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The adhesion between prefabricated FRC posts and composite resin cores: microtensile bond strength with and without post-silanization

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Summary *Objectives.* Aim of the study was to measure the adhesion between two types of translucent prefabricated FRC posts (FRC Postec, Ivoclar-Vivadent, FRC; Light-Post, RTD, LP), and two types of flowable composites used as core materials (UnifilFlow, GC, UF; Tetric Flow, Ivoclar-Vivadent, TF), with or without the application of a silane (Monobond-S, Ivoclar-Vivadent, S) on the post surface.

Methods. The experimental groups were: 1.1 FRC+UF; 1.2 FRC+S+UF; 1.3 FRC+TF; 1.4 FRC+S+TF: 2.1 LP+UF; 2.2 LP+S+UF; 2.3 LP+TF; 1.4 LP+S+TF. The bond strength at the interface between post and core was measured with the microtensile non-trimming technique. Thirty to thirty-five beam-shaped specimens per group were obtained from cylinders of core material, which had been built up around the post by progressively adding small increments of composite resin. Each specimen was loaded in tension until failure at either one of the two post-core interfaces present in each stick. The differences in interfacial bond strength among the groups were tested for statistical significance with the two-way ANOVA.

Results. The measured bond strengths in MPa were:

	FRC+UF	FRC+S+UF	FRC+TF	FRC+S+TF	LP+UF	LP+S+UF	LP+TF	LP+S+TF
Mean	9.05	11.11	10.74	12.88	8.36	12.22	7.87	13.43
SD	5.69	2.49	5.65	3.16	5.46	2.57	2.65	3.05

The statistical analysis revealed that post-silanization had a significant effect on adhesion ($p<0.05$). With any combination of post and core materials tested, the application of a silane onto the post surface prior to building up the core significantly increased the post-core bond strength.

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Significance. For improved adhesion at the interface between prefabricated FRC posts and composite resin cores, post-silanization is advisable.

Introduction

Prefabricated FRC posts are today widely accepted as a viable alternative to cast posts for the restoration of endodontically treated teeth [1].

Prefabricated FRC posts are adhesively luted inside root canals and, in case the loss of coronal structure is substantial, they provide retention to a core portion [2], which is directly built up onto the post with a composite resin.

For the core build-up procedure, a large variety of composite resin materials are available to the clinician, from packable to microhybrid to flowable composites [3].

Some laboratory and clinical evidence has recently been collected that supports the use of flowable materials for building up the core portion in prefabricated FRC post restorations [Monticelli et al., unpublished results]. When the structural integrity of the core material and its adaptation onto the post surface were evaluated through SEM observations of post-and-core units prepared in vitro, flowable composites exhibited better results than hybrid composites and composites marketed as core materials [Monticelli et al., unpublished results]. The use of flowables as core materials has also been validated by the findings of an in vivo trial, where post-and-core restorations thus performed gave proof of satisfactory clinical service over a 2-year follow-up period [Monticelli et al., unpublished results].

In addition to the microscopic appearance of the prefabricated FRC post-composite core interface, the strength of the adhesion between the two materials should also be assessed. In particular, it should also be verified whether the bond between prefabricated FRC posts and composite resin cores can be significantly enhanced by silanization of the post surface.

Silane coupling agents have been diffusely utilized in dentistry since the advent of glass-reinforced and resin-based materials.

In FRC post technology, glass or quartz fibers are coated with a silane in order to improve the adhesion at the fiber-resin matrix interface, protect fibers from damage during handling, modify the catalytic and wettability properties of fiber surfaces [4], and increase the chemical resistance of the fiber-matrix interface especially to water [5].

Fiber pull-out tests have been performed to measure the interfacial bond strength at the fiber-matrix interface [6]. On the other hand, limited information is available as regards the potential effect of silane treatment on the adhesion between the fiber post as a whole and the resin-based material with which the post is to interface, i.e. luting agents and core materials. Only one study has lately been published on the effect of different post surface treatments on the bond strength to resin cements [7]. Conversely, data are still missing regarding the influence of post-silanization on the bond strength to core materials.

This study was therefore conducted with the aim of measuring, using the microtensile non-trimming technique, the bond strength between two types of translucent prefabricated FRC posts and two types of flowable composites used as core materials, with and without silanating the post surface prior to building up the core.

The formulated null hypothesis was that the bond strengths achieved at the post-core interface with the various combinations of post material, core material, and surface treatment tested were not significantly different.

