

Bond Strength Evaluation of Different Adhesive Systems Applied with Passive and Active Manual Application Mode in Endodontically Treated Teeth (An *in-vitro* Study)

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Abstract

Objective: this study was designed to evaluate the adhesive systems application mode in bond strength and degree of conversion after cementation of fiber glass post within the root canal dentin.

Materials and methods: the roots of 44 premolars were undergone root canal treatment and then divided into four subgroups (n=11), according to the mode of adhesive application (manual active, manual passive) and the cementation system (Adper Single Bond 2/RelyX ARC in etch-and-rinse strategy and Single Bond Universal/RelyX Ultimate in self-etch strategy). In each group, seven roots were randomly evaluated by push-out Bond Strength test; four roots were evaluated in micro-Raman spectroscopy for Degree of Conversion.

Results: For both cementation systems, the highest Bond Strength values (MPa) were observed with manual active application mode and Single Bond Universal/RelyX Ultimate in self-etch strategy performed better than other type.

Conclusion: The highest bond strength value was observed to the active application mode especially with Single Bond Universal/RelyX Ultimate in self-etch strategy at the coronal root third.

Keywords: Luting cement; Fiber glass post; Mode of application; Adhesive strategy

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Introduction

After finishing the root canal treatment, the restoration support and retention can be gained through using the fiber glass post. The modulus of elasticity of fiber post is close to that of dentin [1]. This can provides distribution of applied forces and decreases the percentage of failure in addition to enhancement of good bonding between resin cement and tooth substrate [2,3]. Evaluation of the bond strength values revealed the weakness of adhesion between the resin cement and dentin within root canal when compared to adhesion between the resin cement and fiber glass post [4]. This may be attributed to histo-morphological changes along root canal length. Coronal third has density of tubular system better than middle third which is also better than apical one. This is explained different bond strength values along the root length [5,6]. At the apical third, accumulation of endodontic debris and presence of high amount of smear layer lead to lower bond strength values. This is in contrast with area of coronal

third in root canal [7]. For attempting to solve the problems lead to fiber post poor adhesion, several strategies have been mentioned in the literatures to decrease failure rates, in order to gain a better performance of adhesive system. Large numbers of literatures have revealed better values of bond strength when fiber glass post application was in active manual mode compared to passive manual one [8]. Others was in contrast. This might be attributed to deeper monomer penetration to dentin substrate along root length and better solvent evaporation in case of active manual mode [9]. The purpose of the study of was to evaluate the effect of mechanical cycling and cementation strategies on the push-out bond strength between fiber posts and root dentin and the polymerization stresses produced using three resin cements. Results recorded that mechanical cycling did not affect the bond

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strength values ($p=0.236$), while cementation strategies affected the push-out bond strength ($p<0.001$). Luting with RelyX U100 and Scotch Bond Multi-Purpose + RelyX ARC yielded higher push-out bond strength values. The polymerization stress results were affected by the factor "cement" ($p=0.0104$): the self-adhesive cement RelyX U100 exhibited the lowest values, RelyX ARC resulted in the highest values, while Multilink Automix presented values statistically similar to the other two cements [10]. Another valuable study was the study of Pereira et al., the aim of the study was to assess the dentine/fibreglass-bonded interface by scanning electron microscopy and the push-out bond strength of four luting cements. Results revealed that the bond strength was significantly affected by the type of cement ($P<0.001$) and by the longitudinal region of the root canal ($P<0.031$). All cements exhibited gaps at the dentine/cement interface [11]. The present study aimed to assist the bond strength and degree of conversion of the different current adhesive systems applied in the dentin of root canal, with passive and active manual mode of applications.

Material and Methods

A total number of forty four human uni-radicular premolar teeth were selected for this study. The selected teeth were caries free, did not undergo a previous root canal treatment, have no dilacerations at radicular area and with root length not less than 15 mm. Using a low-speed diamond saw (ISOMET 1000, Buehler) under cooling system, teeth were sectioned at cement-enamel junction at transverse direction. Root canal treatment was done for each tooth with white K file (no. 15) till reached black one (no. 40). Then technique of crown down was performed until K file (no. 55). Copious irrigation between successive file was mandatory. Sodium hypochlorite was the irrigant solution in concentration of 1 percent. Then normal saline was used to clean the canals. Canal dryness was performed with paper points. Warm vertical condensation technique was used for sealing 5 mm of apical area. For confirmation, periapical x-ray films were taken to ensure there is no excess of gutta-percha or sealer. Glass ionomer cement (vitrebond) was used as temporary closure for canal orifices. Teeth were then immersed in distilled water and stored in an incubator (37 CO) until preparing the next step. Following the manufacturer instructions (FGM), spaces of fiber glass posts (White Post DC #2) were prepared with a special corresponding drill mounted on low speed hand-piece.

