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Microtensile bond strength of resin-post interfaces created with interpenetrating polymer network posts or cross-linked posts

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Abstract

Objective: The purpose of this study was to evaluate the microtensile strength of composite bonded to interpenetrating polymer network (IPN) or cross-linked glass fibre posts and to observe the failure modes by using light and scanning electron microscopy. **Methods:** Twenty posts containing IPN resin matrix and 20 posts containing cross-linked epoxy polymer matrix were used for testing. One half of the posts from each type was treated with Stick Resin, the other half was treated with OptiBond. Composite resin was used to build up a block on the bonding surface. Tensile strength data was analysed statistically using the non-parametric Kaplan-Meier survival analysis. The distribution of failure modes as a function of post type/bonding agent was evaluated using the χ^2 test. **Results:** The mean tensile strength values were lower for the groups bonded with OptiBond and higher for the groups bonded with Stick Resin ($p = 0.017$), the type of post used had no statistical significance ($p = 0.263$). All the IPN posts showed cohesive failure within the post The cross-linked posts demonstrated a higher number of adhesive failures and lower number of cohesive failures within the post ($\chi^2 = 0.0001$).

Conclusions: Stick Resin was more effective than OptiBond in bonding composite cores to fibre posts. Post fracture was the failure mode of IPN posts, debonding of the composite core was the failure mode of most of cross-linked posts. These different failure modes may appear clinically in endodontically treated teeth restored with the post types tested in this study.

Key words: Fibre posts, microtensile test, resin composite.

Introduction

The restoration of root filled teeth has changed considerably in recent years. Dentin bonding systems, resin composites and fibre posts have widely replaced cast posts and amalgam as core materials. Fibre reinforced composites (FRC) are considered as viable alternatives to metals when strength, stiffness and resistance to corrosion are required (1) in the restoration of root filled teeth. Their properties depend on type, direction of fibres and nature of the matrix. Addition of fibres to a polymer matrix can improve significantly its physical and mechanical properties. Fibres added to the polymer matrix may be woven polyethylene, glass, carbon, silica or quartz. The properties of fibre reinforced materials can be summarized as follows (2): high impact resistance, attenuation and softening of vibration, shock absorption and increased fatigue resistance.

Many different types of fibre reinforced endodontic posts are now available. Fibre posts have been introduced also to achieve optimal aesthetics. Quartz and glass fibre posts may be translucent and potentially allow the use of light curing for cementation.

The adhesion between the fibre posts and the composite materials used for cementation, and between the composite and restoration may be relevant for the long-term success of fibre-post restorations (2).

An interpenetrating polymer network (IPN) is a combination of two polymers in network form. Most IPNs are heterogeneous systems comprised of one rubbery phase and one glassy phase, which produce a synergistic effect yielding either high impact strength or reinforcement.

Ever Stick posts (Stick Tech, Turku, Finland) are made of a semi-IPN polymer, having silanated glass fibres impregnated with an IPN resin matrix. Their linear polymer phases and the cross-linked polymer phases are not bonded chemically together as is the case for a typical copolymer (3). During the contact time of this type of post with the bonding agent, the monomers of the bonding resin diffuse into the linear phases of the IPN polymer matrix, and with subsequent polymerization, they become interlocked. Ever Stick FRC contains polymethylmethacrylate (PMMA) as a linear phase and poly bis-GMA as the cross-linked phase of the polymer matrix. PMMA chains, with molecular weight of 220 KD, plasticize the cross-linked bis-GMA based matrix of the Ever Stick FRC and thus reduce the stress formation in the fibre matrix.

The vast majority of root canals have an irregular, ovoid shape in their coronal and middle thirds. The consequent lack of adaptation of the post to the root canal walls may result in the inability of the luting agent to fill the post-tooth interface completely. This might cause the decementation of the post. When using fibre posts decementation is the most frequent cause of failure (4). IPN posts can be adapted easily to the shape of the root canals, thereby possibly reducing the number of voids and potentially reducing the number of post decementations.

