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Effect of Resin Cement Porosity on Retention of Glass-Fiber Posts to Root Dentin: An Experimental and Finite Element Analysis

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The aim of this study was to evaluate the effect of porosity of self-adhesive resin on the stress distribution, post retention and failure mode of fiber post cemented to human root dentin. Ten human central upper incisors with circular root canal were selected. They were sectioned with 15 mm and were endodontically filled. The roots were scanned using micro-CT after post space preparation for root filling remaining evaluation. Fiber posts were cemented using self-adhesive resin cement (Rely X U200, 3M-ESPE). Two 1-mm-thick slices from the cervical, medium and apical thirds were scanned for resin cement bubbles volume measurements and submitted to a push-out test (PBS). Three operators using stereomicroscopy and confocal laser microscopy classified the failure mode. Stress distributions during the push-out test were analyzed using 3D finite element analysis. PBS values (MPa) were submitted to one-way ANOVA and Tukey's post hoc tests and the failure modes using the Kappa coefficient to assess inter-operator agreement. Chi-square test was used to determine significant differences between the methods ($\alpha = 0.05$). Push-out bond strength was significantly affected by the bubbles presence in all root depth ($p<0.05$). The stress concentration was higher when the bubbles were present. Adhesive dentin/resin cement interface failure was the most frequent type of failure. Confocal microscopy was better than stereomicroscopy for failure analysis. Bubbles generated during resin cement insertion into the root canal negatively affect the stress distribution and the bond strength. The use of confocal microscopy is recommended for failure analysis.

Key Words: fiber post, resin cement, push-out bond testing, micro-CT, confocal laser microscopy.

Introduction

The bond strength of resin cement and fiber post is a critical factor for the success of endodontic procedures. The dentin/resin and cement/fiber post interactions are influenced by several factors, like root canal contamination (1,2), effective polymerization of resin cement (3,4) and the integrity of the bonding interface (5). Post-dentin interface bond failure can increase the risk of failure of endodontic treated teeth (6).

The use of self-adhesive resin cement has been recommended to fix the fiber post achieving better bond strength to the root dentin (7,8). Due to the restrict root-canal geometry, any controlled application of resin cement is difficult (9). Remnants of post space preparation may negatively influence the post retention (10). As a consequence of cement application voids and bubbles are observed within the resin cement caused air entrapment (11). The bubbles and voids may reduce the resin cement contact area with the post surface and ultimately to retention the endodontic post. Micro-CT has been recently used in the endodontic field to analyze several factors linked to root canal preparation and filling procedures (12,13); however few studies have used it to analyze the restorative procedure in the root canal. The bond strength values are important for the studies involved with restorative procedures, however the failure mode classification is essential to qualify the factors that may interfere on the failure process. Push-out bond test failure analysis has been performed using stereomicroscopy (14), scanning electron microscopy (5) and confocal laser microscopy (10,14,15). Nevertheless, few studies have analyzed the efficiency of these methodologies and also the effect of bubbles on stress distributions.

Hence, the aim of this *in vitro* study was to evaluate the effect of porosity generated by resin cement application used for fiber post cementation on its retention to the root dentin and to test the efficiency of the method used to analyze the failure mode after push-out test. Two null hypotheses were: the bubbles volume would not affect the stress distribution on resin/cement interface and the post retention to root dentin irrespective of root region, and the method used to classify the failure mode after push-out test would not reflect on the efficiency of the analysis.

Material and Methods

Sample Selection and Root Canal Preparation

This study was approved by the local Ethics Committee (Protocol 227/09). Ten single-rooted human maxillary central incisors with similar root morphology were obtained from a pool of extracted teeth and stored in distilled water. The specimens were decoronated by transversally sectioning the roots at 15 mm from the apex with a double-faced diamond disc (KG Sorensen, Barueri, SP, Brazil) at a low speed with air/water spray coolant.