Materials and methods

Twenty-eight FRC Postec size 3 posts with a maximum diameter of 2 mm (Ivoclar-Vivadent, Schaan, Liechtenstein; Group 1), and 12 Light-Post size 2 posts with a 1.8 mm diameter (RTD, St Egève, France; Group 2) were used for testing.

FRC Postec posts feature unidirectional glass fibers (61.5% weight), embedded in a polymer matrix of triethylene-glycol-dimethacrylates (TEGDMA) and urethane-dimethacrylates (UDMA) monomers, in combination with highly dispersed silicon dioxide.

DT Light-Posts are made of unidirectional pre-tensed quartz fibers (60% volume), bound in an epoxy resin matrix.

On half of the posts from each group the surface was treated with a silane coupling agent (Monobond-S, Ivoclar-Vivadent, Schaan, Liechtenstein). Monobond-S contains 3-methacryl-oxypropyltrimethoxy-silane (MPS) as the effective silane (1% in weight), in a solution of ethanol (52% in weight) and distilled water (47% in weight), and has a pH of 4 [8]. Following manufacturer's instructions, Monobond-S was applied on the post surface with a brush. After having allowed for a 60 s contact at room temperature, the post surface was dried with air, and the core portion was built up onto the post with a flowable composite resin. UnifilFlow (GC, Tokyo, Japan) and Tetric Flow (Ivoclar-Vivadent, Schaan, Liechtenstein) were used for core build-ups. The following experimental groups were thus formed:

Group 1.1: FRC Postec posts and UnifilFlow ($n=7$);
Group 1.2: silanated FRC Postec posts and UnifilFlow ($n=7$);
Group 1.3: FRC Postec posts and Tetric Flow ($n=7$);
Group 1.4: silanated FRC Postec posts and Tetric Flow ($n=7$);
Group 2.1: Light-Post posts and UnifilFlow ($n=3$);
Group 2.2: silanated Light-Post posts and UnifilFlow ($n=3$);
Group 2.3: Light-Post posts and Tetric Flow ($n=3$);
Group 2.4: silanated Light-Post posts and Tetric Flow ($n=3$).

For the core build-up procedure, each post was positioned upright on a glass slab, and secured with a drop of sticky wax. Then, a cylindrical plastic matrix was placed around the post and adjusted so that the post would be exactly in the middle. The matrix was 10 mm in diameter. In height, the matrix was extended only to the cylindrical portion of the post (about 10 mm in Light-Post and 5 mm in FRC Postec posts), since, for appropriate cutting of the microtensile specimens, it is desirable that the post diameter be constant throughout the post length. The flowable composite was applied onto the post directly from the syringe in 1-2 mm thick increments, which were carefully adapted on the post surface and singularly cured for 20 s with a halogen curing light (Optilux 401, Kerr/Demetron, Orange, CA, USA intensity 750 mm W/cm^2). The material was always irradiated directly from the open upper side of the matrix and through the post. Never was irradiation done through the plastic matrix.

When the matrix was completely filled, the cylinder was taken off the glass slab, and a further 20-s irradiation was done on the side of the cylinder that had faced the glass slab, in order to ensure complete polymerization of the core material. At this point, the plastic matrix was cut-off, and the cylinder of composite resin built up around the post was separated (Fig. 1a).

The procedure of specimens cutting and loading was started immediately, as the intention was to quantify the bond strength reached by the materials around the time when clinically the procedures of core preparation, impression, and temporary crown adaptation and cementation are performed. It is in fact during these phases that the bond between post and core material is first stressed by vibrations, tensile and shear forces.

For cutting, each cylinder of material was secured on the holding device of an Isomet machine (Buehler, Lake Bluff, IL, USA; Fig. 1a). By means of a water-cooled diamond blade, two longitudinal cuts were then performed on two opposite sides of the post at its outermost periphery (Fig. 1b), so as to expose the post surface throughout its length. As a result, a slab of uniform thickness was created, that presented with the post in the center and the core build-up on each side (Fig. 1b). From the slab, 1-mm thick beams were then serially sectioned (Fig. 1c). Thirty to 35 sticks were obtained in each group. Every stick was glued with cyanoacrylate (Zapit, Dental Ventures of America, CA, USA) to the two free sliding components of a jig, which was mounted on a universal testing machine (Controls, Milano, Italy). This set-up was conceived to apply purely tensile forces to the two opposite post-core interfaces. Each specimen was loaded at a cross-head speed of 0.5 mm/min until failure occurred at either one of the two stressed interfaces. Bond strength was expressed in MegaPascals (MPa), by dividing the load at failure (Newtons) with the bonding surface area (mm^2). As the bonded interface was curved, its area was calculated using a mathematical formula previously applied by Bouillaguet et al. [9] for similar purposes.