The bonding to resin composite luting cement of four types of prefabricated FRC posts with a cross-linked polymer matrix and two types of FRC posts with semi-IPN polymer matrix was investigated utilizing a pull-out universal testing machine (5). The IPN posts gave significantly higher pull-out force values than that of the cross-linked FRC posts. A recent study investigated the degree of penetration of two different bonding resins (Scotchbond Multi Purpose Plus and Stick Resin) applied for different contact times on IPN posts (Ever Stick) and cross-linked posts (6). Measurement of penetration of bonding resins into sections of the posts was performed using a confocal scanning microscope. The statistical analyses proved that there was some resin penetration into the IPN posts. The prolonged contact time of bonding agent on the surface of the post increased the resin penetration.

The bond established by the composite core material with the post has been found to be weaker than the bond between the composite and the coronal dentin (7); therefore, it is desirable to develop post and adhesive systems which demonstrate increased bond strength values.

The purpose of this study was to evaluate the microtensile strength of composite bonded to interpenetrating polymer network (Ever-Stick, Stick-tech) or to cross-linked glass fibre posts (Easy Post, Maillefer, Baillagues, Switzerland) and to observe the failure modes at the resin-post interface of the tested specimens.

The null hypotheses tested in this study were (1): there was no significant difference in the bond strength at failure between the four groups (2); failure mode, post and bonding agent type were independent.

Materials and Methods

A total of 40 posts with a diameter of 1.2 mm were used for testing; 20 posts containing IPN resin matrix (Ever Stick post, Sticktech), (Fig. 1a), and 20 posts containing cross-linked epoxy polymer matrix (Easy Post, Maillefer), (Fig. 1b). One half of the posts (n=10) from each type was treated with Stick Resin bonding agent (Stick Tech), whereas the other half was treated with Optibond Solo (Kerr, Glendora CA, USA). The components of the posts and adhesive systems used are summarized in Table 1.

The two post groups were treated according to the manufacturers' instructions. The IPN posts were light polymerized for 60 seconds using a quartz-halogen-tungsten curing unit (Optilux 401, Kerr/Demetron, Orange, CA, USA), with a light intensity of 750 mW/cm². A layer of light curing resin adhesive was applied on both types of posts with a brush. The IPN posts were kept under a light shield for 5 minutes. The light protection was used to prevent polymerization of the light curable resin and to allow the monomers of the resin to diffuse into the polymer matrix. The layer of bond was then thinned by gently blowing the surface of the posts with dry air and polymerized for 10s with the light curing unit.

Table 1. List of materials used in the study

Brand	Manufacturer	Chemical composition	Material type
EverStick post	Stick Tech, Turku, Finland	E-glass, PMMA, Bis- GMA	Resin-preimpregnated continuous unidirectional FRC
Stick Resin	Stick Tech, Turku, Finland	Bis-GMA, TEGDMA	Unfilled resin
Easy post	Dentsply-Maillefer, Baillagues, Switzerland	Zirconium-Enriched Glass Fibers, Epoxy Resin Matrix	Resin-preimpregnated continuous unidirectional FRC
Optibond Solo	Kerr, Glendora, CA, USA	Ethyl alcohol; Bis-GMA; HEMA; GPDM; photoinitiators; barium aluminoborosilicate glass; fumed silica (silicon dioxide); sodium hexafluorosilicate	Dentine bonding agent
Herculite XRV	Kerr, Glendora, CA, USA	Ba-glass filler, Bis-GMA, TEGDMA	Microhybrid composite

PMMA = Polymethylmethacrylate; Bis-GMA = 2, 2-bis (4-2-hydroxy-3- methacryloylxpropoxy)phenyl-propane; TEGDMA = triethylene glycol dimethacrylate; HEMA = 2-hydroxyethyl methacrylate

