### **Original Article**

## Effect of Chlorhexidine and Benzalkonium Chloride on the Long-term Push-out Bond Strength of Fiber Posts

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Received: 29-Jun-2022; Revision: 29-Mar-2023; Accepted: 09-Aug-2023; Published: 21-Sep-2023

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Background and Aim: Fiber posts are widely used in endodontically treated teeth with extensive loss of coronal structure. The purpose of this study was to investigate immediate and the long-term effects of chlorhexidine (CHX) and benzalkonium chloride (BAC) application, on the push-out bond strength of fiber posts. Material and Methods: Sixty mandibular premolars were decoronated, and root canal treatment was performed. After post space preparation, the specimens were divided into three groups according to the post space-surface pretreatment (n = 20); no surface treatment (control group—Group 1), 2%CHX application (Group 2), and 1% BAC application (Group 3). A self-curing adhesive cement and an etch and rinse adhesive were used for the cementation of posts. Three sections (one cervical, one middle, and one apical) of 1 mm thickness were prepared from each specimen. A push-out test was performed immediately on the half of the specimen sections (n = 10). The other half of the specimen sections were subjected to 20.000 thermal cycles before applying the push-out test (n = 10). The failure mode of each specimen was observed under a stereomicroscope at ×40 magnification. Results: The data were analyzed by one-way analysis of variance (ANOVA), Tukey Honestly significant difference (HSD), and Tamhane tests (P = 0.05). The cervical thirds displayed the highest, and the apical thirds showed the lowest values in all groups (P < 0.05), except the control-aged group (P = 0.554). The aged control groups' values were found to be significantly lower than the aged CHX and BAC groups (P < 0.001). Aging significantly reduced the bond strength values of specimens in control groups (P < 0.001). However, aging did not significantly affect the push-out bond strength values of CHX and BAC groups (P > 0.050). The failure types were adhesive between the post and cement (type 1) in all groups, except control-aged group (type 2). Conclusion: The application of 2% chlorhexidine or 1% BAC may be an essential step that can be taken to preserve the bond strength of fiber posts.

Keywords: Benzalkonium chloride, bond sthrength, chlorhexidine, fiber post

#### **INTRODUCTION**

frequently used in teeth iber posts are with extensive structural loss to provide retention for restorations after endodontic treatment.<sup>[1]</sup> The retention between fiber post, cement, and dentin is affected by many factors, including dentin pretreatment.<sup>[2]</sup> Ensuring adequate adhesion of fiber posts is important for the durability of the

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	DOI: 10.4103/njcp.njcp_434_22			

restorations.<sup>[3]</sup> However, the adhesion between resin and dentin is weakened by proteolytic degradation by cysteine cathepsins and matrix metalloproteinases (MMPs).<sup>[4,5]</sup> MMP inhibitors can be applied to the

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How to cite this article: Hazar A, Akgül S, Hazar E. Effect of chlorhexidine and benzalkonium chloride on the long-term push-out bond strength of fiber posts. Niger J Clin Pract 2023;26:1242-8.

dentin surface to preserve the bond strength between resin and dentin.  $^{\rm [6]}$ 

Chlorhexidine (CHX), which is used as an antimicrobial agent in endodontic treatment, is a commonly used MMP inhibitor. Previous studies showed that CHX can reduce the degradation of resin-dentin adhesion by inhibiting the activation of MMP-2, 8, and 9 and cysteine cathepsins.<sup>[6,7]</sup>

Quaternary ammonium compounds (QAC) are cationic structures. They have the same antimicrobial and MMP inhibitor properties as CHX. Benzalkonium chloride (BAC) is a cationic surface agent in the quaternary ammonium group and is used in dentistry as a cavity disinfectant. Studies have shown that BAC significantly inhibits MMP-2, 8, and 9.<sup>[8]</sup>

There are various studies investigating the long-term effects of different MMP inhibitor applications on adhesion.<sup>[9-11]</sup> However, there are no studies evaluating and comparing the immediate and long-term effects of CHX and BAC on the bond strengths of fiber posts.

This study aimed to evaluate the immediate and long-term (aging simulation corresponding to 2 years) effects of BAC and CHX application, on the push-out bond strength of fiber posts in different post space thirds (cervical, middle, and apical). The first null hypothesis was that there was no significant difference between the push-out bond strength of fiber posts in different post space thirds. The second null hypothesis was that dentin pretreatment with different MMP inhibitors had no effect on the immediate adhesion of fiber posts. The third null hypothesis was that the agents applied to the root dentin had no effect on the adhesion of fiber posts after an aging simulation corresponding to 2 years.

