



# Resin push-out bonding strength to root canal dentin: effect of the irrigation solution application prior to post cementation

Resistência de união ao push-out entre resina e dentina radicular: efeito utilização da solução irrigadora antes da cimentação do pino

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## ABSTRACT

**Objective:** To evaluate the effect of different irrigation solutions prior to post cementation on the resin bonding to bovine root dentine. **Material and Methods:** 60 bovine roots (16 mm) were prepared to 12 mm with specific drill of a double-tapered fiber post system, included in PMMA, and divided into 6 groups, considering the irrigation solutions (IS) prior to cementation: Gr1- saline solution (control), Gr2- NaOCl 0.5 %, Gr3- NaOCl 1 %, Gr4- NaOCl 2.5 %, Gr5- NaOCl 5 %, Gr6- Chlorhexidine 2 % (solution). The root canals were irrigated with IS 20 ml during 10 min. Then they were rinsed with 20 ml of distilled water and dried with paper points. One fiber post was molded with polyvinylsiloxane and 60 posts made of resin cement (PRC) were obtained moments before the cementation. The root canal dentin was etched with H<sub>2</sub>PO<sub>3</sub> 37 %/15 s + washing/drying, a multi-bottle etch&rinsse adhesive system was applied and the PRC were resin luted with dual resin cement (Duolink<sup>TM</sup>, Bisco). Each specimen was cut into 4 slices of ±1.8mm in thickness and submitted to push-out test (1 mm/min). **Results:** ANOVA showed that bond strength was significantly affected by IS (P<0.0001). The highest bond strengths (MPa) were those for the groups 4 (3.51 ± 1.52) and 5 (3.0 ± 1.16). The groups 1 (0.70 ± 0.30), 2 (0.80 ± 0.24), 3 (1.26 ± 0.57) and 6 (0.90 ± 0.41) were statistically similar to each other. **Conclusion:** The resin bonding to the root dentine was higher when higher concentrations of hypochlorite solutions were used.

## KEYWORDS

Bond strength; Irrigation solutions; Push-out test; Resin cement; Root canal dentin.

## RESUMO

**Objetivo:** Avaliar o efeito de diferentes soluções de irrigação antes da cimentação do pino na resistência de união da resina à dentina radicular bovina. **Material e Métodos:** Foram preparadas 60 raízes bovinas (16 mm) a 12 mm com broca específica de um sistema de pino de fibra com dupla conicidade, incluídas no PMMA, e divididas em 6 grupos, considerando as soluções irrigadoras (IS) antes da cimentação: Gr1-Solução salina (controle), Gr2-NaOCl 0,5 %, Gr3-NaOCl 1 %, Gr4-NaOCl 2,5 %, Gr5-NaOCl 5 %, Gr6-Clorexidina 2 % (solução). Os canais radiculares foram irrigados com 20 ml de IS durante 10 min. Depois, foram lavados com 20 ml de água destilada e secos com pontas de papel. Um pino de fibra foi moldado com polivinilsiloxano e 60 pinos de cimento resinoso (PRC) foram obtidos momentos antes da cimentação. A dentina radicular foi condicionada com H<sub>2</sub>PO<sub>3</sub> 37 %/15 s + lavagem/secagem, aplicou-se o sistema de adesivo e o PRC foi cimentado com cimento resinoso dual (Duolink<sup>TM</sup>, Bisco). Cada amostra foi cortada em 4 fatias de ± 1,8 mm de espessura e submetida a teste push-out (1 mm/min). **Resultados:** ANOVA mostrou que a força de união foi significativamente afetada pelo IS (P <0,0001). As maiores resistências de união (MPa) foram para os grupos 4 (3,51 ± 1,52) e 5 (3,0 ± 1,16). Os grupos 1 (0,70 ± 0,30), 2 (0,80 ± 0,24), 3 (1,26 ± 0,57) e 6 (0,90 ± 0,41) foram estatisticamente semelhantes entre si. **Conclusão:** A resistência de união da resina à dentina radicular foi maior quando foram utilizadas concentrações maiores de soluções de hipoclorito.