Grouping of specimens

Specimens were divided according the type of cementation system into two main groups ($n=22$).

Group one: Using Adper Single Bond2 (etch & rinse adhesive system, 3M ESPE) with resin cement Relyx ARC (3M ESPE).

Group two: Using Single Bond Universal (self-etch adhesive system, 3M ESPE) with resin cement RelyX Ultimate (3M ESPE). Materials used in this study were collected in **Table 1**.

Manufacturer's instructions of Adhesive Adper Single Bond 2

Etching: Apply etchant to the prepared tooth. Wait fifteen seconds. Rinse for ten seconds. Dry for two seconds. Remove excess moisture with an absorbent paper point.

Adhesive: Apply a uniform coat to etched enamel and dentin. Remove excess pooled adhesive with absorbent paper point. Dry for five seconds. Light cure for ten seconds.

Manufacturer's instructions of Resin cement Relyx ARC

Dispense appropriate amount of cement onto a mixing pad and mix for ten seconds. Apply cement to the bonding surface of the preparation in and around canal using an instrument such as spiral paste filler. Place a thin layer of mixed cement on post. Seat and hold the post in place. Begin cleanup of excess cement approximately 3-5 minutes after seating. Light cure for forty seconds from the occlusal surface to allow immediate placement of core buildup material.

Manufacturer's instructions of adhesive single bond universal

Clean the post with alcohol and dry it with water-free and oil-free air. Use a disposable applicator to apply Single bond Universal Adhesive over the entire surface to be luted, rub in for twenty seconds before blowing a gentle stream of air over the liquid for about five seconds. Light cure for ten seconds.

Manufacturer's instructions of resin cement relyx ultimate

Attach an Endo Tip to the mixing tip "Wide" for application in the root canal. Insert the Endo Tip as deeply as possible in the root canal and apply RelyX Ultimate, beginning apically. Keep the tip of the Endo Tip immersed in the cement and slowly move the Endo Tip upwards as the level of the paste rises. Do not remove the Endo Tip from the cement until the root canal has been completely filled. This so-called immersion filling should be carried out slowly and take at least five seconds to minimize the inclusion of air bubbles. Place the post in the root canal filled with cement; apply moderate pressure to hold it in position. Rotate the post slightly during insertion to avoid the inclusion of air bubbles. Leaving sufficient excess permits the removal of the uncured layer during shaping/polishing without leaving behind a deficit. The exposure time depends on the translucency of the post being used; for RelyX Fiber Post, it is forty seconds.

Each group was further subdivided according to technique of application of adhesive system into two subgroups ($n=11$) either manual passive or manual active application modes. According to manufacturer instructions, a micro-brush was used for spreading of adhesives within the internal walls of canals without agitation or any pressure. This is regarding the passive application mode. This is in opposite to active application mode of adhesives where a pressure was exerted on the micro-brush during adhesive spreading within the internal walls of root canal. Using insulin syringe, the resin cement was dispensed into the internal walls of canal. Steps of cementation of fiber post was achieved following the manufacturer's instructions with different techniques of application as mentioned before. A led light cure device was used for steps of resin cement and adhesives curing. Specimens were kept for a week at room temperature in distilled water, then in an acrylic resin tubes were embedded. Slices of nearly one mm were

Table 1 Materials used in this study.

Material trade Name	Category	Composition	Batch No.	Manufacturer
Phosphoric Acid	Acid etch	37% phosphoric acid	4762A2	3M ESPE Dental Product St. Paul, MN,USA Web site:www.3mespee.com
Adhesive Adper Single Bond 2 /	Etch-and-rinse adhesive system	BisGMA, HEMA, dimethacrylates, ethanol, water, a novel photoinitiator system and a methacrylate functional copolymer of polyacrylic and polyitaconic acids	4763 B	3M ESPE Dental Product St. Paul, MN,USA Web site: www.3mespee.com
Adhesive Single Bond Universal	self-etching adhesive system	Hydrophobic dimethacrylate, Phosphorylated methacrylates, TEGDMA, Silane- treated silica filler, Initiators, Stabilizers	166926B	3M ESPE Dental Product St. Paul, MN,USA Web site: www.3mespee.com
Resin cement Relyx ARC	Resin cement	PASTE A: Bis-GMA, TEGDMA, zircon and silica inorganic fillers (68% wt), photoinitiators, amines and pigments. PASTE B: Silane treated ceramic, copolymer acrylic and itaconic acid, water, HEMA.	R04407	3M ESPE Dental Product St. Paul, MN,USA Web site: www.3mespee.com
Resin cement RelyXUltimate	Resin cement	Base paste: Silane-treated glass powder, 2-propenoic acid, 2-methyl-, reaction products with 2-hydroxy-1,3-propan edyl dimethacrylate and phosphorus oxide, TEGDMA, silane-treated silica, oxide glass chemicals, sodium persulfate, tertbutyl peroxy3,5,5-trimethyl hexanoate, copper acetate monohydrate Catalyst paste: Silane-treated glass powder, substituted dimethacrylate, 1,12-dodecane dimethacrylate, silane-treated silica, 1-benzyl-5-phentyl-ba rbic-acid, calcium salt, sodium p-toluenesulfinate, 2-propenic acid, 2-methyl-, di-2,1-ethanediy ester, calcium hydroxide, titanium dioxide	R265388	3M ESPE Dental Product St. Paul, MN,USA Web site: www.3mespee.com