#### **MATERIAL AND METHODS**

#### Specimen selection and endodontic treatment

Sixty single-rooted mandibular premolar teeth with similar root lengths, freshly extracted for periodontal reasons, were included. The teeth were used with the patients' consent and approval from the Research Ethical Committee of Zonguldak Bülent Ecevit University (protocol number: 2022/15). All teeth were examined by digital radiography (VistaScan, Dürr Dental, Beitigheim-Bissinger, Germany) from buccal and proximal directions to determine the absence of aberrant canal morphology and observed with a dental operating microscope (Leica Microsystems, Wetzlar, Germany) under ×25 magnification to determine the presence/absence of root resorption or fracture. The teeth with root fracture, resorption, root curvature, and endodontic treatment were excluded. Teeth were stored in 0.1% thymol solution at 4°C until use after the removal of soft tissues and calculus on the root with a scaler. The coronal part of the teeth was sectioned at 16 mm from the apex to obtain standardized root lengths. Access cavity preparation was performed, and the apical patency was detected with a #10 K file (Shenzhen Denco Medical Company, Guangdong, China). The working length was determined as 1 mm shorter than the visible file length at the apical foramen. Root canal preparations were performed with WaveOne Gold nickel-titanium (NiTi) instruments (Dentsply Maillefer, Baillagues, Switzerland) used with an endodontic motor (X-Smart plus, Dentsply Maillefer). WaveOne Gold Small (#020/.07) and WaveOne Gold Primary (#025/.07) instruments, respectively, were used at working length according to the manufacturer's instructions. During the instrumentation, 2 ml 2,5% sodium hypochlorite (NaOCl) was used for irrigation between the files used. Following NaOCl irrigation, 2 ml of 17% ethylenediaminetetraacetic acid (EDTA), 2 ml of distilled water, and 2 ml of 2% chlorhexidine (CHX) were used for final irrigation, respectively. WaveOne Gold Primary absorbent points were used to dry the root canal. After the absorbent point was removed from the canal, it was inspected, and if the tip was wet, a new one was placed. An average of four-five absorbent points was used in each prepared canal. Then the root canals were obturated with WaveOne Gold Primary gutta-percha points and AH Plus jet root canal sealer (Dentsply Maillefer) using the single cone technique. The 1-mm cervical part of the obturation material was removed with the heated instrument, and the root canal orifice was sealed by using self-etch adhesive and flowable composite. Specimens were stored for two weeks at 100% humidity and 37°C to set the root canal sealer.

#### Post space preparation and experimental groups

After the storage period, the coronal 10 mm of the root canal was prepared with slow-speed drills for the insertion of the X-Post #2 (Core and Post System, Dentsply Maillefer). Largo Peeso Reamer #1 was used to remove gutta-percha from the canals. Red color-coded EasyPost Precision drills (Dentsply Maillefer) were used to shape the post spaces. The suitability of the post space of the X-Post #2 was checked. The post space was etched for 15 s with DeTrey Conditioner 36 (Core and Post System, Dentsply Maillefer), rinsed for 30 s with distilled water, and then dried with paper points. Next, the specimens were randomly divided into three groups according to the post space irrigation with different solutions used, as follows:

**Group 1 (control)**: The post spaces were rinsed with 2 ml of distilled water using a syringe and a 30-gauge needle (n = 20).

**Group 2 (CHX)**: The post spaces were rinsed with 2 ml 2% CHX solution (Calacept, Nordiska Dental, Ängelholm, Sweden) using a syringe and a 30-gauge needle (n = 20).

**Group 3 (BAC)**: The post spaces were rinsed with 2 ml 1% BAC solution using a syringe and a 30-gauge needle (n = 20). Powdered 10 g of BAC (Sigma-Aldrich Co, St. Louis, USA) material was dissolved in 100 ml of distilled water to obtain 1% solution of BAC and stored at room temperature until use.

After the post spaces were rinsed with solutions for 60 s, absorbent points (#90/.02, Dentsply) were used to remove the solutions. The surface of the X-Posts to be used was cleaned with alcohol. Prime and Bond XP and Self-Cure Activator (Core and Post System, Dentsply) were mixed for 2 s in equal ratio and then applied to post space and post surface. The solvent was evaporated from the post space and post surface by blowing air for 5 s. Core-X flow (Core and Post System, Dentsply) was used into the post space, then X-post was placed and was polymerized for 20 s with a LED light device (Elipar S10, 3M ESPE, Seefeld, Germany, 1200 mW/cm<sup>2</sup>).