## PALAVRAS-CHAVE

Resistência de união; Soluções irrigadoras; Teste push-out; Cimento resinoso; Dentina intrarradicular.

## INTRODUCTION

Prospective clinical evaluations of teeth restored with resin bonded fiber-reinforced composite post (FRC) have noticed that failure occur mainly at the adhesive system - root dentin interface, and consequently debonding of the crown-FRC-cement set [1]. Some factors such as lower hybridization potential of the dentinal substrate [2], high configuration factor in root canals (high polymerization stress of resin cements) [3], difficult light curing inside the root canal [4] and cement layer thickness [5,6] may impair the resin bond between fiber post and root dentin. The optimization of the bonding to root canal dentin is crucial to reduce the chance of the clinical pull-out of fiber post [7]. One of the procedures to optimize it could be the irrigation of the root canal with some solutions prior to fiber post cementation [8].

From the clinical point of view, usually the fiber post cementation is performed some days later after the endodontic treatment has been concluded. Thus, bacteria can contaminate the root dentin, and the application of anti-microbial solutions can be indicated [9]. Commercially available, antimicrobial agent based on chlorhexidine digluconate, sodium hypochlorite, hydrogen peroxide, and iodine have been used to remove oil and bacterial contaminants.

Sodium hypochlorite (NaOCl) is known to alter proteins and collagen fibers [9], thus interfering with the formation of the hybrid layer [11]. Other studies confirm that NaOCl alters the dentin, hindering the resin bonding [12]. Since adhesion to dentin is influenced by the presence of residual calcium on the bonding area, the calcium removal is potentially detrimental, especially for total-etch adhesives [13]. Morris et al. [8] found that the both 5% NaOCl and root canal preparation (RC-Prep) reduced significantly the resin bond strengths to root canal dentin, and the reduction could be completely reversed by the application of either

10% ascorbic acid or 10% sodium ascorbate. Tuncel et al. found that the use of different solutions did not present significant difference in push-out test [14], as Bueno et al. that found no significant difference for evaluated solutions combined with two resin cements [15].

Several authors have investigated the role of sodium hypochlorite used as a disinfectant prior to bonding procedure [16]. The effect of some irrigation solutions on the disinfection of the canal walls was studied and related to the bond strength [8,13,17].

The aim of this study was to evaluate the influence of the antimicrobial irrigation solutions on the resin push-out bond strength to bovine root canal dentin. The null hypothesis was that the irrigation solutions would not influence the bond strength.

## MATERIAL AND METHODS

### *Selection and teeth preparation*

Sixty bovine teeth were extracted and cleaned (no longer than three years-old) and stored in distilled water. The coronal and cervical portions of the single-rooted bovine teeth (mandibular incisors) were sectioned for standardization of the size of the roots at 16 mm (Figure 1a and 1b). The coronal diameters of the canals were measured with a digital caliper (Starrett® 727, Starrett, Itu, Brazil) and specimens presenting diameters larger than the diameter of the post (2.0 mm) were discarded and replaced by other specimens having this requirement.

In all teeth, the working length was determined visually by subtracting 1 mm from the length of a #10 K-file (Dentsply Maillefer, Ballaigues, Switzerland) at the apical foramen. Biomechanical preparation was performed manually with K-files until size #40. After each step in the flare preparation, the canal was irrigated with 2.5% sodium hypochlorite during 1 min. Then, 17% EDTA was used as a

final irrigant for 1 min, after a rinse of distilled water (1 min) to remove any remnants of the irrigating solutions. The canals were dried using paper points. Subsequently prepared at 12mm, using preparation burs #3 of a taper glass fiber-reinforced resin post system (FRC Postec Plus, Ivoclar-Vivadent AG, Schaan, Liechtenstein), at low-speed under water-cooling (Figure 1c).