One trained operator prepared root canals. The root canals were enlarged using Gates-Glidden drills 2, 3 and 4, with diameters of 0.7, 0.9, 1.1 mm, respectively (Dentsply Malleifer, Petrópolis, RJ, Brazil). The apical end (1 mm) was left unprepared to prevent the apical extrusion of solutions and luting cement. The root canal was irrigated with 2.5% sodium hypochlorite solution (2.5% Chlorine Rio, São José do Rio Preto, SP, Brazil) and 17% EDTA (Biodynamics, Ibiporã, PR, Brazil) for 3 min. Roots were rinsed with 5 mL physiologic saline solution (NaCl) to remove the remaining debris. All root canals were filled with gutta-percha and a calcium hydroxide sealer (Sealer 26; Dentsply, Sao Paulo, SP, Brazil). Root canal openings were filled with resin modified glass ionomer cement (Vitremer; 3M-ESPE, St Paul, MN, USA), and the samples were stored at 37 °C in distilled water for 1 week. After this period, the canal relief was performed immediately after filling, using a heated instrument (GP heater; Dentsply Maillefer) to remove the gutta-percha to a depth of 10 mm (16). The post spaces were prepared to a depth of 10 mm with special preparation drills supplied by the manufacturer of the fiber posts (Exacto #3; Angelus, Londrina, PR, Brazil). Post used is tapered with a coronal diameter of 2.0 mm and an apical diameter of 1.1 mm. All procedures were performed by using an operating microscope (DF Vasconcelos, Rio de Janeiro, RJ, Brazil), with 10 × magnification.

Micro-CT Analysis

After post space preparation, the roots were mounted on a custom attachment, and scanned in a micro-CT scanner (SkyScan 1272; Bruker-microCT, Kontich, Belgium). The scanner operated at 100 kV and 111 mA (0.5-mm Al/0.038-mm Cu filter). The resolution used was 1632/1092 pixels - 10 µm. The scanning was performed by 360° rotation around the vertical axis with a rotation step of 0.6. Images of each specimen were reconstructed (NRecon v., Bruker-microCT) providing axial cross-sections of their inner structure. CTAn v. software (Bruker-microCT) was used for the 3-dimensional (3D) (volume, surface area and structure model index) evaluation of the root canal (Fig. 1). CTVol v. software (Bruker-microCT) was used for visualization and qualitative evaluation of the specimens (17).

Fiber Post Cementation

The fiber post was immersed in hydrogen peroxide solution at 24% for 1 min, dried with compressed air application for 1 min, followed by silane application for 1 min (Silano, Angelus, Londrina, PR, Brazil). All roots were dried with paper points and the fiber posts were cemented using handled mix self-adhesive resin cement (RelyX U200, 3M-ESPE). The resin cement was inserted into the root canal using K-file, the posts were covered with cement and slowly seated by finger pressure. The excess cement was removed after 1 min. After 5 min the resin cement was light-activated with a halogen light source at 800 mW/cm² (Demetron 501; Kerr Corporation, Orange, CA, USA). The specimens were stored in water at 37 °C for 7 days. The roots were submitted to a new micro-CT scan and reconstruction, applying the initial parameter settings described.

Push-out Bond Strength Test

The root cement fiber posts were fixed in acrylic plate of 20 mm X 20 mm with adhesive cyanoacrylate (Super Bonder, Loctite, SP, Brazil), and were transversely sectioned by using the water-cooled low-speed diamond saw (Isomet 1000; Buehler Ltd, Lake Bluff, IL), resulting in two 1.0-mm-thick slices from the apical, middle and coronal root regions. Load indenter tips of 1.5 mm and 2.5 mm base were used for the cervical and middle third and 1.0 mm tip and 2.0 mm base for the apical third. Diameter and thickness of the specimens were obtained by using a stereomicroscope and digital micrometer (Mitutoyo, Santo Amaro, SP, Brazil) with 0.01-mm accuracy digital camera. Each slice was submitted to the push-out bond strength test (DL500; EMIC, São José dos Pinhais, PR, Brazil), with the load applied in the apical-coronal direction at a crosshead speed of 0.5 mm/min. The maximum load at failure was recorded in Newtons (N) and converted into MegaPascal (MPa) by dividing the load applied by the bonded area (A), calculated by using the following formula: $A = \pi (R1 + R2) \surd R1 - R2) 2 + h2$; where π is a constant value of 3.14, r and R are the smallest and the largest radius, respectively, of the cross-sectioned tapered post, and h is the thickness of the section.