Each failed specimen was observed with an optical microscope at 20 magnifications (Bausch&Lomb, Rochester, NY, USA), in order to classify the type of failure as adhesive between post and core, cohesive within post, or cohesive within core.

Statistical analysis

The two-way analysis of variance was applied with microtensile bond strength in MPa as dependent variable and type of post, core material, and silanization procedure as factors. The level of significance was set at $p=0.05$. The statistical analysis was processed by the SPSS 11.0 software (SPSS, Inc., Chicago, IL, USA).

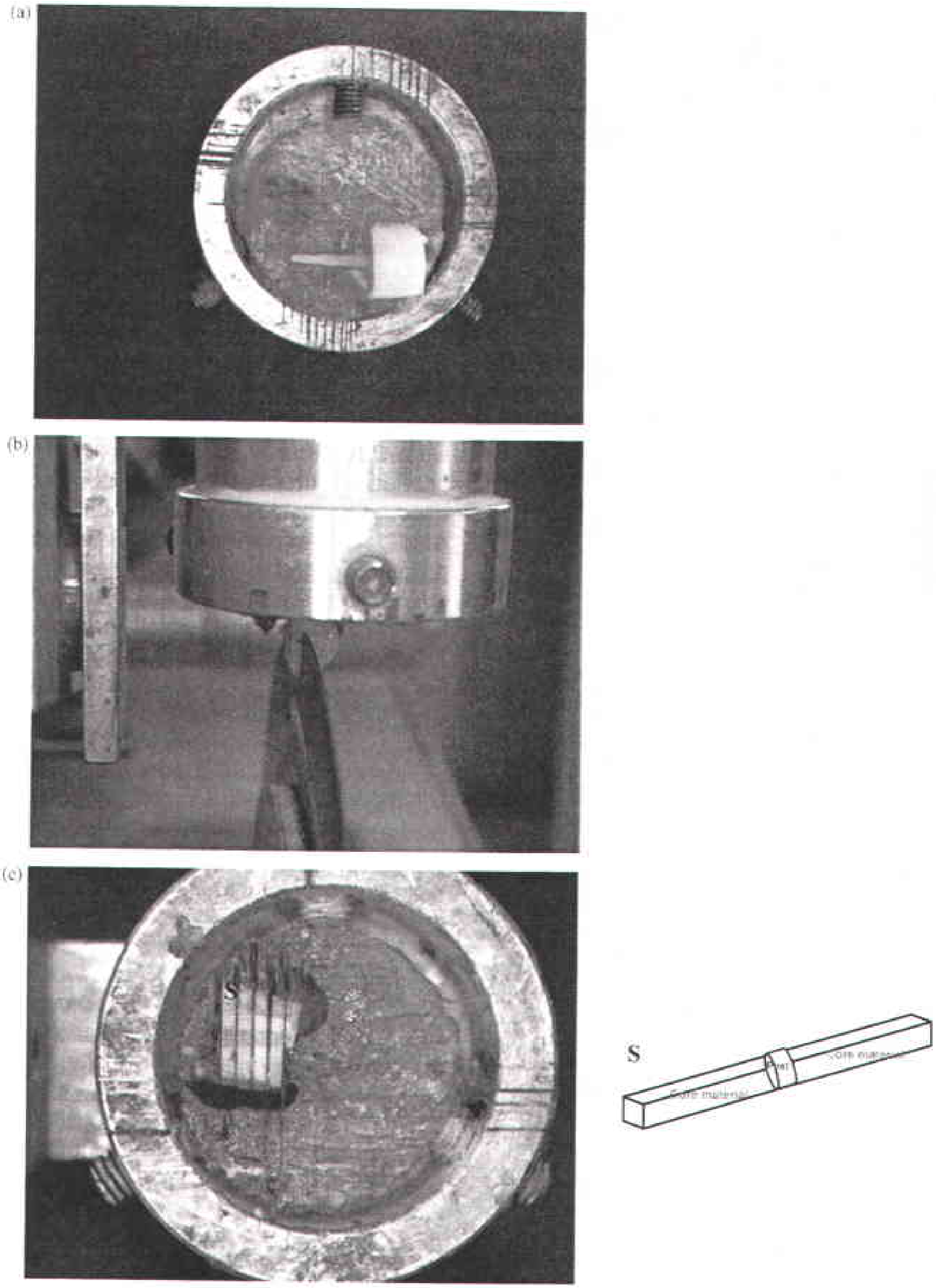


Figure 1 Procedure for the preparation of microtensile specimens. (a) A cylinder of core material was built up onto the glass fiber post by progressively adding small increments of composite resin. With two longitudinal cuts running at the periphery of the post, the post surface was exposed throughout its length. A slice exhibiting the post in the middle and the core material above and below was thus obtained. (b). From the slab, 1-mm thick beams were serially sectioned (c; s, stick).

Results

The mean and SD of the post-core microtensile bond strength values for the eight experimental groups are reported in Table 1.

Statistical analysis revealed that neither the type of post, nor the core material, or the interactions among the factors had a significant influence on bond strength at the post-core interface ($p > 0.05$). Only the post-silanization procedure had

Table 1 Mean and SD values of the microtensile bond strengths measured in all the experimental groups.

Core material		Type of post	
		FRC postec	Light-Post
Unifil Flow	No silane	Group 1.1	Group 2.1
	Mean (SD)	9.05 (5.69)	8.36 (5.46)
	Silane	Group 1.2	Group 2.2
	Mean (SD)	11.11 (2.49)	12.22 (2.57)