Each specimen was embedded in a cylinder-shaped silicon mold filled with chemically cured acrylic resin (Dencrilay®, Dencril, Caieiras, Brazil) (Figure 1d). The following procedure was performed for that purpose: a) the preparation bur of the post system was placed inside the prepared root canal; b) this assembly was attached in an adapted surveyor, so as the long axes of the bur, specimen and cylinder were parallel to each other and to the y axis; c) the acrylic resin was prepared and poured inside the cylinder up to half of the root. [18]

The roots were randomly divided into six groups (n=10) according to the irrigation solutions: Gr1- saline solution (control group), Gr2- 0.5 % sodium hypochlorite, Gr3- 1 % sodium hypochlorite, Gr4- 2.5 % sodium hypochlorite, Gr5- 5 % sodium hypochlorite e, Gr6- 2 % Chlorexidine (solution).

The root canals were irrigated with 20 ml of each irrigation solution (IS) and the last 3 ml were left in the canal. Elapsed 10 minutes, they were rinsed with 20 ml of distilled water, and dried with paper points.

A fiber post (2.0 X 20 X 1.0 mm) were molded with polyvinyl siloxane (Elite HD putty, Zhermack, Badia Polesine, Italy) and 60 posts made of resin cement (Duolink, Bisco, Schaumburg, USA) (PRC) were obtained moments before the cementation.

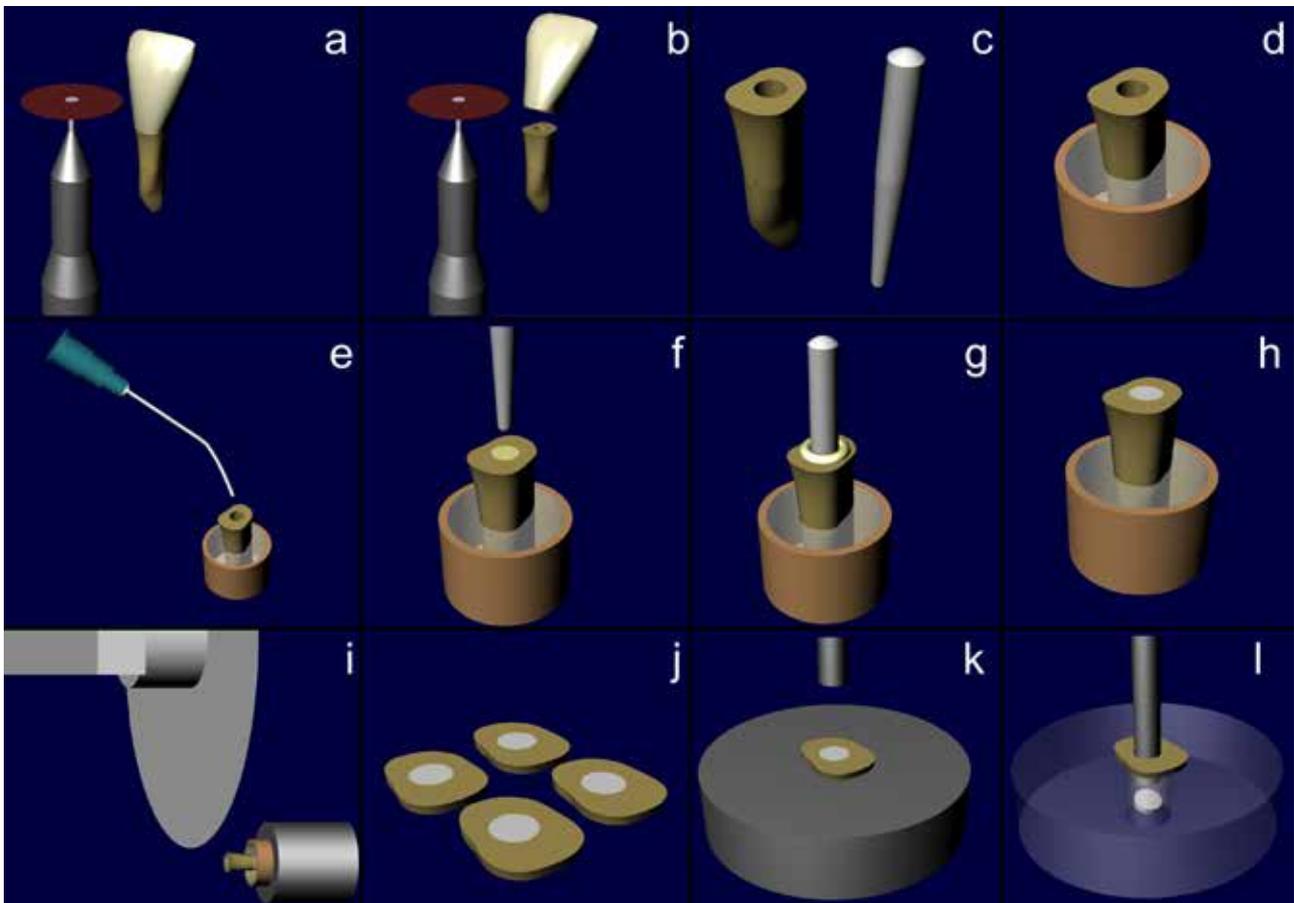
The root canal dentine was etched with 37 % phosphoric acid for 15 s (Figure 1e), rinsed with distilled water for 15 s using an endodontic irrigation needle and dried with paper points. Then the multiple-bottle, etch-