Failure Mode Analysis

To determine failure mode, three calibrated operators using two methodologies analyzed all specimens: a confocal laser-scanning microscope (Carl Zeiss Laser Scanning Systems, LSM510; META, Oberkochen, Germany) and stereomicroscope at 40× magnification, (Mitutoyo, Tokyo, Japan) (Fig. 2). Failures were classified into 1 of 5 categories: (1) adhesive between post and resin cement; (2) between resin cement and root dentin; (3) mixed, with resin cement covering partially of the post surface; (4) cohesive within the fiber post; and (5) cohesive within the dentin.

Statistical Analysis

The push-out bond strength and bubble volume values were tested for normal distribution (Shapiro-Wilk, $p<0.05$) and equality of variances (Levene test, $p<0.05$), followed by parametric statistical tests. Data were analyzed using one-way ANOVA and multiple comparisons were made using the Tukey post hoc test. The values of bond strength and bubble volume at the same region (cervical, middle and apical root regions) were correlated using Pearson correlation test. The Kappa coefficient was used to assess interoperator agreement and the chi-square test to determine significant differences between the imaging methods. All tests employed a 0.05 level of statistical significance and all statistical analyses were carried out with the statistical package SigmaPlot® System version 12.0 (Systat Institute Inc, San Jose, CA, USA).

3D Finite Element Analysis

Finite element models were created from an image of each slice (cervical, middle and apical) obtained through the Micro-CT scanning with and without bubbles and voids (Figs. 1G,1H and 1I). The *.STL file was used to create 3D meshed with 8 nodes, quadrilateral-hexahedral elements in Mimics and 3-Matric software (Materialise, Leuven, Belgium). To create the same models without porosity for each third, the empty spaces were totally filled with the elements. All structures and materials were considered homogeneous, linear-elastic and isotropic, except for the fiberglass post, which was considered orthotropic. The mechanical properties (elastic modulus (E), Poisson's ratio (υ) and shear modulus (G)) of dentin (E=18.60 GPa, υ=0.30) (18), resin cement (E=9.75, υ=0.30) (17), and the fiberglass post (E_x=9.5 GPa, E_y=9.5 GPa, E_z=37.0 GPa; υ_{xz}=0.27, υ_{xy}=0.34, υ_{yz}=0.27; G_{xy}=3.1 GPa, G_{xz}=3.5 GPa; G_{yz}=3.1 GPa) (19) were obtained from the literature. A load of 10 N was applied in the z direction (post's longitudinal direction), with the punch centered on the post. A nonlinear friction contact analysis was performed between the sample and the metallic punch/base (Fig. 1K). A custom-made subroutine (Fortran-based) was used to calculate the modified von Mises equivalent stress (MVM). The MVM takes in account the ratio between the compressive and tensile strength. The used equivalent stress was based on the well-known modified von Mises formulation, modified to take into account the difference between compressive and tensile strength for enamel, dentin, and composite. (20) The compressive/tensile strength ratios used were 37.3, 3.0, and 6.25 for the enamel, dentin, and composite, respectively (20).

