

## ORIGINAL ARTICLE

**Influence of cement thickness on the bond strength of tooth-colored posts to root dentin after thermal cycling**FERHAN EGILMEZ<sup>1</sup>, GULFEM ERGUN<sup>1</sup>, ISIL CEKIC-NAGAS<sup>1</sup>, PEKKA K. VALLITTU<sup>2</sup> & LIPPO V. J. LASSILA<sup>2</sup><sup>1</sup>Department of Prosthodontics, Faculty of Dentistry, Gazi University, Ankara, Turkey, and <sup>2</sup>Institute of Dentistry, Department of Biomaterials Science and Turku Clinical Biomateris Centre– TCBC, University of Turku, Turku, Finland**Abstract**

**Objective.** The purpose of this study was to evaluate the effect of different resin cement thickness on the push-out bond strength of different posts (CAD/CAM zirconia post (ZR post)) and an individually formed glass fiber reinforced composite post (IPN post) prior to and after thermal cycling (TC). **Methods.** Post spaces with a height of 9 mm and a diameter of 1.5 mm were drilled in 80 mandibular premolar teeth. Two groups ( $n = 40$ ) were formed according to the posts used (IPN posts or ZR posts). Then the specimens were randomly assigned into two sub-groups according to the post diameter: (1) 1.5 mm in diameter and (2) 1.2 mm in diameter ( $n = 20$ /per group). All posts were luted with a self-adhesive luting agent according to the manufacturer's instructions by using endo tips. Half of the samples ( $n = 10$ ) were submitted to thermal cycling (5000 cycles, 5–55°C). Thereafter, four 2-mm thick horizontal sections were obtained and subjected to push-out test. Failure modes were assessed quantitatively and morphologically. The data were statistically analyzed with a three-way analysis of variance (ANOVA) ( $p < 0.05$ ). Statistical differences in failure modes were investigated by chi-square tests at a significance level of  $p < 0.05$ . **Result.** Push-out bond strengths were significantly influenced both by the post diameter and thermal cycling. Larger (1.5 mm) diameter post results were statistically higher than 1.2 mm results ( $p < 0.05$ ). Moreover, TC significantly increased the bond strength results ( $p < 0.05$ ). There was no significant difference between ZR and IPN posts ( $p = 0.219$ ). **Conclusion.** The bond strengths of ZR and IPN posts were significantly decreased when the resin cement layer was thick. In addition, thermal cycling drastically influenced bond strengths of the tested post materials. **Clinical significance:** The fit between tooth-colored endodontic posts and post spaces should be as tight as possible.

**Key Words:** bond strength, IPN post, resin cement thickness, thermal cycling, zirconia post**Introduction**

Numerous esthetic posts have become commercially available; such as glass fiber reinforced composite resin posts (FRC) and yttrium stabilized zirconia (Y-TZP)-based ceramic posts [1]. Studies have shown that Y-TZP-based ceramic posts possess high flexural strength and fracture toughness [2], besides they are extremely radiopaque [3] and biocompatible [4]. In addition, these posts have low solubility [5] and are not affected by thermal cycling [6]. On the other hand, the major advantage of the fiber-reinforced composite (FRC) posts is their stiffness, which is equal to that of dentin, as well as their high durability [7]. Previous studies demonstrated that individually formed posts

with continuous unidirectional E-glass fibers and a semi-interpenetrating polymer network (semi-IPN) showed better bonding results to composite resin luting cements than did pre-fabricated FRC posts with a cross-linked polymer matrix [8]. This polymer matrix owns a semi-IPN exhibiting linear polymer phases, cross-linked poly bis-GMA phases and polymethyl methacrylate (PMMA) [9]. The monomers of the adhesive resin and cements may penetrate into the linear polymer phase and form an inter-diffusion bonding by polymerization, called a secondary-IPN bonding [10].

One of the relevant problems clinicians face when restoring endodontically treated teeth is the mismatch between the diameter of the post space and that of the

post [11]. Although the use of size-matched drills supplied by post manufacturers permits good fitting of posts to the canal walls, root canals can have different shapes [12]; thus, the resin cement thickness around the post can also vary [13]. In addition, flared canals from carious extension, trauma, pulpal pathology and iatrogenic misadventure can also compromise the adaptation of the posts to canal walls [14].

