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Durability of adhesion between an adhesive and post-space dentin: Push-out evaluation at one and six months

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SUMMARY

Aims: The aim of this study was to investigate, by means of the push-out test, the bond of Prime & Bond NT at various post-space dentin locations and the influence of time of water storage on bond strength values.
Methods: 30 single-rooted teeth were used for the bond strength measurement. In each tooth fiber posts were cemented with the commercial bonding system "Prime & Bond NT" in combination with the resin-based filling material "Opticore Cure Core Composite". After 24 h (group A: 10 roots), 1 month (group B: 10 roots), and 6 months (group C: 10 roots) of water storage, the specimens were sectioned in 1 mm-thick slices for the push-out test. The data were divided into three regions (coronal/middle/apical) and analyzed using Kruskal-Wallis Test and Mann-Whitney U Test ($p < 0.05$).
Results: The bond strength values registered did not show statistically significant differences within group A and C; within group B statistically significant differences were found between the coronal and the middle thirds and between the coronal and the apical third. The apical bond strength values did not show any statistically significant difference among the three groups (Kruskal-Wallis test); in the middle thirds a statistically significant difference was found at 6 months when compared with the 24 h and 1-month samples. In the coronal thirds was found a statistically significant difference between 1 month and 6 months. Analysis of the specimens under optical microscope revealed a prevalence of adhesive failures between fiber post and root dentin.
Conclusions: Bond strength values are lower at the apical third. Over time the adhesion of the fiberpost/luting cement/post-space dentin does not remain stable.

1. Introduction

Most clinical failures involving endodontically treated teeth restored with fiber post occur through debonding [1]. The bond strength between the resin luting cement and the post-space dentin is conditioned by the different distribution of the adhesive system along the post-space, by the anatomic and histologic characteristics of the root canal, including the orientation of the dentinal tubules [2], and by a non-uniform dentin hybridization in the apical third [3]. Furthermore, the control of moisture after the application and removal of phosphoric acid and the incomplete infiltration of the resin into the dentin significantly condition bond strength [4].

The long-term durability of the adhesive interface is an important factor that conditions the integrity of the restoration regarding all adhesives systems [5,6]. A direct water exposure of the resin–dentin interface for prolonged periods results in decreased bond strength, independent of the adhesive system used [6].

The aim of this study was to investigate, by means of the push-out test, the bond of a commercial adhesive system at various post-space dentin locations and the influence of water storage of 1 month and 6 months on bond strength values.

2. Materials and methods

2.1. Tooth preparation

30 single-rooted teeth extracted for periodontal reasons were selected and stored in 0.5 chloramine T [7] and used within 6 months from extraction. The crown surfaces of each tooth were sectioned below the cemento–enamel junction perpendicular to

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their long axis with a diamond bur under copious water cooling. The roots were endodontically instrumented with rotary Ni–Ti instruments Mtwo (Sweden & Martina, Padua, Italy). During the cleaning and shaping of the root canal, a rinse was performed at each change of instrument with 5% sodium hypochlorite (Ogna, Milan, Italy) and 10% EDTA irrigation (Ogna, Milan, Italy), using sodium hypochlorite for the last irrigation. The prepared root canals were obturated with manual vertical condensation with non-standardized gutta-percha points (Inline, Turin, Italy) and endodontic sealer (Pulp Canal Sealer EWT, Kerr, Romulus, MI).

2.2. Bonding of fiber posts

After 48 h storage in a saline solution, post spaces were prepared to a depth of 7 mm measured from the sectioned surfaces using a post drill of 1.2 mm diameter (Endoclass, IDS, Savona, Italy). Every canal was etched with 37% phosphoric acid (Total etch, Ivoclar Vivadent). The gel was introduced in the canal through a needle, and after 60 s the gel was rinsed with an endodontic syringe [7,8]. Excess water was removed from the post-space with a gentle blowing of air and with paper points, leaving the dentin slightly moist. A microbrush was used to introduce the adhesive (Prime & Bond NT, Kerr, Romulus, MI). After 20 s excess adhesive was removed through a clean microbrush. The adhesive applied was polymerized for 60 s with a halogen lamp (Coltolux, Coltene). The fiber posts (Endoclass, IDS, Savona, Italy) were acid etched with 37% phosphoric acid (Total etch, Ivoclar Vivadent) for 60 s [7]. After rinsing, the adhesive (Prime & Bond NT, Kerr, Romulus, MI) was applied with a microbrush, the excess was removed with a gentle blowing of air, and the adhesive was polymerized for 60 s with a halogen lamp (Coltolux, Coltene). The luting cement (Opticore Cure Core Composite, IDS, Savona, Italy) was injected into the canal with a Composite-Gun and an appropriate needle. The posts were then seated to full depth in the prepared spaces using finger pressure. The excess of the luting agent was immediately removed with a small brush. The resin luting agent was light polymerized for 60 s with halogen light (Coltolux, Coltene). A microhybrid composite (Enamel, Mycerium, Genoa, Italy) was used for the execution of a 2 mm build-up.