and-rinse adhesive system (AllBond 2, Bisco, Schaumburg, USA) was applied in according to the manufacturer's instruction: a) the Primer A® and Primer B® were mixed and placed to the canal with applicator tips (Cavi-Tip, Svenska Dental Instrument AB, Upplands Värby, Sweden); b) the Pre-Bond Resin® was then applied; c) the base and catalyst pastes of the dual resin cement (Duolink™, Bisco) were handling according to the manufacturer's instructions and inserted inside the root canal with a Lentulo spiral n°. 40 (Dentsply/Maillefer, Ballaigues, Switzerland (Figure 1f); d) the resin post was placed in the root canal (Figure 1g and 1h). The cement was then photoactivated cervically for 80 seconds (40 seconds buccally and 40 seconds lingually) with 1200 mW/cm<sup>2</sup> of power (Radii-Cal, SDI [North America], Inc) All teeth were stored in distilled water for 24 h at 37 °C.

#### *Producing of the samples and push-out testing*

Each specimen was fixed on the metallic base of a cutting machine (LabCut 1010, Extec Corp., Enfield, CT, USA), which allowed perpendicular sectioning along the root axis (Y axis) with a diamond disc under cooling spray, preparing approximately 1.8 mm thick slices (Figure 1i and 1j). The first cervical slice (about 1 mm) was discarded, because the excess of cement in that region could influence the result of the bond strength. Four to five other slices per specimen were prepared.

Each specimen was positioned on a metallic device with a central opening ( $\varnothing = 3$  mm) larger than the canal diameter with the most coronal portion of the specimen placed downwards (Figure 1k). For push-out testing, a metallic cylinder ( $\varnothing_{\text{extremity}} = 0.85$  mm) induced a load from the apical to coronal direction on the resin post (Figure 1l). The test was performed in a universal testing machine (EMIC, São José dos Pinhais, Brazil) 50 kgf load cell at a speed of 1 mm/min. [6]



**Figure 1** - Illustrative sequence of push-out methodology. (a,b) Procedure to cut bovine teeth. (c) Cutted root and fiber post. (d) root inserted in acrylic resin. (e) Dentin conditioning. (f) Application of resin cement. (g,h) Post cementation. (i) Set adapted in a cutting machine. (j) Samples. (k) Specimen placed in an universal test machine. (l) Tested sample.