Tetric Flow	No silane	Group 1.3	Group 2.3
	Mean (SD)	10.74 (5.65)	7.87 (2.65)
	Silane	Group 1.4	Group 2.4
	Mean (SD)	12.88 (3.16)	13.43 (3.05)

The graph shows that with any combination of post and core material, post-silanization increased the interfacial bond strength (FRC, FRC Postec posts; UF, UnifilFlow; S, silane; TF, Tetric Flow; LP, Light-Post).

a significant effect ($p<0.05$). In other words, regardless of the type of post or core material used, the adhesion at the interface was significantly enhanced by post surface treatment with a silane coupling agent (Table 1, Fig. 2).

As regards the type of failure, in none of the loaded specimens did fractures develop within the post or the core portion. Failures always occurred adhesively at the post-core interface.

Discussion

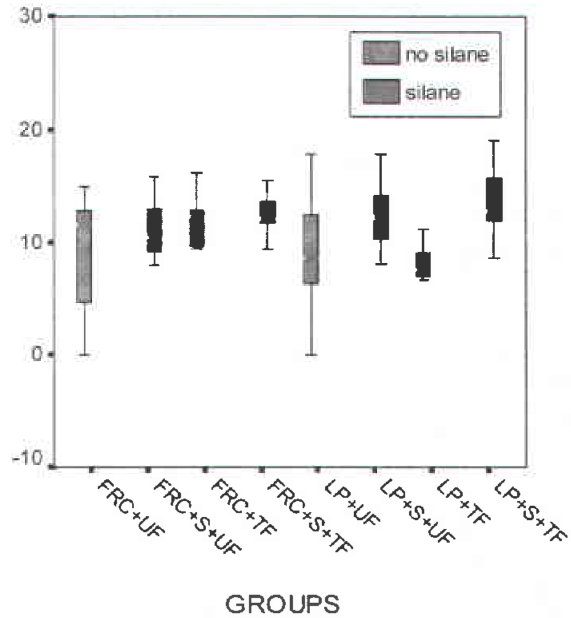
Adhesive posts restorations rely for their retention on the strength of the bonds established at different interfaces. Among them, the interface between root dentin and resin cement has been the object of several studies, involving both bond strength tests [9-14], and microscopic investigations [15-19].

Since the introduction of prefabricated FRC posts, a continuous effort has been made to improve the bonding potential of current adhesive systems inside root canals [10,11,13,17-19], as radicular dentin has been shown to offer far less favorable conditions for adhesion than coronal dentin and enamel [9]. As a matter of fact, the findings of clinical trials indicate that in the case of debonding of prefabricated FRC post restorations, an adhesive failure at the cement-dentin interface is involved most of the time [1].

Although, it is then the root-cement bond which represents the weakest link, the post-cement and post-core interfaces also deserve attention.