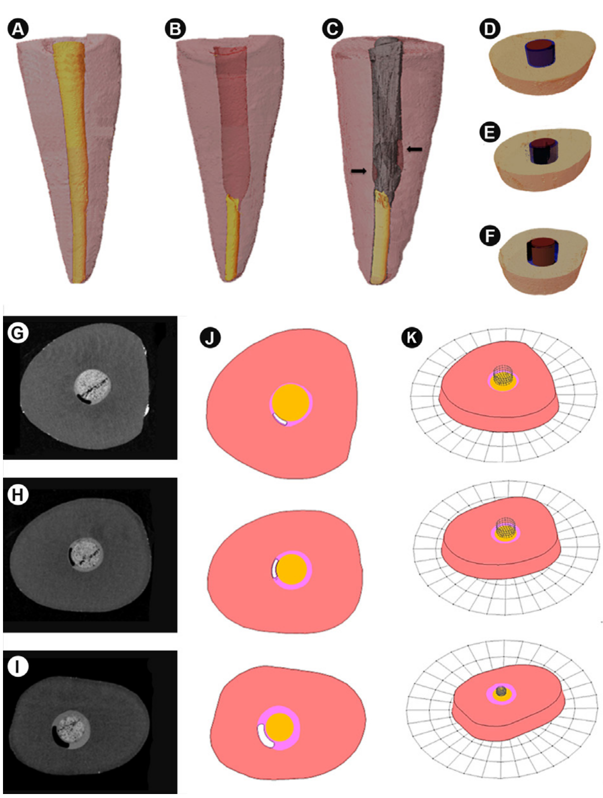

Figure 1. Micro-CT evaluation. A: root canal filling; B: relieved root canal; C: cemented fiber post; D, E and F: slices of three thirds, cervical, medium and apical respectively; G, H and I: slices showing the presence of bubbles on cervical, medium and apical regions respectively; J and K: 3D finite element models of each slice during the push-out test.

Results

Push-out test results and stress distributions are shown in Figure 3. Statistical analysis showed that the factor root

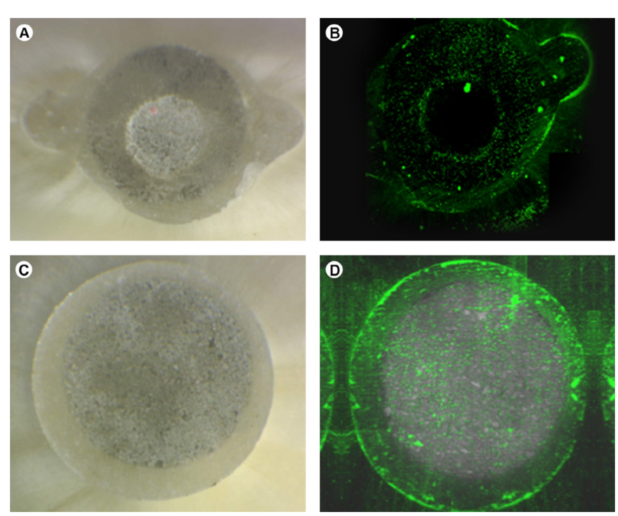

Figure 2. A and C: a stereomicroscope at 40× magnification at the apical region; B and D: a confocal laser-scanning microscope at the medium region.

region affects significantly the bond strength (p=0.03). Tukey test showed that PBS values were significantly higher in the cervical and middle thirds than in the apical region. The bubbles volume values were not affected by root region factor (p=0.723). The bubble volume values had high inverse correlation with push-out bond test on cervical (r=-0.828, p=0.002), middle (r=-0.810, p=0.003) and apical region (r=-0.703, p=0.016). A prevalence of adhesive cement/dentin interface failure was found, irrespective of root region. The stress concentration represented by modified von Mises stress increased on apical slice and on the bubble presence irrespective of region (Fig. 4). Kappa coefficient showed no difference of the inter-operator performance during failure analysis (0.91). The chi-square test showed that confocal laser microscopy had better performance than stereomicroscopy (p=0.022). The failure mode distribution is shown on Table 1. Adhesive dentin/resin cement interface failure was the most frequent type of failure, irrespective of root dentin region.

