Original Article

Effect of Distinctive Moisture Conditions on Push-out Bond Strength of Three Root Canal Sealers—An *In-Vitro* Study

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ABSTRACT

Objective: Endodontic sealer should adhere to both dentin and the core filling material but the moisture conditions of the canals affect the adhesive properties of the sealer. An ideal sealer with perfect moisture conditions will lead to greater strength of the restored tooth, which may provide greater resistance to tooth fracture and clinical longevity of an endodontically treated tooth. The aim of this study was to evaluate the effect of moisture conditions on the push-out bond strength of three root canal sealers: AH Plus® (Dentsply-Tulsa Dental, Tulsa, OK), Epiphany (Pentron Clinical Technologies, Wallingford, CT), and GuttaFlow (Coltene/Whaledent, Altstatten, Switzerland). **Materials and Methods:** A total of 120 single-rooted, non-carious teeth were collected for the study and were stored in normal saline. The root canals were prepared using step-back technique. Teeth were divided into four groups based on type of drying procedure used and further subdivided into three subgroups based on the type of sealer used. The samples were cut horizontally to produce slices and then tested for push-out bond strength using Universal Testing Machine (Servo Series 50 kN; P S I Sales Private Limited, New Delhi, India). The specimens were examined for mode of fracture under magnification and the results were analyzed statistically. **Results:** Distinctive moisture conditions for all sealers were observed and the highest strength of AH Plus® was evaluated under moist condition, Epiphany under dry condition, and GuttaFlow under normal condition, respectively. **Conclusion:** Distinctive moisture conditions affect the push-out bond strength of the sealers.

Keywords: *AH Plus®, epiphany, GuttaFlow, moisture, sealers*

Introduction

A successful endodontic treatment depends on three-dimensional restoration with an inert, dimensionally, and biologically stable material. The quality of adhesion between root canal dentin and sealers depends on the level of moisture present in the root canals before filling.[1] The residual moistures inside the canal have been shown to alter the sealing properties of conventional and resin-based sealers.[2] The manufacturers recommended that dentinal walls are kept moist, not dehydrated to take maximum advantage of the hydrophilic properties of the sealers.[3]

Therefore, the effect of residual intra-radicular moisture on the bond strength of sealers should be determined.

This study aimed to evaluate the effect of residual intra-radicular moisture on the bond strength of three root canal sealers, such as AH Plus®, Epiphany, and GuttaFlow, and to analyze modes of fracture under magnification. The investigators hypothesized that different moisture conditions obtained by drying with ethanol, paper point,

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and low vacuum do not have any effect on the strength of the sealers. The study will evaluate any effect of moisture on the strength of sealers and provide optimal conditions required for sealing the root canal with most commonly available sealers in the market.

Materials and Methods

The sample size calculation was performed using the Power and Sample Size Calculation program (Vanderbilt Biostatistics, Nashville, TN). The estimated power was 0.80 and the significance level was set at $\alpha = 0.05$. A minimum of 10 teeth per group were needed for this study to reject the null hypothesis.

A total of 120 freshly extracted single-rooted, human permanent mandibular first premolars of approximately the same size were selected for this study in a period of one year. The samples were selected based on condition of the extracted teeth having the following exclusion criteria: caries, curved roots, open apices, cracks, or previous root canal treatments. A radiograph was taken for each tooth sample to ensure no intracanal abnormality. The specimens ($n = 10$) were randomly distributed in 12 groups. The samples were stored in sterilized normal saline for one month.

Sealers

The sealers used in the study were as follows: AH Plus® (Dentsply-Tulsa Dental, Tulsa, OK), Epiphany (Pentron Clinical Technologies, Wallingford, CT), and GuttaFlow (Coltene/Whaledent, Altstatten, Switzerland).

AH Plus® is an epoxy resin-based endodontic sealer, which is used in combination with gutta-percha in compaction techniques. AH Plus® comes in a paste–paste system. It has a working time and setting time of 4 h and 8 h, respectively.

