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Original Article

Comparative Evaluation of Push-Out Bond Strength of Glass Fiber Post to Root Canal Dentin Using Different Adhesive Resin Cement - An *ex vivo* Study

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Abstract

Introduction: Adhesive resin cements are currently the most accepted cements for glass fiber post (GFP) cementation. However, the cementation procedure is sensitive due to multiple operatory steps. The bonding effectiveness of resin cements are thus controversial, needing further investigation.

Purpose: To compare the influence of different adhesive resin cements on the push-out bond strength of GFP to root canal dentin and to evaluate the modes of failure using different types of adhesive resin cement using Scanning electron microscope analysis.

Materials and Methods: Eighty freshly extracted mandibular pre-molars were divided into four groups (n = 20). Rotary instrumentation and obturation were done with single cone gutta-percha and AH Plus sealer. Eighty GFPs (4 groups; n = 20) were initially surface treated with tribochemical silica sandblasting and were then luted to the prepared post-space using four different types of adhesive resin cement (Multilink N [Group I], Speed CEM Plus [Group II], Variolink N [Group III] and RelyXU200 [Group IV]), respectively, in the experimental groups. Coronal, middle, and apical levels obtained after sectioning the roots embedded in a resin mold were subjected to a push-out BS test, and the "peak force" at bond failure was measured. The modes of failure between the interfaces were analyzed using Scanning Electron Microscopy. Statistical analysis of the groups was done and the statistical significance of the difference in the proportion of modes of failure between the four groups were measured.

Results: Group IV (RelyXU200) showed the highest push-out BS in the coronal, middle, and apical root levels, followed by Group II (SpeedCEM Plus).

Conclusion: RelyXU200 presented the highest push-out BS to glass-fiber posts and root dentin, followed by SpeedCEM Plus and Variolink N. Multilink N showed the least BS among the four groups.

Key words: Adhesive resin cement, Glass fiber post, Push-out bond strength, Sandblasting, Tribochemical silica coating

INTRODUCTION

The functional and esthetic rehabilitation of root canal-treated teeth presents a unique challenge to the endodontist. A tooth with considerable loss of coronal structure necessitates a post for core retention.[1,2] The traditional prefabricated metal posts were esthetically unacceptable for the endodontically treated anterior teeth. The rising esthetic concern led to the evolution of esthetic post-and-core systems, namely, epoxy or methacrylate resin with quartz glass or carbon fibers, polyethylene fiber-reinforced posts, and zirconia posts. Due to translucency and a more comparable modulus of elasticity (MOE) to root dentin, their superior esthetic property offers a uniform stress distribution within the canal walls.[3]

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The success rate of fiber posts varies from 48% to 100% over the years.[4] The retention and stability of glass fiber posts (GFPs) attribute to the pre-treatment of the post-surface, choice of luting materials, and luting procedures.[5] Among the various categories of post-surface treatments available, the most consistent results were noticed using sandblasting with tribochemical silica followed by coating the post-surface with a Silane coupling agent.[6,7] Integration of adhesive technique for GFPs cementation into root canals created a "monoblock" type of restoration.[8] Adhesive resin cement is the currently most accepted type of cement for GFPs cementation. Self-adhesive (SA) resin cement with a dual adhesive monomer, 10-methacryloyloxydecyl dihydrogen phosphate (10-MDP), adhere chemically to the calcium atoms in the dentin, thereby demonstrating a durable chemical bond.[9] Still, controversies exist regarding its performance, with some studies showing opposing results.[10,11] The role of silane application on tribochemical silica-coated GFPs on the improvement of bond strength (BS) to the SA cement reveals inconsistent results.

The etch-and-rinse (ER) based dual-curing resin cement is the gold standard with which the Self-etch (SE) and SA resin cements are compared due to their superior BS values in research studies.[12] However, the inadequate visibility could complicate the cementation process using ER-based luting cement toward the root apex.[13] This varied observation necessitates a further investigation of the same. The null hypothesis stated that the type of adhesive cement used does not affect the push-out BS of GFP to root dentin at any root level.