produced after sectioning of specimens in transverse direction by a low-speed diamond saw (ISOMET 1000). Sectioning was done under cooling system. A total number of six pieces or slices for each root surface were prepared. Two pieces for coronal portion, two pieces for middle third and the same for apical portion.

Bond strength was measured for seven out of total eleven teeth for each subgroup (n=11) while the remaining four teeth undergo examination for degree of conversion. For evaluation of bond strength, LLOYD Instron universal testing machine was used at a load of 40 kg and a crosshead speed of 0.4 mm/min. push-out test was achieved by applying the load from apical to coronal direction till displacement of glass fiber post (**Figure 1a and 1b**). Data of bond strength values was measured in Mega Pascal, kept and analyzed to reveal different values. For evaluation of degree of conversion at the area of hybrid layer, The Micro-Raman Spectroscopy was used for that purpose by the guidance of the study of Pulido CA et al. [12]. Using a specific equation, the data of degree of conversion of the adhesive system was calculated. Values of bond strength and degree of conversion were inserted into SPSS® software, examined and analyzed by three way ANOVA and Tukeys student post-hoc test ($\alpha = 0.05$).

Results

Evaluation of bond strength values by three way ANOVA test revealed that there was no statistical significant difference among the triple interaction (comparing application technique, adhesive systems and root regions) ($p=0.9$) but the double interaction that comparing the technique of application either to

system of adhesives or to root thirds were statistically significant ($p=0.04$). High bond strength values was observed when a manual active mode of application of resin cement was applied at the coronal root third ($p<0.001$). This was regarding the double interaction comparing different adhesive\cementation application techniques when applied to different root thirds. So better results was noted when manual active mode of application was used especially at coronal third regardless the type of adhesive\cementation was used. In relation to the double interaction between the type of cement used with the type of technique of application mode used, it was detected that also high bond strength values was recorded with manual active mode of application for both types of adhesive\resin cements. But generally, Single Bond Universal adhesive combined to RelyX Ultimate have a very good performance with self-etch strategy than Adper Single bond 2 combined to RelyX ARC with conventional etch-and-rinse strategy. These results were tabulated in **Tables 2 and 3** respectively.

Assessment of degree of conversion by three way ANOVA revealed similar results like evaluation of bond strength values. There was no statistical significant difference among the triple interaction (comparing application technique, adhesive systems and root regions) ($p=0.321$) but the double interaction that comparing the technique of application either to system of adhesives ($p=0.019$) or to root thirds ($p=0.0019$) were statistically significant. Higher Degree of conversion values was observed when a manual active mode of application of resin cement was applied at the coronal root third ($p<0.001$) followed by middle

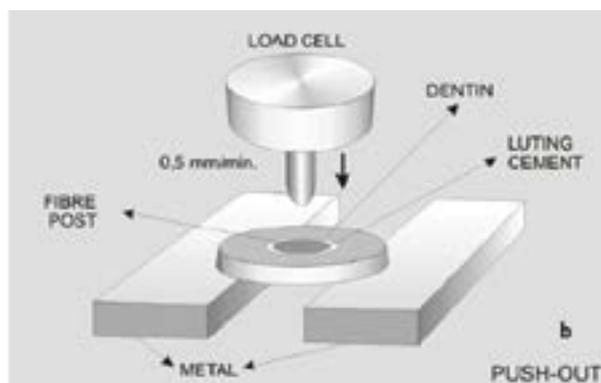
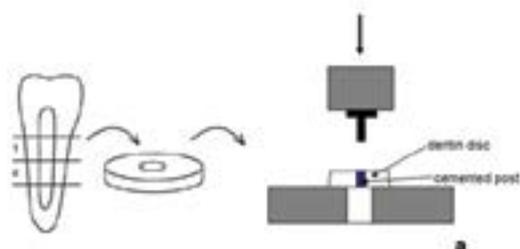


Figure 1 Represent the consequences of the Push out measuring method of this study (a & b).

third and the lowest values were observed at apical third. This was regarding the double interaction comparing different adhesive\cementation application techniques when applied to different root thirds. So better results was noted when manual active mode of application was used especially at coronal third regardless the type of adhesive\cementation system. In relation to the double interaction between the type of cement used with the type of technique of application mode used, it was detected that also high values of degree of conversion was recorded with manual active mode of application for both types of adhesive\resin cements. But generally, Single Bond Universal adhesive combined to RelyX Ultimate have a very good performance with self-etch strategy than Adper Single bond 2 combined to RelyX ARC with conventional etch-and-rinse strategy. These results were tabulated in **Tables 4 and 5** respectively.