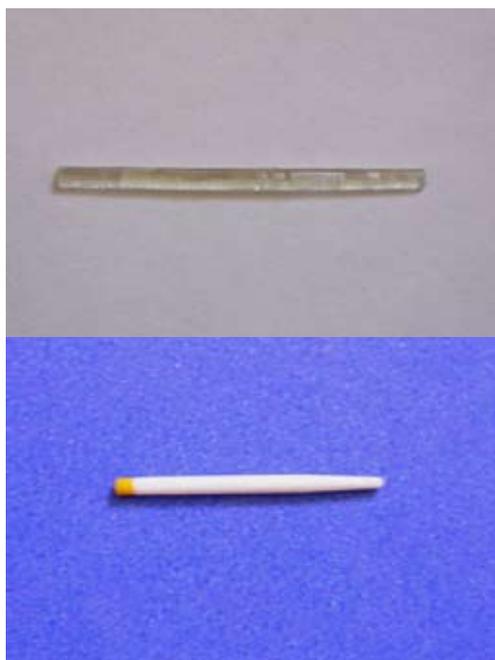


Fig. 1. (a) IPN post (Stick Tech Post); (b) Cross-linked post (Easy Post).



Fig. 2. A specimen attached to the SMAC actuator for testing.

After light curing the adhesive resin, a resin composite (Herculite Kerr Co, Romulus, MI, USA) was used to build up a block on the bonding surface using an incremental technique. Each layer of composite was individually light cured for 20s.

A slow speed, water cooled, diamond impregnated saw (Labcut 1010, Agar Scientific, Stansted, UK) was used to section each block perpendicular to the interface; from each block a 1.5 mm thick slab was obtained, in the Easy-post group the blocks were obtained from the cylindrical portion of the posts. The following experimental groups were thus formed:

Group 1: Ever Stick post/Optibond Solo

Group 2: Ever Stick post/Stick Resin

Group 3: Easy Post/Optibond Solo

Group 4: Easy Post/Stick Resin

The exact width of the specimens was then checked using a calliper (Mitutoyo CD15, Mitutoyo, Kawasaki, Japan), all the specimens were found to be of the desired thickness. Subsequently, under low-magnification stereo microscopy (SDZ-PL, Kyowa Optical Co. Ltd. Hashimoto, Japan), a fine diamond bur was used to cut two slots in each specimen through the composite to the level of the post, thereby isolating bonded areas to undergo tensile testing (Fig 2). Before testing all specimens were observed with a tandem scanning confocal microscope (Noran Instruments, Middleton, WI, USA) using a 2.5 dry lens, to confirm that no groove had been cut on the post surface, all posts were found to be intact.

A miniature load testing device (SMAC Europe, Horsham, West Sussex, UK) was used to test the specimens (Figure 2). The machine consisted of a linear actuator (LAL90-015-53) with a stroke length of 15mm, a peak force of 100N and a resolution of 1µm.

Using cyanoacrylate glue (Zapit, DVA, Anaheim, CA, USA), each specimen was attached to two platforms on

a miniature straining stage, one part of which was stationary while the other could be drawn away, and subjected to tensile forces at 1mm/min until failure. The failure load data were recorded and stored using a PC-software (SMAC Europe, Horsham, West Sussex, UK).

The tested specimens were then measured by imaging the interfaces with a tandem scanning confocal microscope (Noran Instruments) using a 2.5 dry lens. The captured images were relayed to a PC using a CCD camera (Cohu, San Diego, USA). Lucida Analyse image analysis software (Kinetic Imaging, Nottingham, UK) was used to measure precisely the bonded area on each flat interface. Bonded surface areas were calculated by multiplication of these values with the caliper-checked specimen widths. The peak failure load was divided by the surface area to obtain the microtensile bond strength.

The tested specimens were further examined by stereo microscope to assess the failure modes and pictures were taken with a digital camera (Coolpix 990, Nikon, Tokyo, Japan). Additionally, four post-composite blocks from each group were obtained; two of them were observed before and two after testing by scanning electron microscopy (SEM). Each specimen was mounted on an aluminium stub and gold sputter-coated before observation. Low magnification images provided an overview of the specimens. The bonding interface, the penetration of bond into the posts and the structure of the posts were observed at higher magnification.