MATERIALS AND METHODS

Specimen Preparation and Root Canal Instrumentation

The sample collection was done at the Department of conservative dentistry and Endodontics, Amrita School of Dentistry. Sample size calculation and specimen preparation were done at the Department of Biostatistics and the Department of Nanosciences, Amrita Institute of Medical Sciences and Research Centre, Edappally, Ernakulam. The study was conducted with the approval of the institutional ethical committee (IRB-AIMS-2019-157). The primary author performed all the laboratory procedures to avoid bias.

Eighty mandibular pre-molars extracted for orthodontic and periodontal purposes were used. An ultrasonic scaler was used to clean the debris on the specimens and then stored in distilled water for 7 days at room temperature. Diamond discs were used to section the natural crowns perpendicular to the long axis of the tooth, 2 mm above the cementoenamel junction (CEJ), to standardize root canal length to 14 mm [Figure 1]. After decoronation, the tooth samples were divided into four groups (n = 20). Instrumentation was performed at 1 mm short of the apex up to the rotary protaper F3 file of the Protaper universal system (Dentsply, Maillefer, Switzerland). After changing each rotary file, the canals were irrigated using a 1ml solution of sodium hypochlorite (5.25% NaOCl). The canals were finally rinsed and dried with distilled water and F3 paper points, followed by obturation with ProtaperF3 single cone and AH plus sealer. The canal orifices were sealed with a temporary filling and stored in distilled water for 2 days.[14]

Post-Space Preparation, Pre-Treatment, and Post-Luting Procedures

After storage, the temporary filling was removed. Post-space was prepared using peeso reamers from size #3 to # 1(Mani Dental, New Delhi, India), sparing 4mm of gutta-percha at the root apex.5.25% NaOCl and 17% Ethylenediaminetetraacetic acid (EDTA) were used for irrigation with a final rinse using distilled water. The root canals were dried using #25 paper points. All the post-surfaces were subjected to sandblasting procedure using silica-coated alumina (Cojet Sand, 3M ESPE, Seefeld, Germany) 50 μm size for 15 s at 2–3 bar pressure from 10 mm distance followed by application of silane coupling agent (Ultradent Products, Inc.USA) before post-cementation in the canal.

These posts were then luted to the prepared post-spaces using one of the four adhesive resin cement; Multilink N (Group I), Speed CEM Plus (Group II), Variolink N (Group III), and RelyXU200 (Group IV) in the respective groups following manufacturer's instructions [Figure 2]. After post-luting, these were stored in saline at 36.5°C for 24 h.

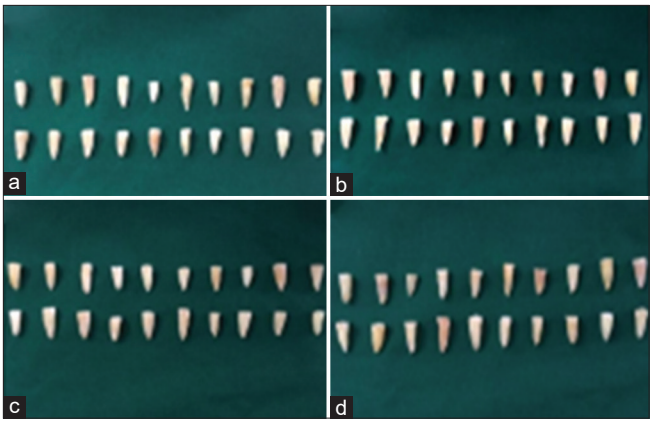

Figure 1: Mandibular single-rooted pre-molars after decoronation in each group; (a) Group I, (b) Group II, (c) Group III, (d) Group IV