In particular, it is from the strength of the chemical and micromechanical interaction between a fiber-reinforced material and a composite resin that the retention of the core portion onto the post depends. This bond has to rapidly reach levels of strength sufficiently high to resist the stress transmitted during core trimming and adaptation of the provisional crown.

In this investigation, the adhesion of two flowable composites to two types of prefabricated FRC posts were assessed with the microtensile



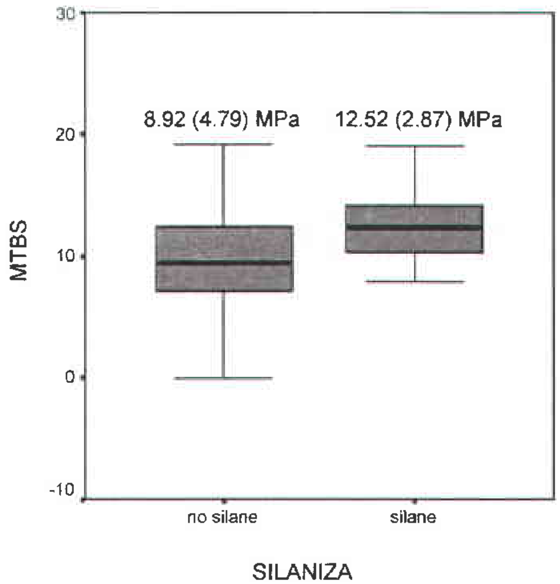


Figure 2 The graph reports the mean and SD (in parentheses) values calculated when the bond strengths of all the silanated and non-silanated posts were pooled together, regardless of the post and the core material used. Significantly higher bond strengths were measured after silanization ($p<0.05$), and the variability of the data was lower than that observed with non-silanated posts.

technique at completion of the core build-up procedure. The most relevant finding of the study was that with any tested combination of post and core material, the interfacial bond strength was significantly enhanced if the post surface had been preliminarily coated with a silane coupling agent. The increase in bond strength as a consequence of post-silanization was more remarkable with Light-Post than with FRC Postec posts (Table 1).

On the other hand, since the statistical analysis revealed the post and core materials interaction to be non-significant, it can be inferred that using a prefabricated FRC post and a composite resin from the same manufacturer does not result in a significantly enhanced interfacial bond strength. As a matter of fact, the adhesion achieved by Tetric Flow on FRC Postec posts was not significantly stronger than that established by UnifilFlow on the same type of posts (Table 1).

According to recently published data [7], treating the post surface with a silane coupling agent is advisable also to enhance the adhesion of the resin cement used for luting. Beside silanization, also post sandblasting and the combination of this with silane coating were found to significantly increase the bond strength of resin cements to glass fiber posts [7]. It may then be of interest to verify whether and also to what extent the adhesive potential of the composite core can be improved with these procedures of post surface treatment.

In this study, the silane agent was used at room temperature. However, it may be worth testing whether heat treatment of the silanized post further adds to the FRC post-core bond strength, similarly to what has been shown for the porcelain-composite bond [20].

Different theories have been proposed in order to explain the bonding mechanism through silane coupling agents. According to the oldest of these, the chemical bonding theory, the coupling action of the silane involves the formation of covalent bonds from the reaction of the organo-functional group (R) and the hydrolyzed alkoxy groups $(R'O)_3$, respectively, with the resin matrix and the mineral substrate (glass or silica) of the composite material [21]. Today, the reversible hydrolytic bond mechanism theory is more widely accepted as, although retaining some concepts of the chemical bond theory, it provides a better explanation for the hydrolytic stability of bonding through silanes. The theory states that the bonds between silane and mineral substrate are reversibly broken and remade in the presence of water, allowing for stress relaxation without loss of adhesion [21].

As the silane agent is only able to chemically bridge resins and OH-covered inorganic substrates, at the fiber post-composite core interface the chemical bond is possible only between the resin of the core material and the exposed fibers of the post. On the other hand, the highly cross-linked polymers of the matrix in FRC posts do not have any functional group available for reaction.

In FRC materials such as everStick (StickTech, Turku, Finland), an attempt to solve the problem of adhering to highly cross-linked polymers has been made by using semi-interpenetrating polymer network (IPN) structures [22]. In this technology, the fibers are preimpregnated with a polymethylmetahcrylate (PMMA), that can be partially dissolved with the application of a light-curing resin for 5 min. As a result of the partial dissolution at the surface of the fiber frame, grooves and undercuts are created where micromechanical bonding can be established in addition to the chemical adhesion. According to the manufacturer, the post surface is thereby 'reactivated' to offer considerably more favorable conditions for adhesion to the core or the luting material [23].