Failure modes, as determined by stereomicroscopy, were classified as:

1. Fracture at the interface between the post and the bonded resin composite (adhesive failure)
2. Fracture line within the post (cohesive failure)
3. Mixed failure (cohesive-adhesive)

The data was analysed using Stata Release 9.0 (Stata Co, College Station, Texas, USA). For all statistical tests

significance was pre-determined at $\alpha = 0.05$. The bond strength was analysed using the non-parametric Kaplan-Meier survival analysis and the effect of post type and bonding agent on bond strength was evaluated using the Cox proportional hazards model. The distribution of failure modes as a function of post type/bonding agent was evaluated using the Pearson's χ^2 test with exact non-parametric inference.

Results

None of the specimens failed prior to testing. Univariate summary statistics for the four groups tested are presented in Table 2.

The mean values of tensile strength were lower for the groups of specimens bonded with Optibond Solo and higher for the groups bonded with Stick Resin. The highest mean value was recorded for the group Ever Stick post/Stick Resin (8.95 MPa), and the lowest for group Easy Post/Optibond Solo (5.01 MPa). The results of the tensile strength test analysed using the non-parametric Kaplan-Meier survival analysis are shown in Figure 3. The vertical axis of the plot shows the probability of success, where 0.00 is failure and 1.00 represents no failure. Subsequent analysis showed that the data conformed to the assumptions of the Cox proportional hazards model (8). This revealed that only the type of bonding agent had a significant effect on tensile strength ($p = 0.017$), whereas the type of post used had no statistical significant effect ($p = 0.263$).

All the specimens failed during the microtensile test. No mixed failure was observed. Post fracture was the only failure mode observed for groups 1 and 2 (Fig. 4a). On the other hand, only one out of ten specimens from groups 3 and 4 fractured within the post whereas the remaining specimens demonstrated a failure between the post and the resin composite (Fig. 4b). The failure mode results are

Table 2. Univariate summary statistics of each post /bond combination.

Groups	Mean tensile strength (MPa)	Probability
Ever Stick post/Optibond Solo [10/0]	6.75±1.82 ^(a)	0.928
Ever Stick post/Stick Resin [10/0]	8.95±5.16 ^(b)	0.190
EasyPost/ Optibond Solo [10/0]	5.01±2.66 ^(a)	0.432
EasyPost/StickResin [10/0]	7.89±4.09 ^(b)	0.805

Different upper lower-case letter indicate a significant difference between the different groups. The type of bonding agent had a significant effect on tensile strength ($p = 0.017$). No differences were observed between the type of post used ($p = 0.263$).

The numbers in parenthesis [10/0] respectively indicate the number of the specimens tested for the microtensile and the specimen failed prematurely.