Epiphany is the first proprietary endodontic sealer that was designed to bond with both dentin and Resilon (Resilon Research LLC, Madison, CT). Resilon–Epiphany system has a self-etching primer and a dual-curable hydrophilic resin-based sealer whose use with Resilon creates a monoblock, which provides a greater resistance to microbial leakage and modest reinforcement of teeth against root fracture.[4]

GuttaFlow is a flowable root canal filling material. It is a combination of both the sealer and gutta-percha in one injectable system. The sealer is silicone based with polymethyl hydrogen siloxane as its main component and finely ground gutta-percha. Because of its better flow properties, GuttaFlow has good homogeneity and adaptation to the root canal walls and also flows into lateral grooves and depressions.[5]

Moisture Conditions with Drying Material

The Root canals were dried with ethanol (Lot no 20160110; Changshu Yangyuan Chemical Co. Ltd., Changshu City, China), paper points (Lot no PE16060726; Meta Biomed Co. Ltd., Chungcheongbuk-do, Korea), and Luer Vacuum Adapter (Ultradent® Products, Coogee, NSW, Australia), Moist Condition was achieved by not drying the root canals.

Procedure

The specimens were decoronated below cementoenamel junction basically perpendicular to long axis using a slow-speed, water-cooled diamond disk (Isomet 2000, Buehler Ltd., Lake Bluff, NY). The root canal of each tooth initially was assessed using #10 K-file (Dentsply Maillefer, Ballaigues, Switzerland) until apical foramen was reached. The working length was determined by Ingles's[6] method. Root canal preparation was carried out using hand K files (Dentsply Maillefer, Ballaigues, Switzerland) up to size #40 by using step-back technique. Root canals were prepared and irrigated with 17% ethylenediaminetetraacetic acid (Smear Clear™, SybronEndo, Orange, CA) followed by rinsing with distilled water. Samples were divided into four groups based on intracanal drying procedure used: Group A—control group: kept moist; Group B—dried with ethanol; Group C—dried with paper points; and Group D—dried under low vacuum. Each group was further divided into three subgroups based on type of sealer used: Subgroup a—AH Plus®; Subgroup b—Epiphany; and Subgroup c—GuttaFlow. All the canals were obturated with cold lateral compaction. Resilon points were used in Subgroup b, whereas gutta-percha points (Lot no GE16030189; Meta Biomed Co. Ltd., Chungcheongbuk-do, Korea) were used in Subgroups a and c.

After obturation, the samples were stored at 37°C for 24 h under 100% of relative humidity. A sharp diamond disk was used for preparing 1-mm-thick transverse sections from the coronal, middle, and apical third of the specimens in the form of disks [Figure 1]. The test was carried out for push-out strength using special jig equipment specially fabricated for the study to mount the specimens on it while testing for the push-out bond strength of the obturating material to dislodge it. The samples were fitted on the jig. It was ensured that the coronal surface of the cut section was in between the metallic supports. The cylindrical plunger with a diameter of 0.9 mm was placed at the center of the root canal, preventing its contact with the dentin surrounding the filling. The amount of push-out force applied by the tip of the cylindrical plunger was measured by a Universal Testing Machine (Servo Series 50 kN; P S I Sales Private Limited, New Delhi, India). The tip of the cylindrical plunger was placed on the surface of the root canal and the force was applied at a crosshead speed of 1 mm/min in the apico-coronal direction. The maximum force necessary to push the filling material of the sample was considered as the bond failure point and was recorded by using the following formula: $A=2\pi r \times h$, where r is the radius of the root canal space measured using a digital Vernier caliper (Mitutoyo, Toyko, Japan) and h is the thickness of the samples in mm. Therefore, the bond strength (δ) was calculated in MPa using the following formula: $\delta=F/A$. All fractured fragments were observed under digital stereomicroscope (Carl Zeiss, Oberkochen, Germany) at ×30 magnification and modes of failure were seen. The criteria for classification of mode of failures were as follows: adhesive failure, cohesive failure, and mixed failure. Representative samples from each failure group were analyzed under digital scanning electron microscope (JSM 6100; JEOL Ltd., Akishima, Tokyo, Japan).