Push-Out Test

The stored specimens were then fixed in a mold of acrylic resin [Figure 3a]. Three segments per tooth were sectioned perpendicular to the long axis starting 1mm below the CEJ using an Isomet device (Isomet, Buehler, USA) under sufficient water cooling [Figure 3b]. Thus, 1mm thick segments were obtained from the roots' coronal, middle, and apical levels [Figure 3c]. The push-out bond strength (PBS) of the root levels obtained was tested using a Universal testing machine (UTM) [Shimadzu Corporation AutoGraph AGS- X Series, Japan] at a crosshead speed of 0.5 mm/min [Figure 4a]. A customized jig was fabricated to attach within the UTM [Figure 4b]. The peak force applied for the post-segment to extrude out of the root was the point of bond failure and was recorded in Newtons (N).[14]

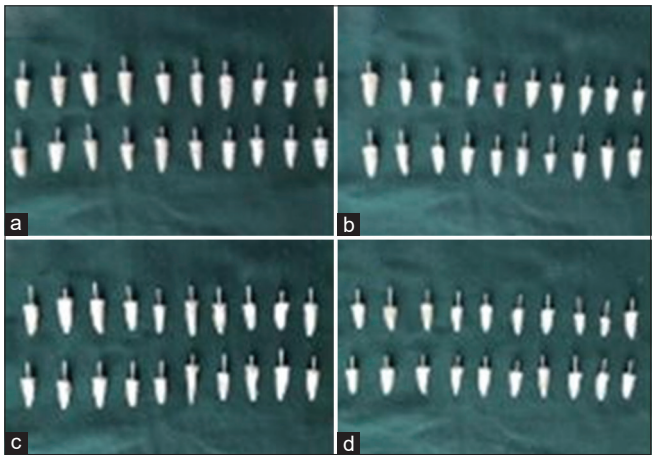

Figure 2: Specimen after post cementation with the respective adhesive resin cements. (a) Group I, (b) Group II, (c) Group III, (d) Group IV

Figure 3: Preparation of the specimen before push-out bond strength test; (a) Roots mounted in acrylic resin, (b) Sectioning of mounted specimen using isomet device, and (c) Sectioned specimens

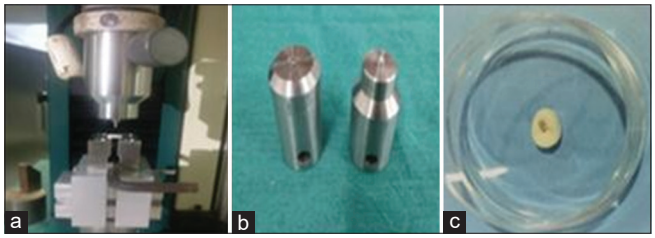

Figure 4: Armamentarium for Push-out bond strength test; (a) Push-out bond strength testing of the sectioned specimen with the customized jig mounted on a universal testing machine, (b) Customized jig used for mounting the specimens for push-out testing, and (c) Debonded root section ready for scanning electron microscopic analysis

This value was then changed to Mpa using the formula:

Debonding stress (MPa) = Debonding force (N)

Area (mm^2)

The area of the specimen was calculated using: $A = 2\pi rh$

Where,

π - is constant 3.14

r - Radius of the surface of the post-bonded to the canal

h - Thickness of the specimen (1 mm).

Out of the 20 sectioned specimens from the different root levels of each group after the PBS test, 10 representative samples from each root level of each group ($n = 30$) were then subjected to scanning electron microscopic (SEM) (Hitachi Science Systems, Japan Pvt Lmd) analysis to assess the various failure modes classified as Adhesive failure; failure either at the resin-dentin or resin-post interface. Cohesive failure; failure within the resin cement or dentin. Mixed failure; failure within the sealer, dentin, or at the interface [Figure 4c]. The specimens were sputter-coated with gold-palladium and examined under SEM at ×150 magnification[15] [Figure 5].

The data were evaluated using the IBM Statistical Package for the Social Sciences, Statistics for Windows, Version 28.0. Armonk, New York, USA (IBM Corp). To test the statistical significance of the difference in the mean, standard deviation, and median of PBS among four groups of resin cement at three different root levels Kruskal–Wallis test and *post hoc* Dunn-Bonferroni test were used. To test the statistical significance of the difference in the proportion of modes of failure between the four groups Chi-square test was used. The statistical significance was fixed at $P < 0.05$.