Specimens were then randomly divided and stored in the saline solution for 24 h (group A), 1 month (group B), and 6 months (group C).

2.3. Push-out test

Immediately prior to the respective periods of bond strength testing, the specimens were sectioned perpendicular to the long axis under water cooling with a low-speed saw (Micromet M; Remet Spa, Casalecchio di Reno, Italy). Six 1 mm-thick slabs were obtained per root and identified as cervical (two slabs), middle (two slabs), and apical (two slabs). Each slice was marked on its coronal side with an indelible marker.

For the push-out test each section was fixed with cianoacrilate (Super Attak Gel, Henkel Loctite Adesivi S.r.l., Milan, Italy) to a custom-made push-out jig, ensuring that the coronal surface faced the jig and the post was centered over the hole in the jig. The push-out jig was placed on an Instron 3344 universal testing machine with a cell load of 1000 N (Fig. 1). Load was applied at a crosshead speed of 0.5 mm/min until the post was dislodged. The maximum failure load (Table 1) was recorded in N and converted to MPa according to the following formula:

$$MPa = N/2\pi\, rXh$$

Statistical analysis was performed applying the Kruskal-Wallis Test and Mann-Whitney U test for the bond strength evaluation among root thirds ($p < 0.05$) and for bond strength evaluation at different storage time ($p < 0.05$).

After push-out testing, the specimens were analyzed by an optical microscope to determine the failure mode [10,27–29]: type I, adhesive between resin cement and fiber post; type II, mixed (failure occurring at resin cement/fiber post interface and at resin cement/dentin interfaces in the same specimen); and type III, adhesive between resin cement and root dentin.

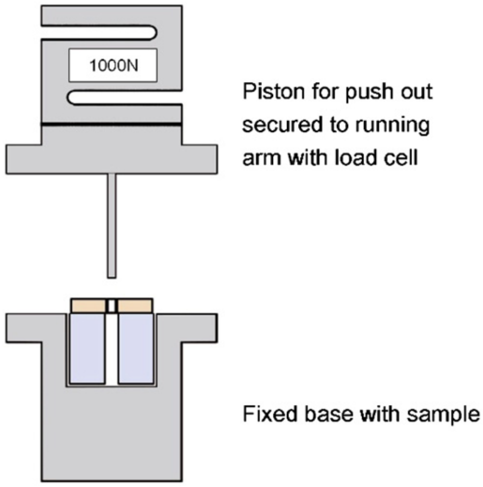

Fig. 1. Instron machine to evaluate the push-out strength at the three levels (coronal section, middle section and apical section of the root canal). The thin section of the root is positioned perpendicular to the direction of the push-out force.

Table 1
Mean bond strength, standard deviation and statistical significance.

	Coronal	Middle	Apical
24 h	9.81 ± 6.83 a1.2	5.74 ± 5.21 a1	4.82 ± 4.83 a1
1 month	9.67 ± 3.81 a1	5.65 ± 5.05 b1	2.33 ± 3.03 b1.2
6 months	7.24 ± 4.85 b2	2.03 ± 1.44 a2	2.39 ± 3.05 a2

Bond strength values are expressed in MPa. For each row: values with different letters indicate statistically significant differences ($p < 0.05$); for each column: different numbers indicate statistically significant differences ($p < 0.05$).

3. Results

For specimens that prematurely failed the bond strength was registered as zero and was not included in the statistical analysis. A general agreement on how to statistically treat the prematurely failed specimens during microtensile bond strength testing is presently lacking in the literature. They may be excluded, considered as zero or greater than zero values. In the present study, it was decided to exclude the prematurely failed slices to the statistical analysis.

The bond strength values registered did not show any statistically significance difference (Kruskal-Wallis test) within groups A and C (Table 1). Within group B bond strength values, a statistically significant difference (Kruskal-Wallis test) was found; the significant difference in this group (Mann-Whitney U test) was observed between bond strength values of coronal third and middle third and between coronal third and apical third (Table 1).