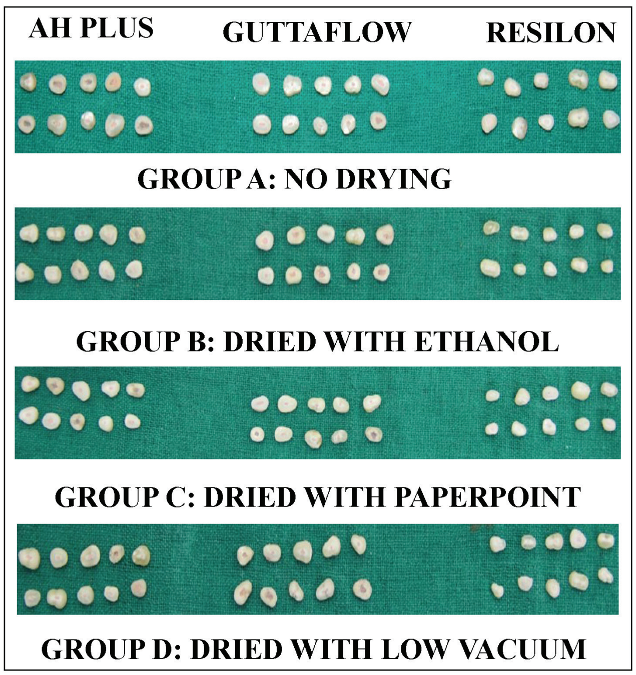

Figure 1: Samples

Statistical Analysis

The analyzed data were tabulated and subjected to statistical analysis using Statistical Package for the Social Sciences software version 18.0 (SPSS, Chicago, IL). Mean and standard deviation were calculated for each group and statistical significance was analyzed using one-way analysis of variance (ANOVA) and *post-hoc* Tukey's honestly significant difference (HSD) tests.

Results

The push-out bond strength [Table 1] of AH Plus® under wet condition, that is, no drying, was 1.301 ± 0.851 Mpa [Figure 2]. The push-out bond strength of AH Plus® when dried with ethanol was 2.252 ± 0.420 Mpa [Figure 3], with paper point was 1.864 ± 0.684 Mpa [Figure 4], and with Luer Vacuum Adapter (low vacuum) was 3.997 ± 1.08 Mpa [Figure 5]. The push-out bond strength of AH Plus® when dried with Luer Vacuum Adapter was higher than that of

Table 1: Mean push-out bond strength (Mpa)

Group	Subgroup	Mean push-out bond strength (Mpa)	Std. deviation	Std. error	95% confidence interval for mean		Minimum	Maximum
					Lower bound	Upper bound		
A: no drying	a: AH Plus®	1.301	.851075	.269134	.69173	1.90938	.357	3.441
	b: Epiphany	1.730	1.160390	.366948	.90030	2.56048	.297	3.537
	c: GuttaFlow	0.826	.180861	.057193	.69631	.95507	.653	1.149
B: dried with ethanol	a: AH Plus®	2.252	.420430	.132952	1.95096	2.55247	1.717	2.837
	b: Epiphany	1.913	.677644	.214290	1.42825	2.39777	.822	2.758
	c: GuttaFlow	0.666	.469177	.148367	.33022	1.00148	.239	1.766
C: dried with paper point	a: AH Plus®	1.864	.684715	.216526	1.21608	2.19571	1.118	3.334
	b: Epiphany	1.764	.852445	.269567	1.27111	2.49071	1.062	3.388
	c: GuttaFlow	1.685	1.484976	.469591	.79460	2.91918	.433	4.459
D: dried under low vacuum	a: AH Plus®	3.997	1.076981	.340571	3.22656	4.76741	1.833	5.580
	b: Epiphany	1.734	.411203	.130034	1.43946	2.02778	1.020	2.595
	c: GuttaFlow	1.267	.819617	.259186	.68114	1.85378	.300	2.524