The trend toward the simplification of clinical luting procedures has led to the introduction of self-adhesive resin cements. These newer systems, that produce bonding in a simple application, eliminate the need for enamel or dentin pre-treatment, simplify the cementation technique and diminish the chances of failures of the clinical procedures [15].

Thermal cycling was proposed as one of the important factors that affects the bond strength of tooth-colored post systems [16]. Thermal changes might cause expansion/contraction stresses within the material [17], which might affect the adhesive stability. Moreover, the cement expansion would also create friction along the root canal that could result in improvement of mechanical retention [16].

In general, studies about the bond strength of FRC and zirconia posts to luting cements have focused on the effect of various surface treatments, different luting agents and adhesive strategies [15,18]. Therefore, the purpose of this study was to evaluate the bond strength of different post diameters in post spaces of the same diameter on the bond strength of tooth-colored endodontic posts, prior to and after being submitted to thermal cycling. Following null hypotheses tested in this study it was decided that: (1) Push-out bond strength is not affected by the type of endodontic post; (2) the accuracy of fit between these tooth-colored posts and post spaces does not influence bond strengths; and (3) bond strengths of zirconia posts (ZR post) and FRC posts with semi-IPN (IPN posts) with different resin cement thickness do not vary with thermal cycling (TC).

## Materials and methods

Eighty single-rooted human mandibular premolar teeth with fully developed apices freshly extracted for periodontal or orthodontic reasons were selected and stored in 0.5% chloramine-T solution at 4°C for a maximum of 1 month prior to use.

### *Preparation of zirconia posts*

A total of 40 CAD/CAM ZR posts were fabricated. Half of the 40 posts were 9 mm in height and 1.5 mm in diameter ( $n = 20$ ) and the other half were 10 mm in height and 1.2 mm in diameter ( $n = 20$ ). CAD/CAM ZR posts were manufactured from pre-sintered Y-TZP disc shaped blocks (Copran Zr<sup>®</sup>, WhitePeaks

Dental GmbH & Co. KG, Essen, Germany). Posts were sintered to full density in a high-temperature furnace (Protherm, B&D Dental Origin Milling, UT, West Valley) at 1450°C for 2 h according to the manufacturer's instructions. All of the ZR posts received an airborne-particle abrasion with 30- $\mu$ m silicized Al<sub>2</sub>O<sub>3</sub> particles (CoJet™ Sand, 3M Espe, Seefeld, Germany) at 280 kPa pressure from a distance of 10 mm for 20 s. Posts were rotated during this procedure using an electric motor (PSR 2.4V; Robert Bosch GmbH, Stuttgart, Germany) at 130 rpm to apply the airborne particles to the entire treatment surface homogeneously. The specimens were then ultrasonically cleaned in 96% isopropyl alcohol for 3 min.

### *Tooth preparation*

The teeth were decoronated using a slow-speed diamond saw (Isomet 1000; Buehler, Lake Bluff, IL) under copious water cooling to create 14-mm-long root segments. The root canals were prepared to apical size 30 (F3) with ProTaper nickel titanium rotary instruments (Dentsply-Maillefer, Ballaigues, Switzerland). Thereafter, canal preparation followed with the ProFile NiTi rotary system (Dentsply Tulsa Dental, Tulsa, OK) until preparation was completed with a 0.06 taper, sizes 40. Between each file 2 ml of 5.25% NaOCl was used as an intra-canal irrigant during instrumentation. After preparation, the canals were irrigated with 5 mL NaOCl followed by 5 mL 17% ethylenediaminetetraacetic acid (EDTA) to remove the smear layer. Finally, the specimens were irrigated with distilled water to avoid the prolonged effect of the EDTA and NaOCl solutions. The canals were subsequently dried with paper points. Then the canals were obturated with single 0.06 taper size 40 gutta-percha cones (Dentsply-Maillefer) in conjunction with AH Plus sealer (Dentsply DeTrey GmbH, Konstanz, Germany) After the completion of endodontic treatment, cervical root canal openings were filled with a provisional restorative material (Cavit-G; 3M ESPE AG, Seefeld, Germany).

### *Post luting procedures*

The gutta-percha was removed with a warm plugger (Sybron Dental Specialties, Romulus, MI) leaving a minimum 4–5 mm apical seal and creating a standard post space of 9 mm from the apical surface. Sixteen peeso reamer no. 5 (ISO sized 1.5 mm in diameter) (1 reamer for five post space preparation) and eight gates-glidden drills no. 5 (ISO sized 1.2 mm in diameter) were used in the study. The conical apical parts of the reamers were flattened with 180-grit SiC papers under water irrigation.