The bond strength ( $\sigma$ ) in MPa was obtained by the formula  $\sigma = F/A$  where, F = load for specimen failure (N) and A = bonded area (mm<sup>2</sup>). For the area calculation, a formula was applied to calculate the lateral area of the geometric figure, a circular straight cone trunk of parallel bases. The formula used for the area calculation was  $A = \pi g \times (R1 + R2)$  where,  $\pi = 3.14$ , g = trunk generatrix, R1 = smaller base radius, R2 = larger base radius. For conic trunk generatrix calculation g, the Pythagorean theorem was used – “the square of the hypotenuse = the sum of the squares of the two catheti” ( $g^2 = h^2 + [R2 - R1]^2$ ) where h = section height. R1 and R2 were obtained by measuring the internal diameters of the smaller and larger base, respectively, corresponding to the internal diameter between the root canal walls. These diameters and h were measured

with a digital caliper (Starrett® 727, Starrett, Itu, Brazil).

#### *Statistical analysis*

The mean bond strength values from each specimen were initially calculated from their respective repetitions. Considering that each group was composed of 10 specimens, 10 bond strength values of each group (n=10) were employed for statistical analysis (one-way analysis of variance and post-hoc Tukey test,  $\alpha=0.05$ ).

#### *Analysis of the failure modes*

The tested specimens were analyzed under an Optic Microscope (magnification of x50) (Zeiss MC 80 DX, Zeiss, Jena, Germany) in order to evaluate the type of fracture in the samples. The classification used was as following: 1)

fracture of the resin post (cohesive); 2) failure between resin cement and root dentin (pull-out of the resin post – cement) (adhesive failure); 3) cohesive fracture of the root dentin (cohesive). All samples were analyzed for three calibrated observers. Some representative specimens were selected for observation under scanning electron microscope (SEM) from x20 to x1000 magnification.

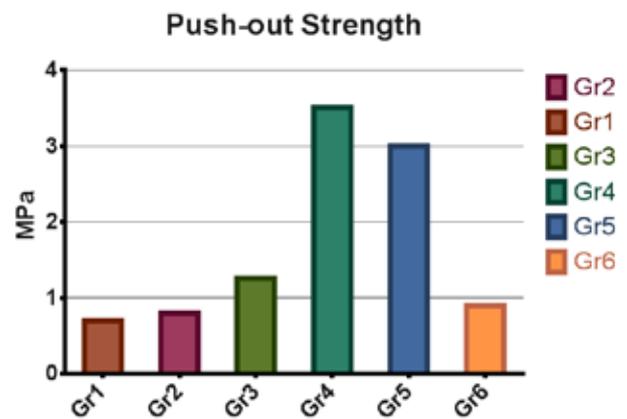
**RESULTS**

ANOVA revealed that the push-out bond strength depends upon irrigation solution significantly ( $P < .0001$ ) (Table I). The post-hoc Tukey All-Pairwise Comparisons Test showed that Gr4 ( $3.51 \pm 1.52b$ ) and Gr5 ( $3.00 \pm 1.16b$ ) were statistically similar to each other and presented the highest bond strengths (MPa). Gr1 ( $0.7 \pm 0.3a$ ), Gr2 ( $0.80 \pm 0.24a$ ), Gr3 ( $1.26 \pm 0.57a$ ) and Gr6 ( $0.90 \pm 0.41a$ ) were similar statistically (Table I). Thus the null hypothesis was rejected. In Figure 2, mean bond strength values are summarized.