Discussion

The push-out test offers bond strength values from different locations of the root canal to detect regional differences using samples with minimal thickness (8). The push-out test would seem to be a more appropriate methodology for evaluation of fiberglass posts bonded to root dentin (15,18,19,21,22). For this performance some

Table 1. Push-out test failure mode distribution (n=20, per root third)

Third	Type 1	Type 2	Type 3	Type 4	Type 5
Cervical	1 (5%)	14 (70%)	3 (15%)	0 (0%)	2 (10%)
Middle	2 (10%)	13 (65%)	2 (10%)	1 (5%)	2 (10%)
Apical	1 (5%)	15 (75%)	2 (10%)	0 (0%)	2 (10%)

Failure modes: 1: adhesive between post and resin cement; 2: between resin cement and root dentin; 3: mixed, with resin cement covering partially the post surface; 4: cohesive within the fiber post; and 5: cohesive within the dentin.

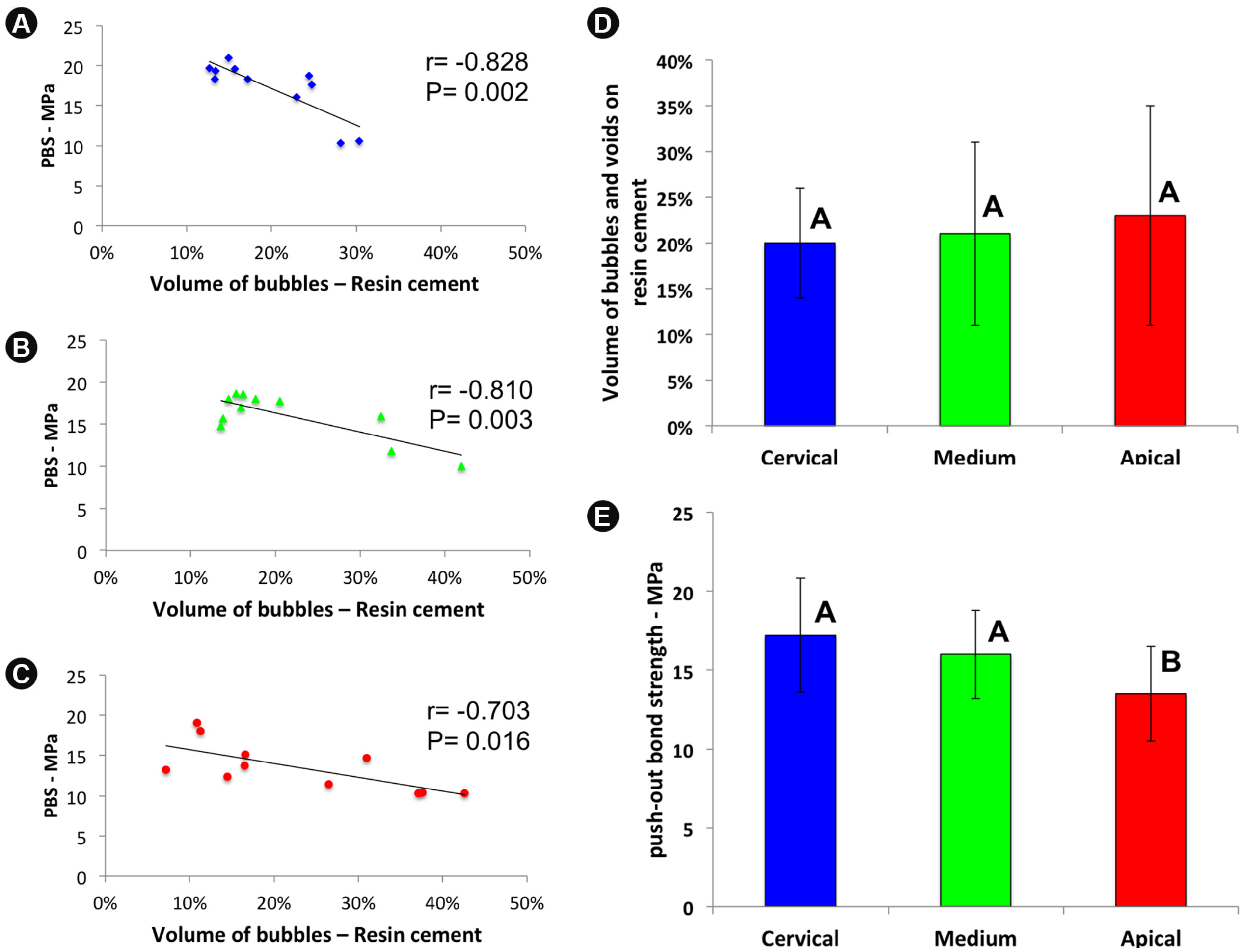

Figure 3. Push-out test results. A, B and C: Significant correlation between volume of bubbles on resin cement and push-out bond strength for cervical, medium and apical thirds respectively. D: Mean values and standard deviation of volume of bubbles on resin cement for root thirds; E: Mean values and standard deviation of push-out bond strength values for each root third, different letters means significant difference found by Tukey's test.

test parameters should be observed such as the punch and base orifice diameter, specimen thickness, which have effects on the push-out bond strength results (19,21,22). The association of the methodology is important strategy to better explain the biomechanical aspects involved in the restorative procedure (18,21,22). The results of the FEA analysis and the push-out bond strength values were very consistent. Nowadays for bond test analysis the correlation with the finite element analysis on tested sample is mandatory to explain the stress concentration and consequently the failure mode (15,18,21,22).