RESULTS

The mean, standard deviation, and median of the PBS values in each of the four groups compared between the coronal, middle, and apical root levels were found to be statistically significant ($P < 0.001$) for groups II, III, and IV [Table 1]. On comparing all groups, the group I showed the lowest PBS at all root levels. On comparing group II with others, group II showed higher PBS values at all the root levels than groups I and III, which was statistically significant ($P < 0.001$). However, group II showed comparatively lower BS values than group IV but was not statistically significant ($P > 0.001$) [Table 2].

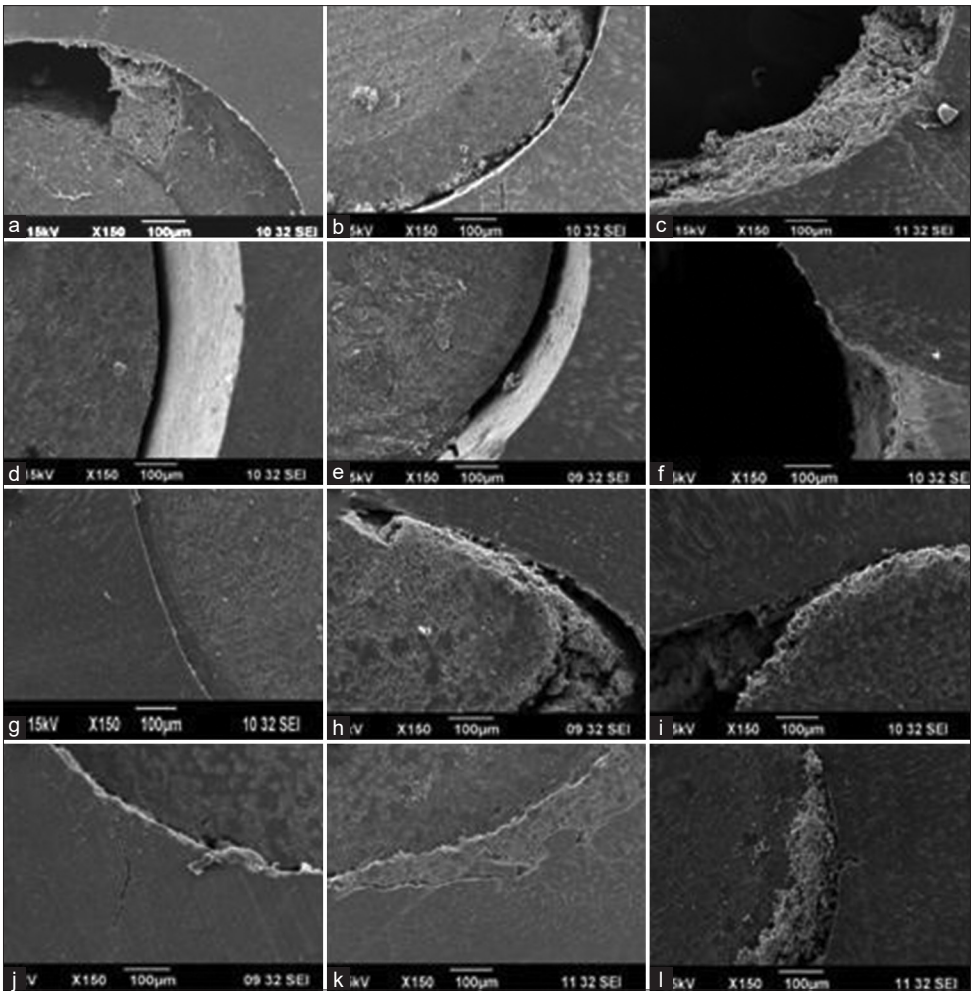

Figure 5: Scanning electron microscopic pictures of the adhesive interface; (a-c) Group I; (d-f) Group II; (g-i) Group III; (j-l) Group IV

Comparison of group III with groups I, II, and IV showed a significantly lower BS compared to group II and IV at all root levels, whereas a higher BS compared to group I. On comparing Group IV with the other groups, group IV showed the highest BS values, followed by group II. The least BS values were observed in group I compared to all the other three groups assessed [Table 2].