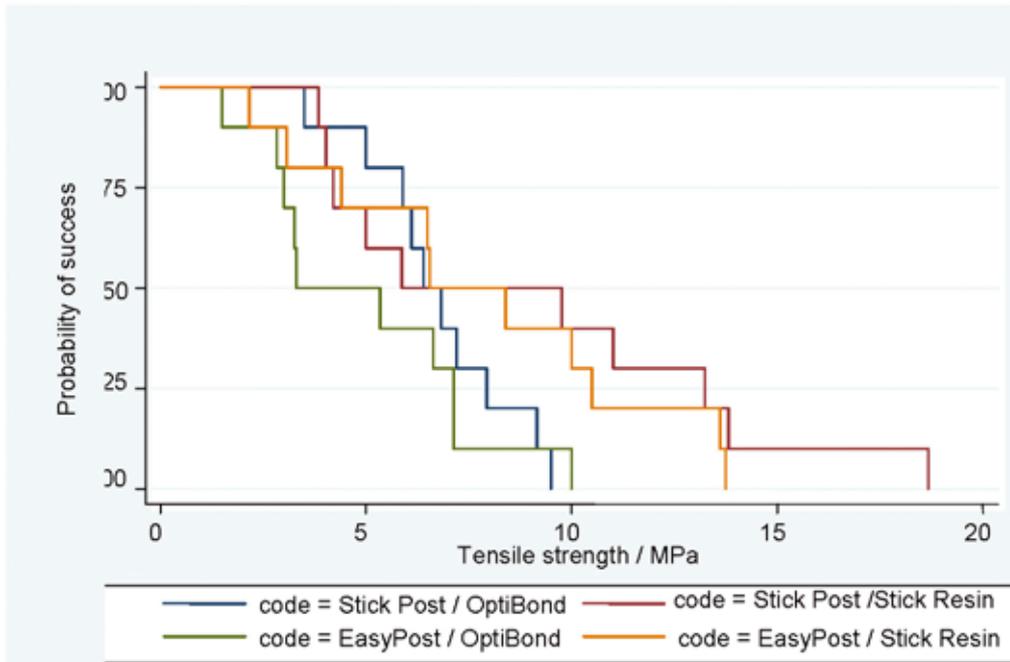


Fig. 3. Kaplan-Meier survival estimates by Post/Bond Combination The vertical axis of the plot shows the probability of success, where 0.00 is failure and 1.00 no failure.

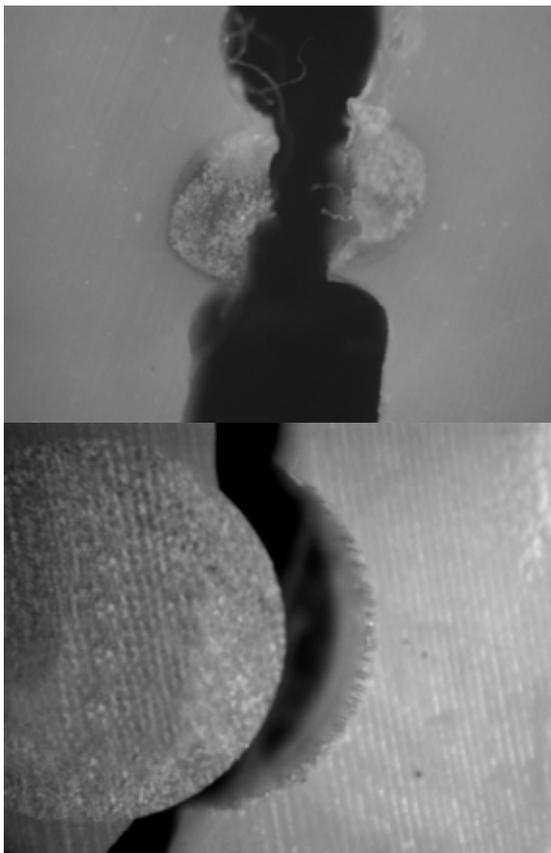


Fig. 4. Failure modes observed (a) IPN post fracture; (b) cross-linked post composite debonding.

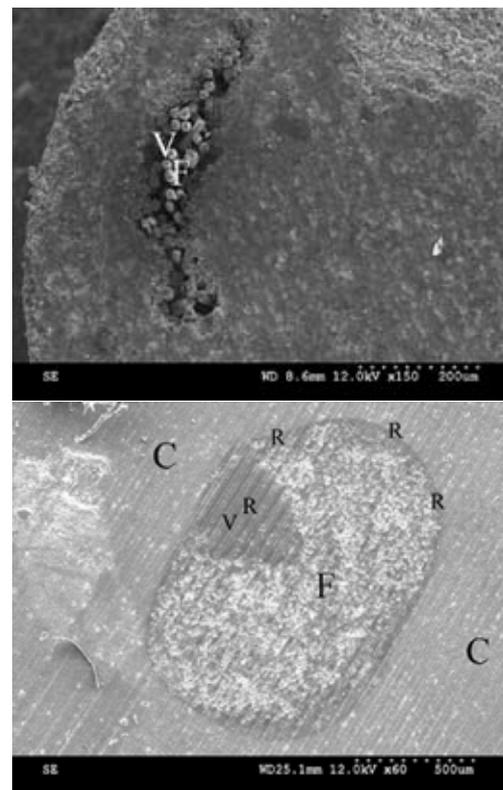


Fig. 5. SEM images (a). Void within cross-linked post (V = void, F = fibres) (b) void within IPN posts filled with resin (V = void, C = composite, F = fibres, R = bonding resin).