the other conditions. The push-out bond strength of Epiphany when dried with ethanol (1.913 ± 0.677 Mpa) [Figure 3] was recorded the highest as compared to the other drying conditions. The push-out bond strength of GuttaFlow was recorded the highest when dried with paper points (1.685 ± 1.484 Mpa) [Table 1 and Figure 5] as compared to the other moisture conditions. The lowest push-out bond strength of GuttaFlow was 0.666 ± 0.469 Mpa [Table 1 and Figure 5].

A one-way ANOVA test, also known as statistical analysis or technique, [Table 2] was performed between each group for all sealers and was found to be very highly significant ($P < 0.001$) with AH Plus®, highly significant ($P = 0.022$) with GuttaFlow, and showed no significant difference statistically ($P = 0.937$) with Epiphany while comparing under moisture conditions.

The post-hoc Tukey's HSD test [Table 3] was performed in each group and was found to be very highly statistically significant ($P < 0.001$) with AH Plus® under moist conditions (dried with low vacuum) as compared to other sealers and GuttaFlow when dried with ethanol.

Predominant mode of failure for all the sealers (AH Plus®, Epiphany, and GuttaFlow) was adhesive failure (n = 101/120), cohesive failure (n = 12/120), and mixed failure (n = 7/120) [Figure 6]. Adhesive failure at sealer dentin interface (N [A1] =62/101) was higher as compared to sealer/core material interface (N [A2] =39/101).

Discussion

This *in-vitro* study was undertaken to determine the effect of distinctive moisture conditions on the push-out bond strength of three root canal sealers: AH Plus®, Epiphany, and GuttaFlow. The findings of the study