Endodontically-treated teeth were divided into two groups in order to place ZR posts and IPN posts

(everStick<sup>®</sup>, Sticknet Ltd, Turku, Finland) ( $n = 40$ /per group). These post groups were divided into two sub-groups according to the post diameter used ( $n = 20$ /per group): (1) 1.5 mm in diameter and (2) 1.2 mm in diameter were placed (Figure 1). Half of the samples in all groups were randomly selected for TC ( $n = 10$ /per group).

To obtain a standard central position for posts in ZR/1.2/CON, ZR/1.2/TC, IPN/1.2/CON and IPN/1.2/TC groups, firstly, a post space preparation was drilled with a gates-glidden drill (1.2 mm diameter) at 10 mm height. Then, the post spaces for ZR/1.2/CON, ZR/1.2/TC, IPN/1.2/CON and IPN/1.2/TC groups as well as the rest of all other groups, were prepared with a reamer with 1.5 mm diameter up to a fixed depth of 9 mm from the CEJ.

All the prepared root canals were finally flushed with 2 ml NaOCl solution (5.25%), after which it was dried with paper points (Dentsply-Maillefer). IPN posts were sectioned to 9 mm by a sharp scissor through the manufacturer's recommendations. ZR post surfaces

were cleaned with alcohol, thoroughly rinsed with distilled water and air dried. Luting agent was injected into the root canal by using endo tips. All posts were luted with a self-adhesive luting agent (Clearfil SA Cement, Kuraray Medical Co., Osaka, Japan) according to the manufacturer's instructions. Following placement of the posts with slight pressure, in all groups, excess luting cement was removed. The luting agent was light cured with a LED light-curing unit (Elipar S10, 3M ESPE, St. Paul, MN) for 20 s in each of four directions (buccally, lingually and proximally). The output of the light was checked with a radiometer on the curing unit itself to ensure accurate light intensity. After the cementation procedures, the coronary part of the exposed dentin was completely covered with glass ionomer cement (Fuji IX, GC Corp., Tokyo, Japan) and the teeth were stored in distilled water for 7 days at 37°C. All specimens were prepared by the same operator.

Half of the samples were thermocycled in distilled water for 5000 cycles in a 5–55°C water bath. Each