Table 3. Failure modes.

Groups	% of fracture	% of debonded
Ever Stick post/Optibond Solo [Group 1] ^(a)	100%	-
Ever Stick post/Stick Resin [Group 2] ^(a)	100%	-
EasyPost/Optibond Solo [Group 3] ^(b)	10%	90%
EasyPost/StickResin [Group 4] ^(b)	10%	90%

Different upper lower-case letter indicate a significant difference between the failure modes of the different groups. The IPN post groups (1 and 2) showed failure within the post. The cross-linked posts groups (3 and 4) showed adhesive failures.

reported in table 3. The exact inferential non parametric inference test ($\chi^2 = 0.0001$) confirmed that there was a significant difference between the failure modes of the different groups. The IPN post groups (groups 1 and 2) showed failure within the post. In contrast the cross-linked posts (groups 3 and 4) demonstrated a high number of adhesive failures.

Voids spaces were observed by SEM in some of the Easy post specimens (Fig. 5a). Penetration of bonding resin within IPN posts was also observed (Fig. 5b).

Discussion

The use of fibre posts in the restoration of root filled teeth has seen increased popularity. The modulus of elasticity of fibre posts is similar to that of dentin, thereby reducing the stress concentrations at the post-dentin interface (9). Clinical studies have demonstrated that the use of fibre posts is a viable alternative to cast posts or metal prefabricated posts (10). The modes of failure when fibre posts are used have been shown to be more favourable in terms of subsequent restorability (11). However, for a successful restoration a strong bond between resin and post and between resin and dentin is needed. Poor bonding at these interfaces will lead to debonding or fracture of the post or core. The observation of failure mode can indicate how the system is working and point out the weakest link.

The rationale behind testing the tensile bond strength is that the higher the actual bonding capacity of an adhesive, the better it will withstand forces applied during mastication and the longer the restoration will survive (12). The concentration of these forces is more dangerous between materials with different mechanical properties. Differences between the relative rigidity of the post and the composite core can generate stress concentration in this area; thus the study of this interface is particularly important for the restoration of endodontically treated teeth.

To evaluate the tensile strength of the two different inter-

faces; post and resin cement, and resin cement and dentin, different methods have been used. The concept that bond strength is inversely related to the bonded surface area has enabled the evaluation of transverse forces across small interfaces such as between the post and resin (13). Smaller surface areas showed higher tensile bond strengths, whereas larger surface areas were associated with lower tensile bond strengths (13).

The thickness of the specimens tested in this study was determined at 1.5 mm. During the pilot study specimens of 1mm and 2 mm thickness were tested. The 1 mm thick specimens proved to be too fragile and prone to fracture. Failure to separate during the microtensile test was noted with the 2 mm thick sections. A surface area of 1.6-1.8 mm² has been recommended for tensile bond tests since this size provided minimum scatter in the results and mainly adhesive failures in the tested specimens (12). A high incidence of premature failures has been reported during hourglass trimming of dentin specimens of 0.5mm thickness (14), whereas specimens of 1.5 mm² cross-sectional surface have shown a minimal percentage of premature failures (15). With regard to the effect of the cross-sectional shape on the tensile strengths and patterns of stress distribution employed in the microtensile test specimens, a comparison between cylindrical and rectangular shapes produced no significant differences (15).

The bonding between the FRC substrate and a resin composite can be achieved by chemical reaction of free carbon-carbon double bonds on the surface of the polymer matrix of the FRC. However, a small number of unreacted carbon-carbon double bonds can be found on the already polymerized resin matrix (16,17). Thus, the adhesion between the already polymerized resin matrix of the conventional fibre post and the methacrylate-based composite substrates of the adhesive is difficult to achieve by means of free radical polymerization bonding (covalent bonding) (17), and it relies mostly on the micromechanical adhesion of the resin composite to the irregularities present on the surface of the posts. The adhesion between two types of translucent prefabricated fibre reinforced posts (FRC Postec, Ivoclar-Vivadent, FRC Light-Post, RTD) and two types of flowable composites was investigated, with or without the application of silane (7). It was concluded that post-silanization had a significant effect on adhesion with any combination of post and core material tested. In two more recent tensile strength studys (18,19) the silanization of fibre posts did not produce any increase in the bond strength. For this reason in the present study no surface treatment of the cross-linked posts was performed.