Discussion

The results of this study revealed that active manual mode of application of adhesive\cement showed the highest bond strength values regardless of type of adhesive\cement used. This is agreeing with the study of Plotino et al. [13]. that evaluated and compared different methods of adhesive\cement application techniques. They demonstrated that adhesive agitation into the substrates of root canal accelerates evaporation of adhesive solvent that promotes formation of better hybridization through good penetration of smear layer in case of self-etch adhesive [14,15]. It is known that several reasons might contribute in determining the bond strength values and adhesive performance at different regions of root canals. Skills, experience and knowledge of the operator, type of adhesive\cement used, morphology of root canals and there cleaning and shaping all are factors control the performance [16,17]. Also, solvent evaporation play an important role in good adhesive performance at this region.

At this study, two types of adhesive systems were used. Adper Single Bond adhesive with etch-and-rinse strategy and Single Bond Universal adhesive with self-etch strategy. All higher bond strength values were recorded with Single Bond Universal adhesive with self-etch strategy regardless location of root thirds. This was explained by technique sensitivity of etch-and-

rinse strategy with subsequent over-dryness of dentin within root canals that would promote collapse of collagen fibrils. So improper full penetration of adhesive monomers would occur

Table 2 Mean bond strength values (MPa) and standard deviation with double interaction comparing root thirds and mode of application.

Root Thirds	Mode of Application	
	Active Manual	Passive Manual
Coronal Third	18.4 ± 3.7	13.3 ± 1.4
Middle Third	14.2 ± 1.6	10.1 ± 0.2
Apical Third	10.6 ± 1.4	07.5 ± 0.8

Table 3 Mean bond strength values (MPa) and standard deviation with double interaction comparing system of post cementation and Mode of application.

System of post cementation	Mode of application	
	Active manual	Passive manual
Adper Single Bond /RelyX ARC	17.2 ± 04.0	09.2 ± 01.7
Single Bond Universal/ RelyX Ultimate	21.3 ± 04.3	12.3 ± 02.9

Table 4 Mean degree of conversion values (%) and standard deviation with double interaction comparing root thirds and mode of application.

Root thirds	Mode of application	
	Active manual	Passive manual
Coronal third	94.7 ± 1.5	89.1 ± 1.7
Middle third	94.2 ± 1.1	88.3 ± 4.6
Apical third	90.7 ± 1.8	87.7 ± 4.8

Table 5 Mean degree of conversion values (%) and standard deviation with double interaction comparing system of post cementation and Mode of application.

System of post cementation	Mode of application	
	Active manual	Passive manual
Adper Single Bond / RelyX ARC	93.0 ± 0.9	92.3 ± 2.5
Single Bond Universal/ RelyX Ultimate	93.4 ± 3.5	87.1 ± 1.8

and poor hybridization will be the result. In addition, over-wet of dentin within root canals would promote chemical dilution and water globule formation. Both over-dry and over-wet phenomena are disadvantages of etch-and-rinse strategy [18]. On the other hand, self-etch adhesives contain water in their composition and control the moisture within the layer of hybridization [19]. All these previously mentioned facts may explain the better bond strength results obtained by Single Bond Universal adhesive (the self-etch strategy) in root dentin, compared to Adper Single bond with (conventional etch-and-rinse strategy). This is in agree with the study of Bergoli et al. They concluded that the self-adhesive cement appeared to be a good alternative for luting fiber posts due to the high push-out bond strengths and lower polymerization stress values [10]. Also, the results of the study of Pereira et al. augmented the results of the present study. They concluded that the self-adhesive materials and the glass ionomer cements had the highest push-out bond strength values when compared with the dual-curing resin cement [11].

Results of this study revealed that there was no statistical

significant differences among different groups regarding the degree of conversion. Passive technique of application combined with Adper Single Bond adhesive (etch-and-rinse strategy) recorded the lowest values of degree of conversion while best results recorded with Single Bond Universal adhesive (the self-etch strategy). This is agreeing with the results reported in other studies that investigated the degree of conversion within the hybrid layer in root canal [19,20].

Conclusion

With the limitation of this study, it may be concluded that the active manual application techniques of the adhesive systems during cementation of fiber glass post within root canal region should be the first choice for operators to get better results of bond strength. Single Bond Universal adhesive combined to RelyX Ultimate have a very good performance with self-etch strategy than Adper Single bond 2 combined to RelyX ARC with conventional etch-and-rinse strategy.

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