The apical bond strength values did not show any statistically significant difference among the three groups (Kruskal-Wallis test); the Mann-Whitney U test showed a statistically significant difference between 24 h and 6 months. The middle bond strength values showed a statistically significant difference among the three groups (Kruskal-Wallis test); the Mann-Whitney U test showed a statistically significant difference between 24 h and 6 months and between 1 month and 6 months. The coronal bond strength values did not show any statistically significant difference among the three groups (Kruskal-Wallis test); the Mann-Whitney U test showed a statistically significant difference between 1 month and 6 months (Table 1).

Analysis of the specimens under the optical microscope revealed that there was no adhesive failure between fiber post and resin cement. A prevalence of adhesive failures between fiber post and root dentin was observed.

4. Discussion

The object of this investigation was to compare the regional bond strength of the commercial adhesive Prime&BondNt with root canal dentin and determine the bond resistance of resin–root dentin to degradation after 1 month and 6 months of water storage.

Most *in vitro* post and core experiments have been accomplished evaluating the tensile force or pulling force to remove the post from the root canals [10]. However, this rarely occurs clinically. The microtensile method of adhesion testing permits a more uniform stress distribution along the bonded interface [11]. Furthermore, this technique enables the measurement of bond strength to very small areas and allows the assessment of regional differences of adhesion at the three levels of the root canals [12]. The trimming version presents an extremely high number of premature failures due to the uncontrolled stress applied on the bonded interfaces during sample preparation [13]. In non-trimming technique studies the measurement of bond strength has been conducted either between resin cement and just one side of the root dentin [12] or between fiber post and resin cement [1,14]. The push-out test seems to be the most accurate and reliable technique to measure the bonds of fiber posts to root dentin [13].

The push-out force increases with increased thickness of dentin disk [15]. The optimal thickness of dentin disk is still controversial, with values ranging from 4 to 1 mm [13,15–17]. In the present study 1 mm disks were used, since the use of thicker disks seems to increase the area of friction and may lead to an overestimation of the bond strength.

In agreement with the literature [9,18], the highest bond strength values of the tested material were found in the coronal samples in all groups. The premature failures occurred in the present study were observed in the apical thirds in all groups in the last apical slice. This may be related to a difficult accessibility of the middle and apical thirds of the root canal with a lower control of the humidity and a lower conversion degree [19]. Moreover, it has been shown that the control of moisture after the application and removal of phosphoric acid and the incomplete infiltration of the resin into the dentin significantly affect bond strength [20].

The best method for simulating *in vivo* conditions is yet to be determined [21]; the different aging methods of the adhesive interfaces, such as thermal stresses, chemical attacks, masticatory forces, or their combination, cannot actually replace the effects of time. The most commonly used artificial aging technique is long-term water storage. The bonded specimens are stored in fluid at 37 °C for a specific period that may vary from a few months [20] up to 4–5 years or even longer. The storage solution may be used alone [8] or impleted with antibacterial substances such as chloramine T [7], sodium azide [5], or thymol (Phrukkanon et al., 1999).

Degradation studies on adhesives interfaces have focused mainly on coronal [7,19], or cervical dentin [22]; the resin–dentin bond strength reductions and the resistance of the resin–dentin interface to degradation observed are dependent on the different adhesives systems tested [5], the degree of conversion [19], or the presence of a peripheral rim of resin bonded enamel [5,7].

A decrease in bonding effectiveness is usually supposed to be determined by hydrolysis of interface components (resin and/or collagen). Water can infiltrate in the resin matrix and reduce the mechanical properties of the polymer chains, in a process known as plasticization [23,24]. Furthermore, some interface components such as uncured monomers or break-down products can be eluted and can weaken the bond [25].

In agreement with the literature [20,26], the bond strength values of the tested adhesive system to radicular dentin observed over time showed a significant reduction.

In this study water storage was assessed without thermal and load cycling, for more similarities to the oral conditions it is recommended to perform fatigue test in combination with water storage.

5. Conclusions

Whitin the limitations of the present investigation it may be concluded that:

1) bond strength values appeared lower at the apical third.
2) the push-out strength of fiber post was significantly influenced by a time of water storage. Over time the adhesion of the fiberpost/luting cement/post-space dentin does not remain stable. Considering the fact that post–core complex is not directly exposed to oral fluids *in vivo* and that water exposure usually occurs after leakage: the times assessed could be attributed to longer times *in vivo*. However, the manner in which the fiber post in 1 mm-thick root slice is exposed to dislodging force during push-out testing cannot be directly compared with functional force that the post needs to withstand during clinical service.

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