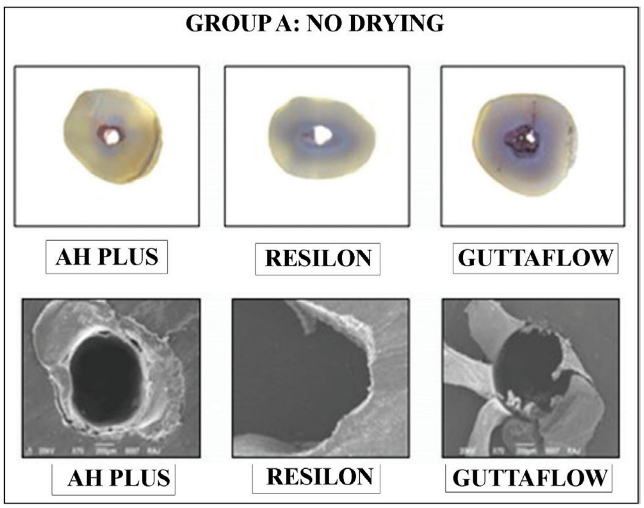

Figure 2: Steriomicroscope and SEM images of Group A

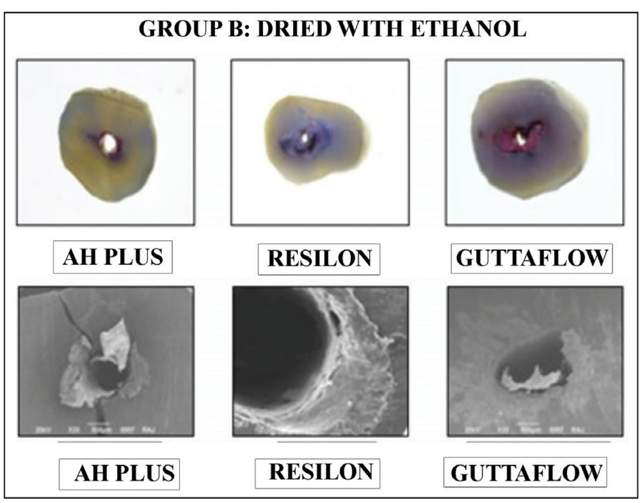

Figure 3: Steriomicroscope and SEM images of Group B

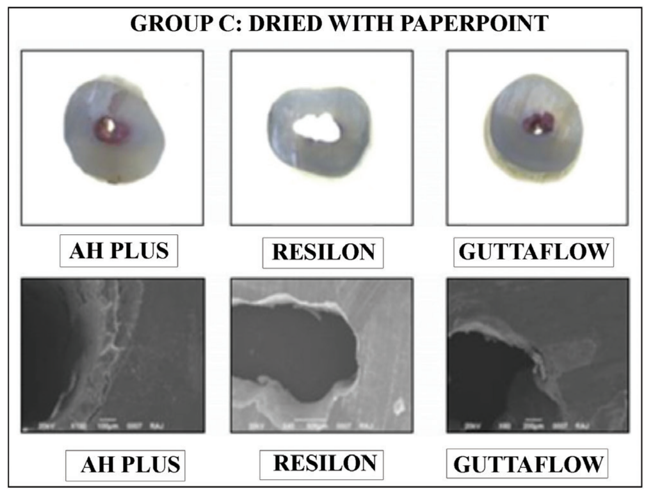

Figure 4: Steriomicroscope and SEM images of Group C

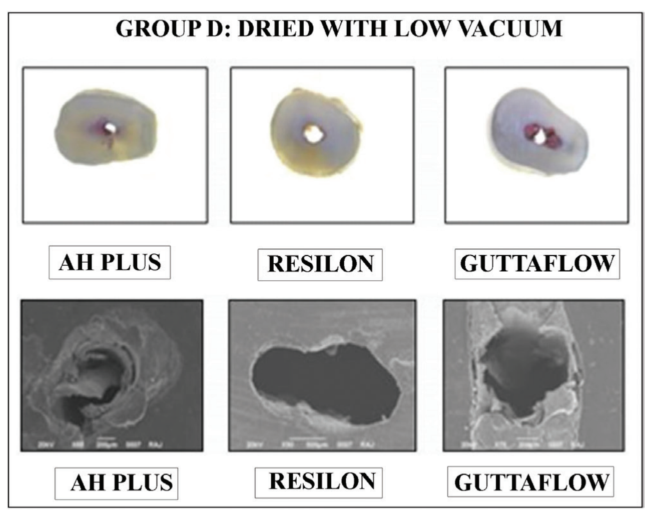

Figure 5: Steriomicroscope and SEM images of Group D

Table 2: One-way ANOVA statistical test

ANOVA

Push-out bond strength

Subgroup		Sum of squares	df	Mean square	*F*	Sig.
AH Plus®	Between groups	42.332	3	14.111	22.311	<.001[a]
	Within groups	22.768	36	.632		
GuttaFlow	Between groups	8.530	3	2.843	3.634	.022[b]
	Within groups	28.168	36	.782		
Resilon	Between groups	.277	3	.092	.137	.937
	Within groups	24.313	36	.675		

[a] The mean difference is very highly significant at the <0.001 level

[b] The mean difference is significant at the 0.05 level

Table 3: *Post-hoc* (Tukey's HSD) test

Multiple comparisons

Dependent variable: Push-out bond strength

Tukey's HSD

Group	Subgroup (I)	Subgroup (J)	Mean difference (I–J)	Std. error	Sig.	95% confidence interval	
						Lower bound	Upper bound
No drying	AH Plus®	GuttaFlow	.474865	.374481	.425	–.45363	1.40336
		Resilon	–.429832	.374481	.494	–1.35833	.49866
Dried with paper point	AH Plus®	GuttaFlow	–.150997	.476141	.946	–1.33155	1.02955
		Resilon	–.175016	.476141	.928	–1.35557	1.00554
Dried with ethanol	AH Plus®	GuttaFlow	1.585859[a]	.238899	<.001[a]	.99353	2.17819
		Resilon	.338703	.238899	.346	–.25363	.93103
Dried under low vacuum	AH Plus®	GuttaFlow	2.729528[a]	.365217	<.001[a]	1.82400	3.63505
		Resilon	2.263369[a]	.365217	<.001[a]	1.35784	3.16889