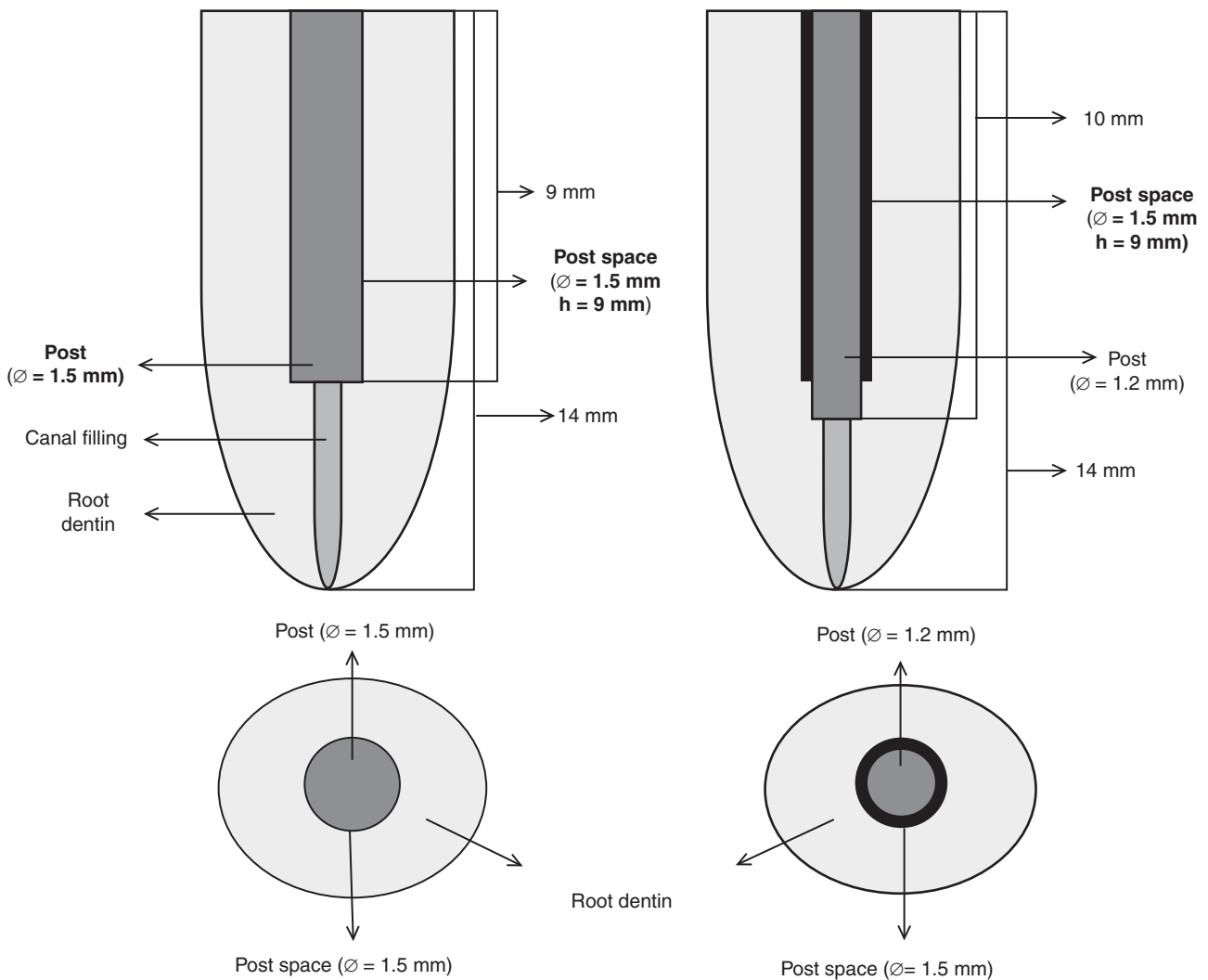


Figure 1. Illustration of specimen preparation.

cycle lasted 60 s: 20 s in a 5°C bath, 10 s to transfer samples to another bath, 20 s in a 55°C bath and 10 s to transfer samples back to the 5°C bath.

#### *Push-out bond strength test*

Bonded specimens were attached to the arm and were longitudinally sectioned into four slices ( $2.0 \pm 0.1$  mm thick) with a microtome (Leitz 1600 Microtome, Wetzlar, Germany) passing through the root under water cooling. Root sections demonstrating oval root canal form were discarded and replaced with another specimen prepared in accordance with the experimental protocol of that group. Each slice was marked on its apical side with an indelible marker and the thickness of each specimen was measured and recorded by a digital caliper (Liaoning MEC Group, Dalian, China) with an accuracy of 0.01 mm. Micropush-out ( $\mu$ -PO) tests were performed by applying a compressive load to the apical aspect of each slice via a cylindrical plunger mounted on a Universal Testing Machine (Lloyd LR 30K Plus; Lloyd Instruments Ltd, Fareham, UK). Each slice was placed ensuring that the coronal surface faced the plunger and that the post was centered over the hole of the plunger. The post segments were loaded with a cylindrical punch tip of 1 mm in diameter centered on the post segment. Care was also taken to ensure that the contact between the punch tip and the post section occurred over the most extended area, to avoid notching of the punch tip into the post surface. The load was applied to the apical aspect of the root slice and in an apical–coronal direction, so as to push the post towards the larger part of the root slice, thus avoiding any limitation to the post movement. Loading was performed at a crosshead speed of 0.5 mm/min until the post segment was dislodged from the root slice. A maximum failure load value was recorded (N) and converted into MPa considering the bonding area (A, mm<sup>2</sup>) of the post segments. Because of the cylindrical shape of both cemented posts, the bonding area was calculated using the formula [19]:

$$A = 2\pi r \times h$$

where  $r$  is the post radius,  $\pi$  is the constant 3.14 and  $h$  is the thickness of each post section.

#### *SEM evaluation*

Failure modes were examined visually using an optical microscope at  $\times 40$  magnification (Stereomicroscope, Wild M3B) and classified within four categories: adhesive failure between cement and dentin, adhesive failure between post and cement, cohesive failure within cement and mixed failure.