The PBS results obtained in this study showed significantly higher values in the cervical region and middle thirds than the apical. It could be attributed to the higher stress concentration of the apical slice and also because it was found better intimacy between the glass-fiber post and root canal walls at the cervical region. It could also be attributed to the limited light-transmitting ability of fiber posts reflecting in a reduction of the degree of conversion of fiber-reinforced composite with increased length of simulated root canals (3,23). The light intensity that reaches the cervical and middle thirds provided higher bond strengths to dentin (24). Furthermore the presence of smear layer generated during post space preparation, which is deposited on the root canal walls and jeopardize proper contact between the acidic methacrylates of self-adhesive resin cements and the underlying dentin during adhesive procedures, interfering with its bond strength (2). High polymerization shrinkage stresses have been reported in resin cements because of the high C-factor in the root canal (22). Under shrinkage effect, gaps occur when those stresses are higher than bond on adhesive interface. Gaps represent the potential local of bonding failure, reducing the PBS values. However, the delay of light activation (5 min) reduced significantly the shrinkage stress reducing this effect (19). When the cement is mixed by hand, air bubbles may be introduced. This can be observed in this study, which demonstrated that the bubble volume values had high inverse correlation with push-out bond test. Consequently, the use of analysis of correlations between the morphological characteristics of the root dentin-adhesive interface and bond strength might better explain the bonding performance of the resin cements to root dentin. When higher porosity presence was observed, higher stress concentration occurred on dentin/resin cement surface, and consequently lower bond strength was measured. It was more evident on apical region.

The bubbles and voids can be evaluated by micro-CT technology, which was able to nondestructively measure the volume of bubbles and voids within the cement layer at the dentin/cement/post interfaces (21,25). Micro-CT system using microfocal spot X-ray sources and high-resolution detectors allow projections rotated through multiple viewing directions to produce 3D reconstructed images of samples. Since the imaging process is nondestructive, the internal features of the same sample may be examined