[a]The mean difference is very highly significant at the <0.001 level

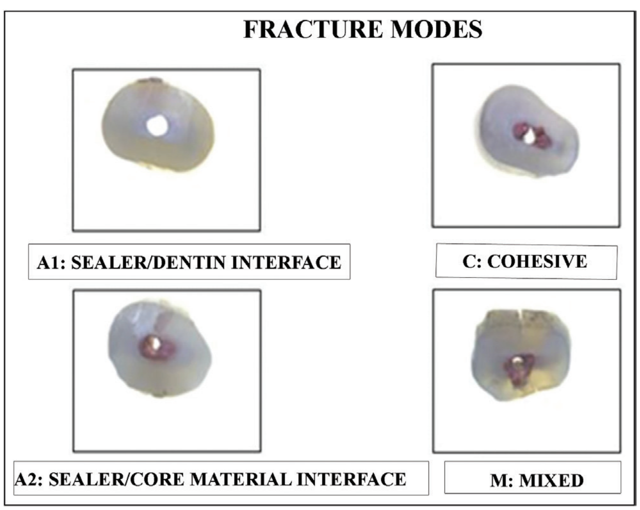

Figure 6: Fracture modes

suggest that AH Plus® in moist conditions shows the highest push-out bond strength as compared to Epiphany and GuttaFlow. These findings are in agreement with those obtained by *Nagas et al.*,[1] who compared dentin moisture conditions and stated that keeping the dentin slightly moist also provides an optimal substrate for the tested sealers. The gutta-percha and AH Plus® sealers showed the highest bond strength due to the formation of a covalent bond by an open epoxide ring to any exposed amino groups in collagen. However, AH Plus® and Epiphany sealers show the lowest bond strength under wet conditions because hydrophilicity of AH Plus® sealer is never sufficient to displace water in a totally wet root canal, and the resultant entrapment of water droplets between the sealer dentin interface leads to disruption of the bond.[7-9] The resin-based sealer, that is, Epiphany, in wet moisture conditions would also decrease the monomer conversion, leading to incomplete resin polymerization and hence a decrease in bond strength.[1] Other researchers also concluded that moisture in dentin surfaces represents a critical variable during bonding procedures.[10-12]

Similar researches concluded that silicon-based root canal sealer (GuttaFlow) shows the least bond strength due to expanding capacity of GuttaFlow and the partial dissolution of the sealer.[13,14]

In various studies, Resilon was compared with other sealers using a fluid filtration model. Results showed less microleakage with Resilon and Epiphany sealer. Lesser microleakage with Resilon/Epiphany was because of monoblock that was created as opposed to AH Plus® and gutta-percha.[15-17]

Dias et al. concluded that canals dried with 70% isopropyl alcohol showed significantly higher bond strength values than those with paper points. Considering the hydrophilic propensity of the resin-based sealers, it may be speculated that isopropyl alcohol (C_3H_7OH), which has lower polarity than ethanol (C_2H_5OH), promoted less removal of the water from dentinal tubules, enhancing the dentin wettability, increasing the degree of conversion of the sealers, and consequently improving their adhesion.[16] *De-Deus et al.* showed that AH Plus® and gutta-percha root fillings have significantly higher push-out bond strength than Resilon/Epiphany and Resilon/Epiphany SE as the methacrylate-based materials undergo volumetric shrinkage during polymerisation, and shrinkage of adhesive sealer is incompatible with optimal bonding condition to root dentin.[18]

Paula et al.[19] carried out a study to show the effect of drying protocol on the push-out bond strength. AH Plus® showed the highest bond strength values, which is in agreement with our study.

