2$ root canal dressings was noticed. The potential reason for better removal in our present study could be because of higher circulation volume and concentration of irrigation solutions used that had given desirable results within a less period of time.[17,19]

Different mechanical methods have been suggested for evaluation of bond strength to a material, such as shear bond strength, microtensile bond strength, and push-out tests. The rationale for using push-out test in this study include: Being easy to reproduce, interpret, and being able to realistically record the bond strength to dentin even at low levels.[31,32]

The results of the current study showed that propolis has significantly highest push-out bond strength values at the apical third when compared with the other three groups, followed by the next higher values seen for CHX, $Ca(OH)_2$, and the control group in the same order. At coronal and middle third regions, the difference noted in bond strengths was not statistically significant between all the four experimental groups. These results were in accordance with similar studies done by Shivanna and Bhargavi[19] and Üstün et al[33] in which they compared the effect of propolis and other intracanal medicaments on the push-out bond strength of AH Plus resin sealer. The most probable reason for this was due to the hydrophilic resin components present in propolis paste that tightly bound to the hydrophilic surface of dentin. In addition, the circulation volume of irrigation solutions at apical third was lower than at coronal and middle thirds. Thus, the removal of resinous sticky form of propolis from the root dentin walls became more difficult, which could be accountable for binding to AH Plus Jet sealer.[19,33]

The lesser bond strengths of CHX and $Ca(OH)_2$ could be due to their incomplete removal from root canals after irrigation, which further prevented the penetration of sealer into the dentinal tubules, thus adversely affecting the sealer bond strength. This was in accordance with the study done by Srivastava et al[34] in which the results showed that CHX when used in liquid form could be easily removed, thus improving the sealing ability.

The similar bond strengths obtained at coronal and middle thirds for all the four experimental groups could be due to the fact that canal space at coronal and middle third was anatomically larger than that at apical third. Thus, with a higher circulation volume of irrigation solutions, it facilitates better removal of smear layer and intracanal medicaments at coronal and middle thirds than at apical third. These results were in accordance with the study done by Akcay et al[35] and few other studies, where there was no significant difference in push-out bond strength of resin sealer at coronal and middle thirds with the use of different intracanal medicaments.[19,33]

No adhesive failures were appreciated for the propolis group at apical third in our current study, but not so for the remaining three groups (Table 2). This result could be explained by the high bond strength of propolis group at apical third. Similar results were obtained in the study done by Üstün et al.[33]

Based on the results of this study, propolis as an intracanal medicament does not have any adverse effect on the push-out bond strength of AH Plus Jet sealer at apical third, which is critical for the success of endodontic therapy. Further studies should be directed to evaluate the long-term effect of propolis on dentin microhardness and chemical structure.

CONCLUSION

Within the limitations of this study, propolis intracanal medicament showed significantly higher push -out bond strength with AH Plus Jet sealer as compared with $Ca(OH)_2$ and CHX at the apical third, which is critical for the success of endodontic therapy. However, the exact chemical reaction between resin ingredients of propolis and the epoxy resin sealer should be further investigated.

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