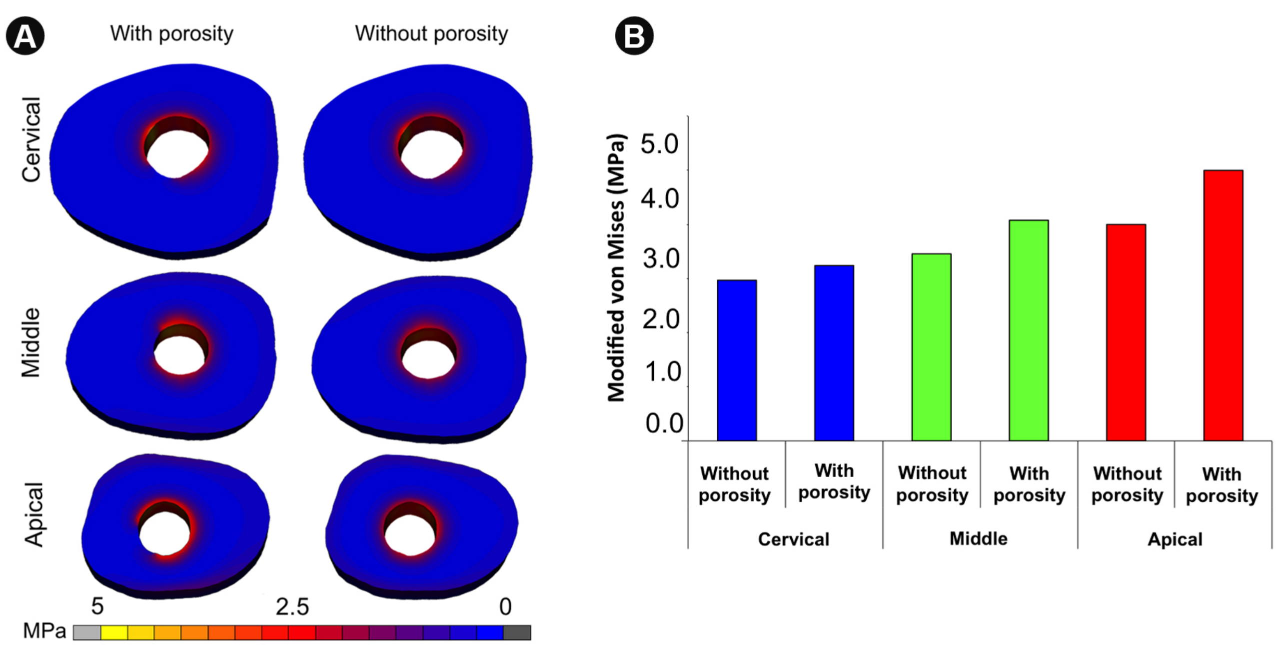

Figure 4. A: Modified von Mises stress concentration for cervical, middle and apical thirds, with and without porosity. Observed homogeneous stress distribution for samples without resin porosity and no stress on bubble's region and higher stress concentration on the remaining dentin interface compared. B: Peak of mvm stress values for each root region with or without porosity.

many times and samples remain available after scanning for additional bonding testing. Internal and external anatomy could be demonstrated simultaneously or separately. It is possible to analyze many aspects of the inner structure of a tooth. Therefore, data from micro-CT could serve as a basis for further analysis (26,27). Micro-CT combined with 3D reconstruction allows not only inspecting the internal arrangement rendered by fiberglass adhesively bonded to root dentin, but also estimating the volume of voids between the adhesive interfaces, allowing to obtain comprehensive imaging of the spatial organization of the specimen structures. The bubbles and voids may affect the longevity of adhesive bonding in two ways: first, voids can be located directly at the interface between dentin and cement, decreasing the contact bonded area. Second, as mechanical properties are highly dependent on flaw distribution and void formation, it would be expected that presence of voids decreases the strength of cement and creates sites for crack initiation and propagation as shown in this study. Third, because the bubbles presence acts as stress concentration factor, reducing the bond area and reflecting the higher peak of the stress on the remaining intact interface. The higher porosity on apical region is possible due to the difficulty of the resin cement insertion and the possibility of the air confined during post insertion. This aspect reflected on higher stress concentration, reducing the bond strength on this region.