# Specimen preparation, post dislodgement, and failure types

The roots of each specimen were embedded in acrylic resin and were sectioned parallel to the horizontal axis using a low-speed diamond saw (Mecatome T180, PRESI, Grenoble, France) under water cooling to obtain 1.0-mm slices from each post space third (one cervical, one middle, and one apical). Twenty apical, 20 middle, and 20 cervical slices of post space thirds were obtained from each group. Post space thirds of each group were randomly divided into two subgroups (a, b) according to the aging process (n = 10). Group (1a-2a-3a): The subgroups were stored at 100% humidity and 37°C for 24 h. Group (1b-2b-3b): Subgroups were subjected to 20.000 thermal cycles in cold water at 5°C followed by hot water at 55°C for 30 s in each bath for clinically equivalent aging to 2 years (MTE 101; MOD Dental, Esetron Smart Robotechnologies, Ankara, Türkiye). The push-out test was performed using a universal testing machine at 0.5 mm/min crosshead speed (MOD Dental MIC-101, Esetron Smart Robotechnologies, Ankara, Türkiye) for each specimen slice. The push-out test load was applied in the apical to cervical direction, until the post was dislodged. The measured values were recorded in Newton (N). To obtain the push-out bond strength in megapascal (MPa), the maximum failure load recorded

in newtons (N) was divided by the area  $(mm^2)$  of the post-dentin interface, according to the formula described by Sagsen *et al.*<sup>[12]</sup> The mean value was recorded for each root section tested.

After push-out tests, all specimens were observed at ×40 magnification under a stereomicroscope (Olympus SZ60, Tokyo, Japan). Representative images of failure types are shown in Figure 1. Failure modes were examined and classified as follows: type 1—adhesive failure between post and cement; type 2—adhesive failure between dentin and cement; type 3—mixed.

#### Statistical analysis

The data were analyzed using IBM SPSS V23. Shapiro–Wilk test was used to evaluate conformity to the normal distribution. The normally distributed measurements were compared with one-way analysis of variance (ANOVA). Tukey HSD and Tamhane tests were used for multiple comparisons. The normally distributed values in each group were compared using paired sample *t*-test (P = 0.05).

#### RESULTS

Table 1 presents the mean push-out bond strength and standard deviation values of the groups. In the comparison of post space thirds, a significant difference was found between push-out bond strength values of the cervical and the apical thirds of all groups (P < 0.05), except the control-aged group. (P = 0.554). The highest bond strength values were measured in the cervical thirds of roots, while the lowest bond strength values were measured in the apical thirds of post spaces.

Table 2 shows the mean and standard deviation values of push-out bond strengths of groups independent of the post space thirds. According to the results, no significant difference was found between bond strength values of non-aged groups (P = 0.880). When we compared the aged groups, the values of the control group were significantly lower than the CHX and BAC groups (P < 0.001). In the control-aged group, the values were significantly lower than the values of control non-aged group (P < 0.001), but no significant difference was found in the CHX and the BAC groups (P > 0.05).

Table 3 presents the failure mode percentages of the groups. In non-aged groups of control, CHX, and BAC, failures were predominantly type 1—adhesive failure between the post and cement. In aged groups of CHX and BAC, the failure types did not change, while the control-aged group had the highest rate of type 2—adhesive failure between dentin and cement.

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Figure 1: Representative images of failure types that were used for classification (×40). a) Adhesive failure between the post and cement (type 1). b) Adhesive failure between dentin and cement (type 2), the post is completely covered with a uniform layer of cement. c) Mixed (type 3). The black arrows indicate the different types of failures

Table 1: Mean and standard deviation values of push-out bond strengths (MPa) in the different post space thirds					
	Post space thirds	Control	CHX group (mean±SD)	BAC group (mean±SD)	
		group (mean±SD)			
Immediate	Cervival	15.22±0.88 <sup>b</sup>	15.69±2.76 <sup>b</sup>	15.8±2.37 <sup>b</sup>	
	Middle	13.68±2.49 <sup>ab</sup>	$14\pm 2.04^{ab}$	$13.8 \pm 1.57^{ab}$	
	Apical	$12.79 \pm 1.75^{a}$	$12.82{\pm}1.8^{a}$	$12.76 \pm 1.46^{a}$	
One-way ANOVA test		F=8.159-P=0.004	F=4.166- P=0.026	F=7.05- P=0.003	
After aging	Cervival	9.63±1.93ª	15,25±1,54 <sup>b</sup>	15,43±1,29 <sup>b</sup>	
	Middle	9.05±1.3ª	$13,98 \pm 1,16^{ab}$	$13,4\pm1,67^{a}$	
	Apical	8.88±1.53ª	12,77±1,22ª	12,69±1,3ª	
One-way ANOVA test		F=0.604-P=0.554	F=8.85-P=0.001	F=9.851-P=0.001	
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The different lowercase letters in columns represent statistical different (P < 0.05)