The percentages of failures in all groups were 84.17 % mixed, 15 % cohesive in resin cement, 0.42 % cohesive in dentin and 0.42 % adhesive (between dentin/resin cement) (Table

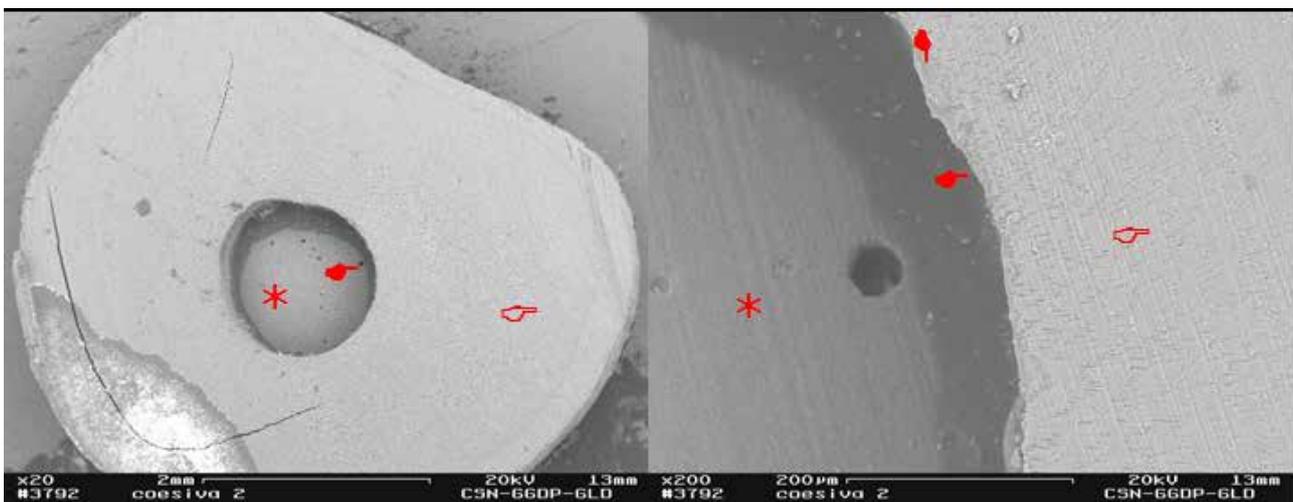
**Table I** - Results of 1-way ANOVA

Source	df	SS	MS	F	P
Between	5	76.011	15.2021	21.1	<0.0001*
Within	54	38.960	0.7215		
Total	59	114.970			



**Figure 2** - Graphic of means and standard deviations of the testing groups (Gr1- saline solution; Gr2- 0.5% sodium hypochlorite; Gr3- 1% sodium hypochlorite; Gr4- 2.5% sodium hypochlorite; Gr5- 5% sodium hypochlorite; Gr6- 2% Chlorhexidine).

II). The Figure 3 shows the representative micrographs of a mix failure from tested specimens



**Figure 3** - Representative micrograph of a mix fracture from pushed-out specimen (x20 and x200): Asterisk \* = the resin post; ♦ = rupture of the dentin-resin cement interface combined with a fracture of the cement (♦); ◀ = dentin.

**Table II** - Distribution of failure types

Groups	Mixed	Cohesive in resin cement	Cohesive in dentin	Adhesive	Total
G1	28	11	0	1	40
G2	31	8	1	0	40
G3	31	9	0	0	40
G4	40	0	0	0	40
G5	40	0	0	0	40
G6	32	8	0	0	40

## DISCUSSION

This study observed that higher concentrations of sodium hypochlorite promoted higher values of bond strength. The sodium hypochlorite treatment can remove the organic components of dentin, mainly collagen. This should increase the tubules diameter [16] allowing the penetration of monomers into the demineralized dentin structure [19]. Probably this fact occurred in this current study and can be responsible for the improvement of the bond strength when higher concentrations of sodium hypochlorite (G4 and G5) were used.