Two specimens typical of the failure modes from each group were prepared for scanning electron microscope (SEM) analysis. The specimens were sputter-coated (Bal-Tec SCD 050 Sputter Coater; Bal-Tec, Liechtenstein) with gold and observed with a SEM (JSM-5500; JEOL, Tokyo, Japan).

#### *Statistical analysis*

Three-factor analysis of variance (ANOVA) (SPSS, Chicago, IL) was used to test the effects of post materials, post diameters and thermal cycling ( $p < 0.05$ ). One-way ANOVA and Tukey's post-hoc tests were performed to determine the differences in  $\mu$ -PO results among the groups including assessment of possible interaction, which was used at a significance level of  $p < 0.05$ . Moreover, statistical differences in failure modes were investigated by chi-square tests at a significance level of  $p < 0.05$ .

## **Results**

Mean  $\mu$ -PO bond strength values (MPa) and standard deviations (SD) of the tested materials are shown in Figure 2. Three-way ANOVA revealed that both the post diameter and TC had significant effects on bond strength values ( $p < 0.05$ ). Additionally, the overall bond strength values of ZR and IPN posts were similar ( $p = 0.219$ ).

There were significant two-factor interactions between the post materials and the TC ( $p = 0.039$ ), as well as between the post materials and the post diameters ( $p < 0.05$ ). However, the interaction between TC and post diameters was not significant ( $p = 0.334$ ). Furthermore, significant interactions were observed between post materials, post space diameters and TC ( $p < 0.05$ ).

Statistical analysis demonstrated that 1.5 mm post diameter bond strength values ( $7.8 \pm 3.5$  MPa) were significantly higher than 1.2 mm post diameter values ( $6.2 \pm 3.3$  MPa) except the IPN-control group (Figure 2). Moreover, TC significantly increased bond strengths from  $5.1 \pm 2.7$  MPa to  $8.9 \pm 3.1$  MPa.

In addition, failure modes were significantly affected by post types ( $p < 0.05$ ), by TC ( $p < 0.05$ ) and by post diameters ( $p < 0.05$ ). IPN posts demonstrated more adhesive failure between dentin and cement and mixed failure patterns (adhesive failure between dentin and cement and cohesive failure inside the post). However, ZR posts showed adhesive failure between dentin and cement. In control groups, adhesive failure between dentin and cement was predominant for both posts, while mixed failure was typical in the TC groups. Posts with 1.2 mm diameter showed mostly adhesive failure between dentin and cement. Posts with 1.5 mm diameter demonstrated adhesive failure between dentin and cement and also mixed failure (Figure 3).

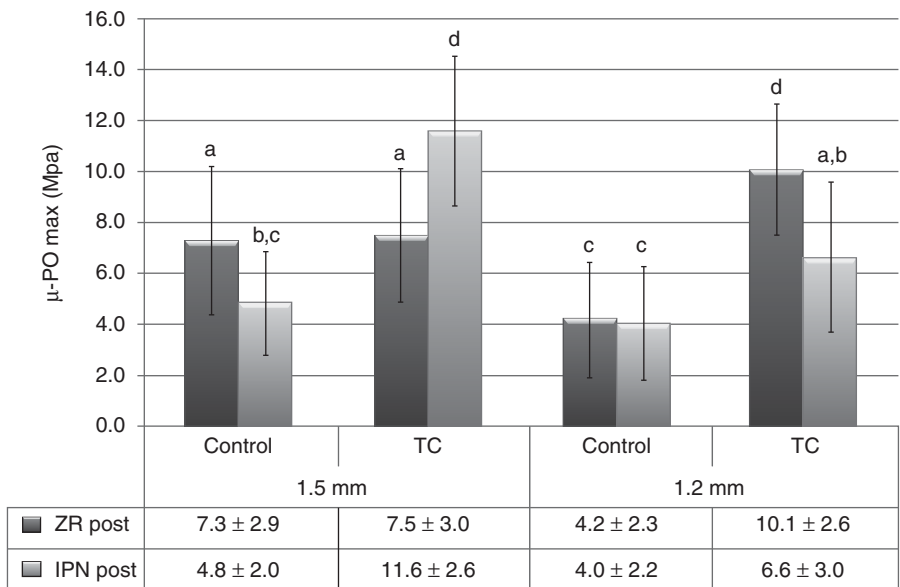


Figure 2. Micropush-out bond strength values in each group.

The SEM observations (Figure 4) after bond strength test showed typical failure modes in the ZR post group and the IPN post group.