The test revealed the highest PBS values at the cervical root level, whereas the apical root level showed the lowest BS values in all groups, irrespective of the cement type. The BS values in the cervical and middle root levels were found to be statistically higher than the apical ones in all four groups ($P < 0.001$) [Tables 1 and 3]. The chi-square test was applied to identify the most prevalent mode of failure among the four groups and found that mixed mode of failure was the most predominant in group I (66.7%), adhesive failures were predominant among group II (40%), and group III (40%) while group IV showed predominance for cohesive failures (76.7%) [Table 4].

DISCUSSION

A clinical challenge is finding the ideal resin luting agent for GFPs cementation within a root canal, which simulates a monoblock. The adhesion of the resin cement to GFPs and root dentin can be measured through the BS between the three components. In the present *ex vivo* study, single-rooted mandibular pre-molars were selected to simulate clinical conditions.[16] However, human teeth represented relatively large variations in morphology size and mechanical properties.[17] This limitation was overcome by standardizing the working length of selected teeth, endodontic treatment and post-space preparation with similar dimensions throughout the study. Angelus reforpost GFPs were chosen as these are parallel, serrated intraradicular posts with a conical tip that provides good retention and excellent adhesion to resin cement and root dentin. They exhibit good esthetic properties, flexural and fatigue strength, MOE comparable to that of dentin, easy to handle and relatively cheap.[18]

Table 1: Comparison of median push-out bond strength among three different root levels of four groups of resin cements

Root levels	Groups	Sample size (*n*)	Mean and standard deviation	Median (Q3, Q1)	*P*-value
C	Group I	20	1.20±0.21	1.15 (1.34,1.06)	0.153
M		20	1.29±0.30	1.27 (1.48,1.09)	
A		20	1.36±0.30	1.38 (1.58,1.18)	
C	Group II	20	11.67±1.08	11.73 (12.32,10.91)	<0.001
M		20	10.24±2.30	10.44 (11.07,9.87)	
A		20	7.92±2.65	7.74 (9.58,6.33)	
C	Group III	20	5.52±0.40	5.62 (5.92,5.23)	0.015
M		20	5.24±0.65	5.23 (5.76,4.69)	
A		20	4.94±0.76	4.97 (5.44,4.33)	
C	Group IV	20	13.07±1.05	13.04 (13.48,12.78)	0.014
M		20	13.45±1.71	13.49 (14.71,12.04)	
A		20	11.75±1.92	11.63 (13.52,10.56)	

Table 2: Comparison of median push-out bond strength among four groups of resin cements at three different root levels

Root levels	Groups	Sample size (*n*)	Mean and standard deviation	Median (Q3, Q1)	*P*-Value
C	Group I	20	1.20±0.21	1.15 (1.34,1.06)	<0.001
	Group II	20	11.67±1.08	11.73 (12.32,10.91)	
	Group III	20	5.52±0.40	5.62 (5.92,5.23)	
	Group IV	20	13.07±1.05	13.04 (13.48,12.78)	
M	Group I	20	1.29±0.30	1.27 (1.48,1.09)	<0.001
	Group II	20	10.24±2.30	10.44 (11.07,9.87)	
	Group III	20	5.24±0.65	5.23 (5.76,4.69)	
	Group IV	20	13.45±1.71	13.49 (14.71,12.04)	
A	Group I	20	1.36±0.30	1.38 (1.58,1.18)	<0.001
	Group II	20	7.92±2.65	7.74 (9.58,6.33)	
	Group III	20	4.94±0.76	4.97 (5.44,4.33)	
	Group IV	20	11.75±1.92	11.63 (13.52,10.56)	