The bond strength between resin and dentin was not affected by some irrigation solutions (NaOCl 5.25% and chlorhexidine 2%) [20]. However, chlorhexidine, used individually, does not seem to be able to completely recover the collagen matrix, preventing the collapse of the fibers [21], which would explain why it did not interfere with the immediate bonding strength. Despite this, the use of chlorhexidine would be able due to inhibition of the metalloproteinase, which could increase the longevity of the hybrid layer in the long follow-up time [22]. The NaOCl has a wide range activity against bacteria and fungus, that it can destroy the microbial biofilm effectively and that it has an excellent tissue dissolving ability [23]. However, its effects on the bond strength of bonding systems are still controversial [23]. While few studies justify the type of adhesive used it could be related to the oxidizing effect of hypochlorite solution, causing a decrease in adhesive strength of the root

surfaces that received these materials combined [24,25]. Several other authors corroborate with this study, in the results of superiority of solutions with NaOCl due to its capacity of removal the hybrid layer and immediate effect on the dentine's substrate [16,19,26,27].

NaOCl in varied concentrations promoted different effects on the penetration into dentinal tubule. The association of temperature and exposure time with this, had a significant relationship presenting a depth penetration [28]. Thus, some authors suggest for increase bond strength, an association of 5.25 % sodium hypochlorite, 17 % EDTA and 10 % of gallic acid an antioxidant to avoid the residual oxygen left in the dentinal tubules [20,29].

Others studies [16,30] have also demonstrated that if acid etching is followed by NaOCl treatment higher bond strengths can be achieved. According to Prati et al. [17] the use of the acidic conditioner for exposure of the collagen matrix exposes a soft delicate mesh that can collapse, thereby interfering with resin infiltration. If acid etching is followed by NaOCl treatment high bond strength can be achieved via "reverse hybrid layer" formation, a proposed new mechanism of micromechanical resin retention. In the current study, it was used a different procedure: the root dentin was firstly treated with NaOCl and, after that, it was etched, but even so, higher values of bond strength were achieved compared to the control group. Theoretically, it is possible that have formed the reverse hybrid layer independent of the order of the factors.

The use of the sodium hypochlorite with higher concentrations (2.5 % and 5 %) could contribute to improve the bond between restorations with resin bonded fiber-reinforced composite post and dentin. However, further laboratorial and clinical studies are necessary to support these findings. Results suggest that the protocols used for G1, G2, G3 and G6 groups shall be avoided due to the fact that a bond strength obtained through push out test (with similar methodology for specimen's confection) should be around 3.8 MPa [31].

Resin posts are bonded into the root canal as a "substitute" to the fiber post [32]. Resin posts were cemented in this study to evaluate the influence of different adhesive strategies on the bond of resin to root dentin, independent of the resin bond to the fiber post surface, thus preventing the possibility that the failures occurred between the resin cement and fiber post. At the same time, the higher configuration factor of root canals was reproduced [3,32]. It is obvious that the cementation of the resin post is not clinically indicated.

The current study used bovine teeth to evaluate the push-out bond strength. The use of bovine teeth as a substitute for human teeth in bond strength or microleakage tests is controversial. While many studies have shown similarities of bovine teeth to human teeth [35,36], other studies have found discrepancies between the two and stated several criticisms to the use of bovine teeth as a substitute-substrate [34,35]. It is suggested that bovine teeth can initially be used to evaluate an adhesive-material or technique before implementing it clinically. Wherefore, conductance ( $L_p$ ) and diffusional water flux ( $J_s$ ) of human and bovine dentin are not statistically different [37]. Although human and bovine teeth show results of shear bond strength statistically different, with the highest values observed for the human teeth, exists a similar behavior for depths of dentin tested [38]. Using bovine teeth can be the first parameter in the evaluation process.

## CONCLUSION

According to the methods, results and limitations of this study, it was concluded that the resin bonding to root canal dentin can be improved by using solutions based on NaOCl in high concentration (2.5 % and 5 %) prior to fiber post cementation.

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