The prefabricated FRC posts tested in this study do not contain semi-IPN structures. Since the contribution of the chemical bond in coupling post and core materials through silanes can be expected to be low, the mechanism most likely involved in the enhancement of the post-core bond seen in the present study can be identified in the improvement in post surface wettability following silane coating.

The surface wetting theory recognizes a key role to the wetting capacity of the silane for improved adhesion. According to this theory, the silane, thanks to its low viscosity, would assist substrate wetting, and once an intimate contact between the interfacing materials is established, also van der Waals' forces would become effective, providing a physical adhesion, which contributes to the chemical reactions [5].

Although the results of this study provide clear evidence that silane coating of the post surface increases the post-core bond strength, some uncertainty remains around the mechanism actually responsible for the enhancing effect. Matinlinna et al. [8] have come to a similar conclusion regarding the use, in general, of silane in dentistry in their recent extensive literature review on dental silanes: although the majority of clinical results indicate a significant role of silanes in the adhesion process, however, the silane reaction mechanisms still remain not fully understood.

To testify to the action of the silane as a post-core adhesion promoter in this microtensile study was not only the recording of higher values of bond strength after silanization, but also the finding of no premature specimen failures with silanated posts. On the other hand, few specimens from untreated posts failed during cutting or adhesion, and were taken into account in the statistical calculations as zero values. The inclusion of 'zero bonds' may have contributed to the greater spread of the bond strengths of non-silanated posts. Also, the finding of more limited SDs in Groups 1.2, 1.4, 2.2, and 2.4 may be interpreted as the indication that with post-silanization a more uniform bond with the core material is developed. Silane coupling agents are claimed to exert other favorable actions in interfacial adhesion. They are believed to increase the resistance of the bonds to chemical dissolution, particularly from water [5]. In addition, due to their elastic properties, silanes would be able to absorb the stress that may develop at the interface as a result of differences in thermal expansion coefficients of the interfacing materials [21]. Unfortunately, these supposed effects of silanization could not be assessed in this investigation. As microtensile bond strength tests were performed immediately after specimen preparation, no inference can be drawn from this data regarding the durability of the bonds. In order to investigate this aspect, a fatigue test, possibly involving thermocycling, could be performed on post and core units prepared in vitro, and a further validation of the results could be provided by a longitudinal clinical trial of adhesive post and core restorations performed with and without post-silanization.

As regards the materials on trial, although the manufacturers of FRC Postec and Light-Post posts recommend the use, respectively, of a microhybrid composite, Tetric Ceram, and a core material, Lumiglass, to build-up the abutment portion, in this study it was decided to test two flowable composites as core materials. This choice was suggested by the findings of a previous microscopic investigation, where the abutments prepared with Tetric Flow and UnifilFlow exhibited the highest structural homogeneity and the best adaptation on the post [Monticelli et al., unpublished results].

It should be pointed out that on carbon FRC posts silanization would not be so effective at enhancing the post-core bond as it is on glass FRC posts. Since carbon fibers do not have a significant number of hydroxyl groups on the surface, their reaction with silanes is improbable [5].

The microtensile technique in this trial was adopted as it is currently regarded as the most reliable method for bond strength testing [24]. The small size of the specimens is the condition for a more uniform distribution of the stress on loading, which limits the chance of cohesive failures, thus allowing for an accurate assessment of the interfacial bond strength [24,25].

In particular, the non-trimming variant of the technique was chosen as there are indications in the literature that it is less aggressive than the variant which involves trimming the specimen to an hourglass shape at the bonded interface [24,26]. As a matter of fact, in this trial very few specimens broke prematurely, whereas in a previous attempt to apply the trimming method to measure the bond strength between post and core materials, a great number of premature failures were recorded, which considerably elevated the SD of the data set [27].

Alternatively, the push-out technique could be applied to measure the post-core bond strength, provided that, as done in microtensile, the specimen size is kept small for improved stress distribution [26], thus performing what could be called a 'micro-push-out test'. Practically this would involve cutting the cylinder of post and core build-up into 1 mm thick slices, from which the post portion is then extruded by means of an appropriately sized loading punch.

Conclusion

The results of this microtensile study support the use of a silane agent as an adhesion promoter at the interface between translucent FRC posts and composite resin cores. The exact mechanism by which this enhancing effect takes place remains not fully understood.

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