A different way of achieving bonding between a FRC and resin is the so-called interpenetrating polymer network bonding based on the interdiffusion of monomers into the substrate. The substrate must be a linear polymer and the monomers of the resin must have a capability of dissolving the linear phases of the substrate. Higher shear

bond strengths were noted for FRC specimens treated with resin containing Bis-GMA, TEGDMA or HEMA, when compared with untreated specimens, indicating that these monomers are capable of dissolving the IPN matrix (17). It has been also shown that not all of the monomers used in dental bonding agents can dissolve the PMMA-based IPN structures. The monomer octahydro-4, 7-methano-1H-indenediyl, bis (methylene) diacrylate, used in Sinfony Activator Liquid (3M-ESPE, Seefeld, Germany), has been shown to be unable to dissolve completely the PMMA matrix (20).

Both bonding agents used in this study (Optibond Solo and Stick Resin) contain suitable substrates that have been shown to have a good capability of dissolving the linear or semi-linear PMMA phases of the FRC substrate (5). Quantitative comparison though, with regards to tensile strength between cross-linked and linear polymers, had not been yet conducted. In the present study, specimens treated with Stick Resin composed by Bis-GMA and TEGDMA, demonstrated higher tensile strengths when compared with specimens treated with Optibond Solo (Bis-GMA and HEMA); the first null hypothesis was therefore rejected.

The observation of the tested specimens revealed two failure modes. The majority of the specimens (90%) in groups 3 and 4, showed adhesive failure at the interface between the Easy Post and the resin composite. Only 1 in 10 specimens showed a fracture of the post; the second null hypothesis was therefore also rejected.

The observations regarding failure mode were similar to those of a study evaluating microtensile bond strength of a dual-cure resin core material to glass and quartz cross linked fibre posts, using different surface treatments (21). Adhesive failures were also seen, primarily with Bis-GMA containing monomers, in a study measuring the tensile bond strength of adhesive cements and different posts (22).

All specimens in groups 1 and 2 failed within the IPN post. No adhesive failure mode was observed in these groups. In a study comparing the bonding of luting cement to fibre reinforced root canal posts, with either cross-linked or a semi-IPN polymer matrix, the latter performed better than the former (5). In this study the bond strength between the luting agent and post was studied with the use of a push-out test; it has been suggested that in this type of study, a non-uniform stress may be developed at the interface (23). In addition, as a result of the nature of the test, the retention may be created to some extent through the frictional fit between the two surfaces (21). A combination of the different experimental design and the different post and composite materials tested may, therefore, explain why the results of our work are not in agreement with those of the push-out test study (5). A strong bond between posts and composite cores is certainly desirable, on the other hand if, as happened in our study, the force that produces the

failure is the same for the two post types, the debonding of the post from the core may be a clinically more favourable failure mode than fracture of the post: a composite core fracture may be repaired by adding composite whereas a post fracture can only be repaired by completely removing the fractured post, a procedure that is certainly more challenging, and associated with some risk of root perforation and weakening of the root structure.

It was also observed that even though both selected posts were of the same diameter (1.2 mm), the Ever Stick post was not always symmetrical and round in cross-section. In most of the cases the post had an oval shape (Fig. 5b). It is for this reason that individual measurements of the bonded surface of each post were made by confocal microscopy. In conclusion, Stick Resin was more effective than Optibond Solo in bonding composite cores to fibre posts. Post fracture was the failure mode of IPN posts, debonding of the composite core was the most frequent failure mode of cross-linked posts. The different failure modes observed may be reflected clinically in different failure modes of endodontically treated teeth restored with the post types tested in this study.

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