**Discussion**

The push-out technique used in this study for testing the adhesion of various posts to resin cement systems was designed as a μ-PO test since the dimension of the specimens was reduced for the benefit of a more uniform stress distribution [19]. This method also has some limitations such as in relation to specimen

position and also the angle at which the load is applied could influence the final result [20]. However, it provides a better estimation of bond strength than the conventional shear test, as fracture occurs in parallel (not transverse) with the bonding interface, which simulates the clinical conditions [21]. Therefore, this test has been generally accepted for bond strength evaluation [19].

The results of this study demonstrated that different tooth-colored post materials did not affect the push-out bond strength, necessitating acceptance of the first null hypothesis. Overall μ-PO bond strength

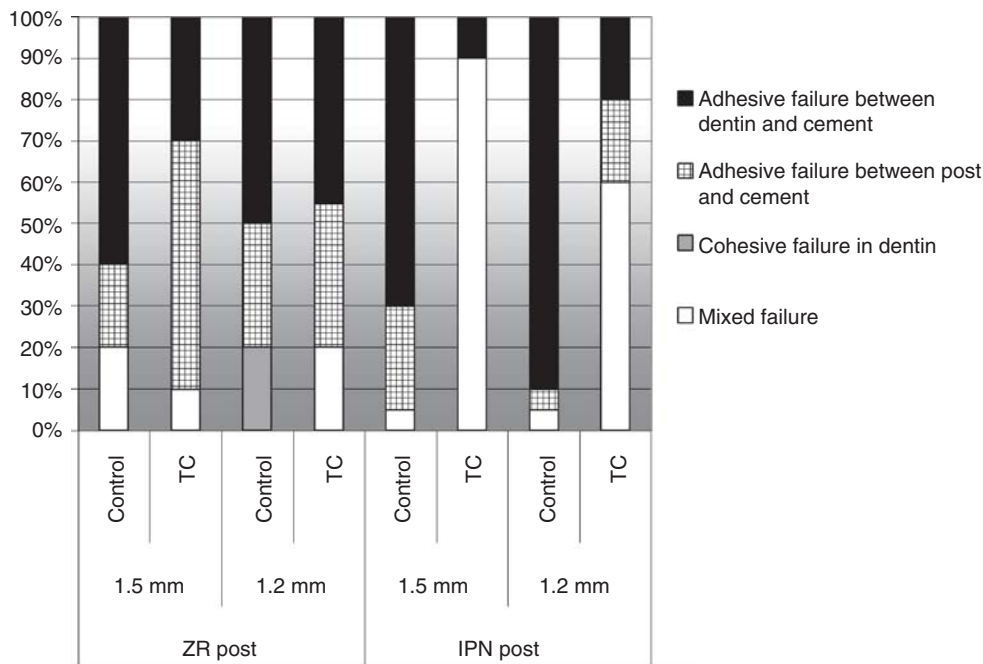


Figure 3. Distributions of failure modes of samples in each group.