Table 3: Pairwise comparison of push-out bond strength of four groups at the coronal, middle, and apical root levels of tooth samples

Root levels					
Coronal		Middle		Apical	
Group A-Group B	*P*-value	Group A-Group B	*P*-value	Group A-Group B	*P*-value
Group 1-Group 3	0.039	Group 1-Group 3	0.026	Group 1-Group 3	0.010
Group 1-Group 2	<0.001	Group 1-Group 2	0.000	Group 1-Group 2	<0.001
Group 1-Group 4	<0.001	Group 1-Group 4	0.000	Group 1-Group 4	<0.001
Group 3-Group 2	0.012	Group 3-Group 2	0.041	Group 3-Group 2	0.155
Group 3-Group 4	<0.001	Group 3-Group 4	0.041	Group 3-Group 4	<0.001
Group 2-Group 4	0.273	Group 2-Group 4	0.115	Group 2-Group 4	0.093

Table 4: Comparison of the modes of failures among the four groups

Group	Modes of failure			*P*-value
	Adhesive *n* (%)	Mixed *n* (%)	Cohesive *n* (%)	
Group I (30)	6 (20)	20 (66.7)	4 (13.3)	<0.001
Group II (30)	12 (40)	10 (33.3)	8 (26.7)	
Group III (30)	12 (40)	10 (33.3)	8 (26.7)	
Group IV (30)	0 (0)	7 (23.3)	23 (76.7)	

The irrigation protocol used during post-space preparation involves an initial 5.25% NaOCl irrigation for 5 min, followed by 17% EDTA for 1 min, and distilled water was used as the final irrigant. Earlier research had observed an effective smear layer removal when EDTA was combined with NaOCl for canal irrigation.[14] One-minute irrigation using 17% EDTA establishes effective elimination of the smear layer. In a study by Arisu *et al.* based on post-surface treatments on PBS of GFPs, it was concluded

that SE-based resin cement systems significantly improved dentin BS when the canal was irrigated with a combination of NaOCl and EDTA.[19]

All the GFPs were surface treated with Cojet Sand (3MESPE, Seefeld, Germany) at 2–3 bar for 15 s from a 10 mm distance, followed by application of silane for 60 s and drying with a gentle air spray(Ultradent Products, Inc.USA). Tribochemistry creates chemical adhesion by applying kinetic energy. The CoJet system uses micro-blasting sand, especially silica-modified aluminum trioxide, and provides the post-surface with an active silica-rich outer surface prone to salinization.[20] In a recent review by Mishra *et al.* that studied the outcome of pre-treatment of fiber posts with silane on the BS to dentin, it was concluded that silane alone could not enhance the BS of GFPs and resin cement to dentin.[6] The push-out test was chosen in the present study as it is considered the most reliable for measuring the GFPs BS to resin cement and root dentin as this test can better simulate a clinical scenario.[21] Resin-luting cement has been widely used along with GFPs to provide adhesive cementation with better retention and lower solubility than conventional luting cement.[22]

It improves marginal adaption creating an apical seal,[23] and presents favorable fracture patterns in situations of excessive masticatory load. The present study evaluated and compared the PBS of four different resin cements to the GFPs and root dentin. The respective types of cement include Multilink N (Group I), Speed CEM Plus (Group II), Variolink N (Group III), and RelyXU200 (Group IV). The results from the present study rejected the null hypothesis that the adhesive resin cement does not affect the PBS of GFPs to root dentin at three different root levels. Considering different root levels, the coronal root level presented the highest BS, followed by the middle in all four groups regardless of the resin cement. This might be attributed to several reasons, such as the density of the canaliculi, area of the dentinal tubules,[24] type and chemical composition,[25] difficulty of inadequate flow of the cement toward the apical root level,[26] poor access to curing light toward the apex during the photoactivation of the cement[27] and difficulty in controlling humidity leading to incomplete cement penetration.[28]