Table 2: Mean and standard deviation values of push-out bond strengths in the groups independent of post space							
	Control group (Mean±SD)	CHX group (Mean±SD)	BAC group (Mean±SD)	One-Way ANOVA Test			
Immediate	13.89±2.04 <sup>Aa</sup>	14.17±2.47 <sup>Aa</sup>	14.12±2.19 <sup>Aa</sup>	F=0.128- P=0.880			
After aging	$9.19 \pm 1.58^{Ab}$	$14{\pm}1.64^{Ba}$	$13.84{\pm}1.82^{Ba}$	F=79.114-P<0.001			
Paired Sample t-test	t=10.456- P<0.001	t=0.458- P=0.650	<i>t</i> =0.635- <i>P</i> =0.530				
			1 1 1 1 1 22 (5)	0			

The different uppercase letters in rows and different lowercase letters in columns represent statistical different (P<0.05)

Table 3: Percentage distribution of failure modes in the groups								
Failure Types	Control	Control-after aging	CHX	CHX-after aging	BAC	BAC-after aging		
Type 1 (adhesive failure, post-cement)	66.7	36.7	73.3	70	73.3	66.7		
Type 2 (adhesive failure, dentin-cement)	26.7	53.3	20	26.7	23.3	23.3		
Type 3 (mixed)	6.6	10	6.7	3.3	3.3	10		

Mixed (type 3) was the least common type of failure in all groups.

#### DISCUSSION

When conducting *in vitro* studies of restorative materials, some artificial aging process is often used to simulate clinical scenarios. These aging processes can be performed using thermal cycling, cyclic loading, or both at the same time.<sup>[13]</sup> It has been stated that the use of thermal cycling performs the aging process by imitating the thermal changes occurring in the mouth, and every 10.000 cycles applied during the procedure is clinically equivalent to 1 year.<sup>[14]</sup> In this study, 20.000 cycles of thermal cycling were applied to the

groups to be tested for an aging process corresponding to 2 years clinically.

The push-out test provides a more accurate evaluation of the adhesion mechanism inside the root canal, allowing for better simulating a clinical scenario.<sup>[15]</sup> Therefore, in the current study, the push-out test was used to evaluate the bond strengths before and after the thermal cycling aging.

The push-out bond strengths were evaluated in the cervical, middle, and apical post space thirds, in the current study. According to the results, the apical third presented significantly lower bond strength in all groups except for the after-aging control group. Therefore, the

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first null hypothesis was rejected for this study. These results are compatible with several studies' findings.<sup>[16,17]</sup> However, contrary to these results, some researchers found equal bond strength values in different post space thirds.<sup>[18,19]</sup> Also, in another study, researchers reported higher tensile strengths in the apical post space thirds than in the cervical and middle thirds.<sup>[20]</sup> It can be thought that the difference between the values is the result of poor cement penetration due to the difficulty of accessing the apical third and the incomplete removal of the smear layer in this region.<sup>[21]</sup> In addition, the polymerization problems caused by the distance of this region from light access may have caused these differences in values.

When the effect of CHX application to the root canal was considered, CHX application to the root dentin did not interfere with the immediate bond strength of fiber posts, in the current study. Therefore, the second null hypothesis was rejected. These findings are supported by several studies' results indicating that the CHX solution does not seriously affect the immediate push-out bond strength of fiber posts.[7,22-24] Nevertheless, Cecchin et al.<sup>[25,26]</sup> stated that chlorhexidine application is not a procedure that increases the immediate bond strength of fiber posts in root dentin. Contrary to the current results, several studies have shown that the application of chlorhexidine provides a higher bond strength.[27,28] These authors indicated that certain properties of CHX, such as increasing the surface energy of dentin, could lead to an enhancement in the primers' wetting ability, thus improving adhesion. Some authors stated that the application of CHX negatively affects the adhesion between dentin and resin.<sup>[29]</sup>

There is no study examining the short- and long-term efficacy of BAC application on the push-out bond strengths of fiber posts. In previous studies, only the effects of BAC application on the coronal dentin were investigated. According to the current results, the application of 1% BAC to the root dentin did not interfere with the immediate push-out bond strength of fiber posts. Similar to the current findings, Pashley et al.[30] showed that the use of 1% BAC did not impair the immediate bond strength of the adhesive. Contrary to current results, a few studies indicated that BAC may cause a decrease in the immediate bond strength.[31,32] This difference in results may be due to different study methodologies. In these studies, bond strengths were evaluated by shear testing and BAC was applied either in different concentrations or not individually but in a disinfectant called Tubulicide red with 0.1% BAC in it.