A possible explanation for such a percentage of voids might be attributed to the cement mixing method, which may have introduced air bubbles into the material. The resin cements nowadays have been available in automix presentation that may reduce the bubble generation. Other explanation for bubbles and voids are the method used to introduce resin cement into the root canal (28). We used the more common method used by clinicians, inserting into the root canal using k-files and on the fiber post concomitantly. However the presence of the resin cement on both sites may increase the resin cement defects.

The most prevalent failure mode observed in this study reinforce the statement that the most sensible interface is between dentin and resin cement. It is the interface where the higher stress concentration is concentrated. The similar performance among three evaluators demonstrated that the training before the analysis is essential and also that the reproducibility of both methods used. The analysis of failure modes revealed that the confocal laser scanning microscopy (CLSM) had better performance than stereomicroscopy, which is in accordance with the results of previous investigation (15). These results show that the confocal analyses of the failure modes allow a more detailed description of failures as described previously and has been used in adhesive dentistry to visualize the micromorphologic characteristics of the dentin–adhesive interface (29). Although the failure mode analysis was conducted using mostly stereomicroscopy and confocal microscopy was used and appears to be a noteworthy alternative for the evaluation of the bond failure pattern in loaded specimens, since it is less time consuming and does not require any preparation of samples. However, its is necessary to consider that confocal is more time consuming, and it is a methodology much more expensive. More than method used for failure mode analysis is to perform this analysis.

Bubbles and voids generated during self-adhesive resin cement insertion into the root canal negatively affect the stress concentration on resin cement/dentin interface and reduced the fiber post bond strength. New studies should be developed using regular resin cement with different flowability and also testing different resin cement insertion methods into root canal. The use of confocal microscopy is recommended for failure analysis after push-out test. Therefore, the clinicians should take into account the method of the resin cement manipulation and application trying to reduce the resin cement/interfaces defect. When the push-out test was selected for testing fiber post bonding interaction with root dentin, the failure analysis is essential and the use of confocal methods tends to generate more predictable classification of the failure mode. In the absence of this method the use of stereomicroscopy is an acceptable alternative.

Resumo

O objetivo deste estudo foi avaliar o efeito da integridade do cimento resinoso autoadesivo, expresso pela presença de bolhas, sobre a distribuição de tensão, resistência adesiva e modo de falha de pinos de fibra cimentados à dentina radicular humana. Dez incisivos centrais superiores humanos com canais radiculares circulares foram selecionados. Os mesmos foram seccionados com 15 mm e tratados endodonticamente. As raízes foram digitalizadas utilizando micro-CT após preparo do pino para avaliação de remanescentes de material obturador. Pinos de fibra foram cimentados utilizando cimento autoadesivo (Rely X U200, 3M-ESPE). Duas fatias de 1 mm de espessura dos terços cervical, médio e apical foram escaneados para mensuração do volume de bolhas no cimento resinoso e submetidos ao teste de push-out. Três operadores classificaram o modo de falha utilizando microscopia confocal à laser e lupa estereoscópica. Distribuição de tensão foi analisada pelo método de elementos finitos 3D. Os valores de resistência adesiva (MPa) foram submetidos ao teste ANOVA em fator único seguido do teste de Tukey. Foi utilizado o coeficiente de Kappa para avaliar a concordância entre operadores. O teste Qui-quadrado foi utilizado para determinar diferenças significativas entre os métodos (α=0,05). A resistência adesiva foi significativamente afetada pela presença de bolhas independentemente da profundidade radicular (p<0,05). A concentração de tensão foi maior na presença de bolhas. Maior frequência de falha adesiva ocorreu na interface cimento/dentina. A microscopia confocal foi melhor do que estereomicroscopia para análise de falhas. A presença de bolhas afetou negativamente a distribuição de tensão e a resistência de união. Recomenda-se uso de microscopia confocal para análise de falhas.

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