Among the cements, it was observed that the coronal and middle root levels of group IV (RelyXU200) exhibited the highest PBS. This was followed by group II (Speed CEMPlus). This is attributed to 10-MDP, a functional monomer incorporated in both RelyXU200 and SpeedCEMPlus resin cement. The monomer bonds chemically to hydroxyapatite of enamel and dentin to form stable calcium monomers resistant to hydrolysis and show more substantial adhesion capabilities.[29] The number of inorganic particles in SA cement is more than in conventional cement, thus lowering the cement polymerization shrinkage and increasing stability. The results from the present study were supported by studies of Aleisa *et al.* and da Silveira-Pedrosa *et al.*,[30,31] who observed that SA cement had better results by comparing BS rates with those presented by resin cement based on ER or SE adhesive approach.[32]

Among group I (Multilink N) and group III (Variolink N), group III showed comparatively higher BS at all root levels. The same was attributed to the adhesive approach considered where the group I employed a SE approach, presenting inferior BS results compared to an ER approach. Group I (MultilinkN) was universal adhesive resin cement used in a SE mode for the present study. The lowest BS in this group can be related to the effect of the type of irrigant solution used for canal disinfection.[33] Following the preparation of post-space, the smear layer observed along the root canal wall is quite thick, and it can be assumed that functional monomers in the mild SE mode might not be able to etch deeply enough into the existing smear layer without any additional pre-treatment of the dentin.[34] Consequently, the adhesive might not establish a sufficient bond with the underlying dentin, suggesting compromised bonding efficacy.[11]

The SEM analysis of the present study revealed a predominantly mixed mode of failure for Multilink N (group I). This finding is not supported by any other studies that include this cement. Here, the cement interface combines cohesive and adhesive failure.[35] A mixed mode of failure for the cement may be attributed to the incomplete solvent evaporation in the deeper canal region and failure to remove excess adhesive from the canal walls.[36] A predominance of adhesive failures was observed for both SpeedCEM Plus (group II) and Variolink N (group III). In SpeedCEM Plus, most bond failures occurred between the GFPs and the resin cement. Whereas with Variolink N, failure mainly occurred between root dentin and the resin cement. This could be attributed either to the non-uniform and increased cement thickness around the post that can negatively influence the BS or the polymerization stress seen at the interface between dentin and the resin cement.[37] Group IV showed predominance for cohesive failure. Cohesive failures are an acceptable mode of failure as it suggests that the adhesive resin cement had a firmer grip on the bonding substrate than itself, showing a high degree of surface adhesion.[35]

However, the present study failed to conduct thermo-mechanical ageing of the specimens. Therefore, the results obtained cannot be considered a complete clinical simulation. RelyXU200 and SpeedCEM plus,

SA resin cement incorporated with 10-MDP functional monomer, produce superior BS to GFPs pre-treated with a combination of tribochemical silica sandblasting and silane application. RelyXU200 and SpeedCEM Plus showed adequate BS even at the apical level of the root. Hence, this study opens an arena for more studies to explore the influence of applying pre-activated silane coupling agents over GFPs to improve its BS and adhesion to resin cement and root dentin. Further studies can be conducted on the effect of different root conditioning agents to improve the BS of SA resin cement to root dentin.

CONCLUSION

Within the limitation of the present study, it was observed that the adhesive resin cement tested significantly influenced the push-out BS of GFPs to root dentin at three different root levels. RelyXU200, 10-MDP-based dual-cure SA resin cement presented the highest push-out BS to GFPs and root dentin, followed by SpeedCEM Plus and Variolink N resin cement. Push-out BS was the highest at the coronal root level, followed by the middle for all four groups, irrespective of the resin cement used. Regarding the assessment of the predominant mode of failure between the resin cement by SEM analysis, SpeedCEM Plus (Group II) and Variolink N (Group III) showed an adhesive mode, RelyXU200 (Group IV), cohesive failure and Multilink N (Group I) showed a mixed mode of failure.

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