Several studies indicated that BAC increases the immediate bond strength at the resin-dentin interface.<sup>[33,34]</sup>

In these studies, Sabatini et al.[33,34] reported that the use of 1% BAC increased the bond strength at the instant resin-dentin interface. Unlike the current study, these studies tested bond strengths in coronal dentin. De Goes et al.[35] reported that although the adhesion procedures were similar in coronal and root dentin, the bond strength values in root canal dentin was lower than the values in coronal dentin. It has been stated that the reason for this may be the differences in structural components and mineral composition in coronal and root dentin.<sup>[36,37]</sup> In addition, in a study examining dentin sections. Caiado et al.<sup>[38]</sup> stated that root canal dentin contains much less peritubular dentin than coronal dentin. Therefore, it can be thought that this difference may change the bond strength by affecting the resin diffusion.

In this study, it was found that the bond strength of the control group without MMP inhibitor application decreased significantly after aging. MMPs are a class of endopeptidases found in the mineralized dentin matrix, and the presence of MMPs is a factor that reduces the bond strength at the resin-dentin interface over time.<sup>[39]</sup> Many studies have indicated that MMPs may be responsible for the degradation of resin-dentin bonds over time due to endogenous proteolytic activity.<sup>[22,28,33,34,40]</sup> The etching process in bonding applications can activate latent MMPs in the dentin matrix, as low pH environments cause changes in enzymes that expose their catalytic domains.<sup>[40]</sup> It can be thought that the decrease in the push-out bond strength of the after-aging control group is a result of endogenous proteolytic activity.

The application of CHX and BAC into the root dentin preserved the push-out bond strength of fiber posts after aging corresponding to 2 years; therefore, the third null hypothesis was rejected.

In the current study, 2% CHX<sup>[26]</sup> and 1% BAC<sup>[41]</sup> solutions as MMP inhibitors were used after acid application and before cement application to prevent endogenous proteolytic activity. Several studies have reported that CHX and BAC preserve resin-dentin hybridization and prevent degradation of adhesion over time by inhibiting MMPs.<sup>[16,42]</sup> The current results are in accordance with previous studies, where it was indicated that the use of CHX preserves the resin-dentin bond for a long time in fiber post applications.<sup>[22,28,43]</sup>

There is no study evaluating the long-term effect of BAC on push-out bond strength in root dentin, but in studies on coronal dentin, the use of 1% BAC was found to be effective in maintaining the long-term adhesive interface.<sup>[33,42,44]</sup> In addition, consistent with the current

results, Sabatini *et al.*<sup>[44]</sup> reported that 1% BAC preserves the resin-dentin bond after 6 months and 1 year.

In this study, the immediate and long-term effects of CHX and BAC were also compared with each other and no significant difference was found. Therefore, BAC can be considered as an alternative to CHX to obtain reliable long-term bond strength in fiber post application.

When the failure types were observed, type 1 was the most common failure in all groups except for the after-aging control group, which had the lowest bond strength values. The failures observed in the after-aging CHX and BAC groups are mostly adhesive failures between the post and the cement (type 1). According to these results, it can be stated that the adhesive interface between cement and dentin is partially preserved after long-term aging.<sup>[45]</sup>

A limitation of this study was using only one concentration of BAC. It is necessary to examine the effects of different concentrations of BAC. Also, further *in vivo* studies are needed to evaluate the effect of BAC on the bond strength of fiber posts.

#### CONCLUSION

Within the limitations of this *in vitro* study, 1% BAC application provided a long-term bond strength as effectively as 2% CHX at the resin-dentin interface of fiber post restorations. According to the current results, the application of 1% BAC and 2% CHX may be an essential step that can be taken to preserve the bond strength of fiber posts.

#### Financial support and sponsorship

This study was supported Zonguldak Bülent Ecevit University Scientific Research Project Coordination Unit (Project no: 2019-77431340-01).

#### **Conflicts of interest**

There are no conflicts of interest.

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