Donnermeyer et al. carried out a study to show the effect of different irrigation solutions on the push-out bond strength of three different sealers, such as AH Plus®, BioRoot RCS, and GuttaFlow2. AH Plus® revealed significantly higher bond strength than other sealers. The push-out bond strength of GutttaFlow2 was not affected by the irrigation protocol.[20]

Phukan et al. carried out an *in-vitro* study with different sealers and evaluated the fracture resistance of the endodontically treated teeth. They concluded that to obtain a better adhesion with root canal and to obtain a good secondary monoblock, AH Plus® should be used as it shows better adhesion than most of the other root canal sealers, which is in agreement with our study.[21]

Donnermeyer et al. carried out a study on the comparison of push-out bond strength of calcium silicate-based endodontic sealers with an epoxy resin-based sealer (AH Plus®). They concluded that the push-out bond strength of the investigated calcium silicate-based sealers was lower than that of AH Plus®.[22]

The results of this study showed that the mode of failure for all sealers (AH Plus®, Epiphany, and GuttaFlow) was adhesive failure (84%). The adhesive failure mainly between sealer and dentin (61%) was observed under dry, moist, and wet conditions. The adhesive failure between sealer and main core (39%) was observed under normal moisture conditions.

According to *Nagas et al.*, the predominant fracture mode was adhesive failure along the sealer/core material interface under normal moisture and moist conditions. On the contrary, the failure was adhesive between sealer/dentin interface under dry and wet conditions, suggesting an inadequate level of adhesion between sealer and dentin in terms of bond strength.[23] GuttaFlow2 showed adhesive failure mainly at sealer dentin interface due to lack of chemical union between sealer and dentin, which is in agreement with the study of *Naser et al.*[24,25]

Ungor et al. made a comparison between different pairing of Epiphany–Resilon and AH Plus®–gutta-percha, and concluded mainly adhesive bond failure of sealer to dentin for all groups.[26]

The push-out bond strength of resin sealer was much lower when the sealer was present as a thin layer in combination with the main cone. Bulk sealer showed predominately adhesive failure, whereas the thin film of sealer showed cohesive failure.[8,9]

On the contrary, *Carneiro et al.* noted the predominated presence of adhesive failures in the specimens having Epiphany SE as sealer, whereas AH Plus® and Sealer 26 had mixed and cohesive failures more frequently, regardless of the root filling technique.[27]

Ushikubo carried out a similar study on the effect of different moisture conditions before filling procedure with Well–Pulp ST as root canal sealer on the push-out bond strength and concluded that the moist group showed higher bond strength than the dry and the wet groups when using Well–Pulp ST as root canal sealer. Under dry and moist conditions, the most common type of failure was adhesive failure along the sealer and gutta-percha interface. Under wet conditions, the majority of specimens showed adhesive failure along the sealer and dentin interface. The results are in agreement with our study.[28]

Hence, null hypothesis was rejected, because moisture has an effect on the bond strength of the sealer in our study and is corroborating previous research findings also. However, several aspects of bond strength of root canal sealers to root dentin need further research. The push-out bond strength test is based on geometric parameter of specimen, elastic moduli of dentin, and intra canal filling material. Therefore, different hybridization protocols of root dentin must be evaluated to increase the long-term adhesion and bond strength for root canal sealers.

Conclusion

Distinctive moisture conditions affect the push-out bond strength of the sealers. The best moisture condition for all three root canal sealers showing highest strength was observed for AH Plus® under moist condition, Epiphany under dry condition, and GuttaFlow under normal condition, respectively.

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Conflicts of interest

There are no conflicts of interest.

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