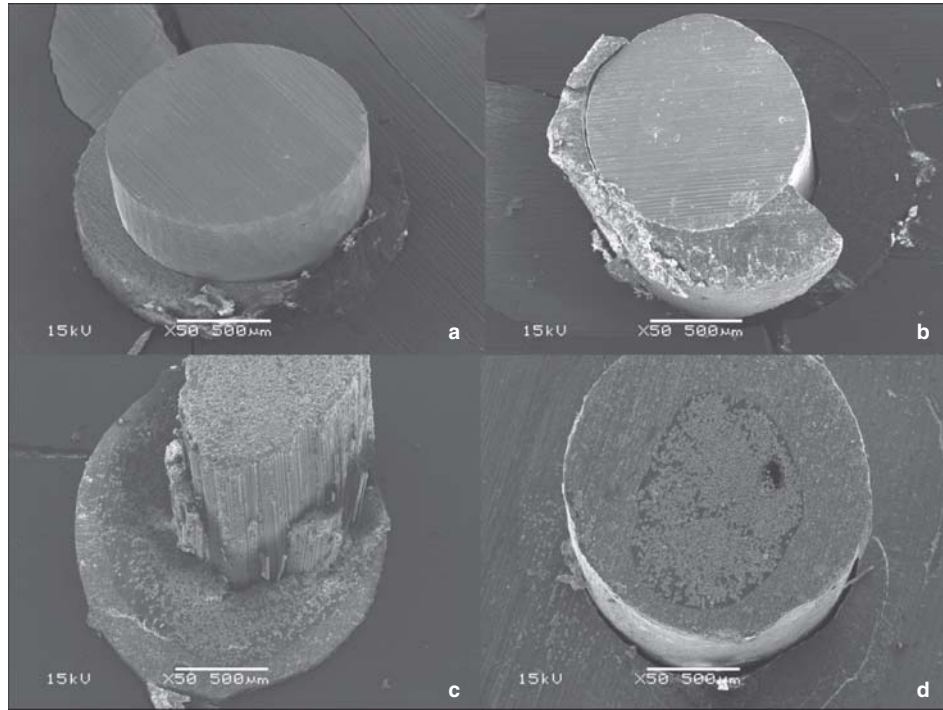


Figure 4. SEM-photomicrographs of typical failure modes of zirconia and semi-IPN posts after micro push-out tests (original magnification  $\times 50$ , bar = 500  $\mu\text{m}$ ). (A) Adhesive failure between post and cement of ZR/1.5/TC group. (B) Adhesive failure between cement and dentin of ZR/1.2/CON group. (C) Mixed failure pattern of IPN/1.5/TC group. (D) Adhesive failure between cement and dentin of IPN/1.2/CON group.

results indicated no significant difference between the mean  $\mu$ -PO values of ZR post ( $7.3 \pm 3.2$  MPa) and IPN post ( $6.8 \pm 3.7$  MPa). It is a well-known fact that individually formed posts with a semi-interpenetrating polymer network (semi-IPN) showed greater bond strength results to resin cements compared to pre-fabricated posts [8,22] because of its composition. On the other hand, for a satisfactory resin bond to zirconia, airborne-particle abrasion is a treatment choice to create micromechanical interlocking between a composite resin and ceramic surface [23]. Durable chemical bonding to zirconia ceramics has been obtained with MDP (10-methacryloyloxydecyl dihydrogen phosphate)-containing composite resins and with airborne-particle abrasion of the ceramic surface [24]. A previous study comparing ZR and FRC posts by means of push-out bond strength indicated higher bond strength values of FRC in comparison to ZR posts [25]. However, it should be pointed out that, in all of these studies, the tested cements were not self-adhesive resin cements. These results might be related with the luting cements and luting procedures. Moreover, in the current study, ZR posts received airborne-particle abrasion with 30- $\mu\text{m}$   $\text{Al}_2\text{O}_3$  particles, whereas no surface treatment was applied to IPN posts. Thus, for ZR post specimens, the major portion of bond strength might be related with the microretention of the surface roughness formed by the Cojet system. In addition to these factors, the low bond strength values obtained

with IPN post could be attributed to different cross-sections of this post, which was not always symmetrical and round, as opposed to the ZR post.

The aim of this investigation was to evaluate the accuracy of fit between the post and post space diameter on the  $\mu$ -PO bond strengths of ZR and individual IPN posts. The results of this study demonstrated that cement thickness affected the  $\mu$ -PO bond strength significantly, leading to the rejection of the second null hypothesis. Bond strength results of posts with 1.5 mm diameter ( $7.8 \pm 3.5$  MPa) were found to be statistically higher than the 1.2 mm post diameter results ( $6.2 \pm 3.3$  MPa). A previous study demonstrated that increased cement thickness surrounding the FRC post did not impair the bond strength [26]. Moreover, Perdigao et al. [27] implied that the thickness of the resin cement has no effect on push-out bond strengths of FRC posts. A previous study by D'Arcangelo et al. [14] assessed the effect of resin cement film thickness on the pull-out bond strength of FRC posts (0.9 mm in the apical diameter). They reported that bond strength was significantly enhanced in oversized post spaces (1–1.2 mm in the apical diameter) when compared with post spaces prepared with a matched calibrated drill (0.9 mm). However, the pull-out resistance of fiber-reinforced posts decreased when the cement film thickness was too great (post space: 1.4 mm). In accordance with that previous study, in the present study, when 1.2 mm diameter posts were placed in

1.5 mm diameter post spaces, the dislocation resistance of both ZR and IPN posts were significantly decreased. One of the reasons of these lower results obtained with the 1.2 mm diameter of both ZR and IPN posts in 1.5 mm diameter of post space might be related with the formation of bubbles or voids, representing the areas of weakness within the material, which is less likely to be seen in a thin and uniform layer of cement [14]. Moreover, a previous study by Grandini et al. [28] reported that the polymerization stress developed within a relatively thin film of cement would be minimal. Another factor that may play a role in lower bond strength values could be related with the C-factor which is defined as the ratio of bonded to unbonded surface areas. The prevalence of voids might have been reduced by using a lentulo and the deleterious effect of a high C-factor may actually be compensated by the stress relaxation provided by the air in the structure of the cement [27].

In this study,  $\mu$ -PO bond strength results of all tested groups were reflected on the failure pattern of debonded specimens as examined by an optical reflection microscope and confirmed by scanning electron microscopy. Although the force at the point of failure was similar, the failure mode and sight on the samples were different. This is important because it indicates the quality of bonding between post and cement or cement and dentin. Therefore, it can apparently influence the clinical longevity of a post-system. Failure mode analysis showed that typical failure mode was adhesive failure between dentin and cement for ZR posts, whereas more mixed failure was observed for IPN posts. This finding suggests that, although the compositions and surface structures of ZR and IPN posts were different, the differences in failure mode could be related with the bonding properties of luting agent. In the ZR/1.5/TC group, the failure pattern was mainly adhesive failure between post and cement. Also, in the IPN/1.5/TC group, the failure pattern was mainly mixed failure, indicating an increase in the bond strength that might be related with the post-curing effect of TC. Similarly, in the ZR/1.2/TC group, the failure pattern wasn't changed significantly, whereas a mixed failure pattern was predominant for IPN/1.5/TC group. Moreover, in the IPN/1.2/TC group, cohesive failures inside the post indicate that the bond between the post and cement seemed to exceed the strength of the material itself.

Another finding of the present study was that TC significantly affected the  $\mu$ -PO bond strength results of ZR and IPN posts. For both tooth-colored posts and for both cement thickness, TC groups exhibited significantly higher bond strength results than control groups, supporting rejection of the third null hypothesis. A previous study by Mazzoni et al. [29] found a significant reduction in retention and an increase in interfacial nanoleakage for posts luted

with two self-adhesive cements after 40,000 thermal cycles. Furthermore, Mazzitelli et al. [16] reported that bond strengths of two self-adhesive resin cements were not significantly affected by TC, while it increased the bond strength of another luting agent of the same category. Conversely, Bitter et al. [30] indicated an incremental increase in the retentive strength of posts luted with a self-adhesive resin cement after being submitted to 5000 thermal cycles. Similarly, in the present study, it was speculated that the thermal stress occurred during the thermal cycling test would act as a post-curing effect and could enhance the chemical polymerization of the luting cement and IPN post, promoting their complete setting reactions [16,30]. Although no differences were found between two tooth-colored post materials by means of overall  $\mu$ -PO bond strengths; statistical analysis revealed that the IPN/1.2/CON group gave the lowest  $\mu$ -PO bond strength results. However, the IPN/1.5/TC group and ZR/1.5/TC group gave the highest.

The direct exposure of the root to the different temperatures might have promoted the degree of monomer conversion of self-adhesive cement and composite-based individual-IPN post. Additionally, TC may have caused water sorption of self-adhesive cement and composite-based individual-IPN post, resulting in an expansion and in a better contact between the cement and dentin. This could be the cause of higher sliding frictional retention and consequently higher  $\mu$ -PO bond strengths of TC groups than non-TC ones.

The general outcome of this study suggested that fit between post space and post diameter and TC increased the bond strengths of ZR and IPN posts by improving the contact between cement and dentin. Thus, further evaluations, mainly clinical investigations, are necessary to support if the bonding ability would be increased with the use of matching post diameter in proper post space.

## Conclusions

Within the limitations of this study the following conclusions could be drawn:

- (1) Although it is controversial that a thin layer of cement can improve the quality of the luting procedure, the bond strength of tooth-colored ZR and IPN posts was significantly decreased when the cement layer was too thick.
- (2) TC drastically influenced the bond strengths of post materials in both post diameters. Future studies should investigate the post-curing like effect of bonding interface between self-adhesive resin cements and IPN posts with respect to the clinical long-term behavior of tooth colored post restorations of endodontically-treated teeth.

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**Declaration of interest:** The authors report no conflicts of interest. The authors alone are responsible for the content and writing of the paper.

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