Penetration of bonding resins into fibre-reinforced composite posts: a confocal microscopic study

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Abstract

Mannocci F, Sherriff M, Watson TF, Vallittu PK. Penetration of bonding resins into fibre-reinforced composite posts: a confocal microscopic study. *International Endodontic Journal*, **38**, 46–51, 2005.

Aim To compare the penetration of two different bonding resins applied for different contact times, on glass reinforced–reinforced composite root canal posts showing an interpenetrating polymer network (IPN) or cross-linked polymer matrix. The research hypothesis was that the bonding resins were equally able to penetrate into the IPN post whereas less penetration was evident into the cross-linked post. In addition, the prolonged contact time of the bonding resin on the surface of post was assumed to increase the resin penetration.

Methodology A total of 36 posts, comprising 18 IPN posts (everStick Post) and 18 cross-linked posts (C Post Millennium) were divided into 12 groups of three posts. All posts had a diameter of 1.2 mm. Both bonding resins (Scotchbond Multi Purpose Plus and Stick Resin) were labelled with Rhodamine B for determination of the resin penetration into the post. After contact times of 1, 30 and 300 s the bonding resins were light cured for 60 s. Penetration of bonding resins into three sections of each post was measured by confocal scanning light microscope. The influences of post and type of adhesive resin on resin diffusion into the polymer matrix of the post were assessed by nonparametric methods.

Results For C Post Millennium and for 1 s contact time, no penetration of bonding resins into the polymer matrix of posts occurred in any section of any post. For the everStick Post, penetration was always noted after 300 s contact (mean 22.8 μm) and nearly always after 30 s contact (mean 3.5 μm), with very little difference between the two adhesives. The degree of penetration into everStick Posts was invariably greater after 300 s contact than after 30 s ($P < 0.001$). Both sets of results were highly significantly greater than the zero penetration recorded using C Post Millennium or 1 s contact time ($P < 0.001$).

Conclusions The ability of bonding resins to penetrate into everStick Post with IPN polymer matrix may give the opportunity to establish a good link between fibre-reinforced composite posts, luting cements and composite cores.

Keywords: bonding resins, confocal microscopy, fibre posts, fluorescent probes, polymers, Rhodamine B.

Received 10 May 2004; accepted 24 September 2004

Introduction

Fibre-reinforced composites (FRC) are used extensively for the construction of fibre posts. These posts are frequently used in the restoration of endodontically treated teeth (Fredriksson *et al.* 1998, Ferrari *et al.* 2000, Mannocci *et al.* 2002, Qualtrough & Mannocci 2003). Recently, FRC posts made of a material having silanted glass fibres impregnated with an interpenetrating polymer network (IPN) resin matrix (everStick Post; Stick Tech Ltd, Turku, Finland) have been introduced to the market. In IPN, or more precisely, semi-IPN polymer, linear polymer phases and the

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cross-linked polymer phases are not bonded chemically together as a single network as they are in the case of a typical co-polymer (Sperling *et al.* 1994). It has been claimed by the manufacturer that bonding of the FRC post with the IPN resin matrix to composite resin and to adhesives/composite cements was improved by an interdiffusion bonding mechanism. In this type of posts, during the contact time of the bonding resin to the surface of IPN resin matrix, the monomers of the bonding resin diffuse into the linear phases of the IPN polymer matrix and after polymerization become interlocked. EverStick FRC contains polymethylmethacrylate (PMMA) as a linear phase and poly bis-GMA as the cross-linked phase of the polymer matrix. On the surface of the everStick FRC there is an enriched layer of PMMA. Suitable adhesive resins for diffusing into the PMMA phase are those having solubility parameters close to that of PMMA. Adhesive resins of that kind have been proposed to be those containing hydroxyethylmethacrylate (HEMA) monomers or dimethacrylate monomers (Le Bell *et al.*, in press).

The aim of the present investigation was to compare the penetration of two different bonding resins, applied for different contact times, into an IPN polymer matrix FRC post and into a conventional cross-linked FRC post. The hypothesis was that the bonding resins were equally capable of penetrating into the IPN post whereas less penetration would be evident into the cross-linked post. In addition, prolonged contact time of the bonding resin on the surface of post was assumed to increase the resin penetration.

Materials and methods

A total of 36 posts, 1.2 mm in diameter, were included in the study: 18 IPN resin matrix containing everStick Posts (Fig. 1a) and 18 cross-liked epoxy polymer matrix containing C Post Millennium (Sogeva, Varese, Italy) (Fig. 1b). The posts were divided into 12 groups of three posts. The components of the adhesive systems and of the FRC posts used are summarized in Tables 1 and 2. A schematic drawing of an horizontal section of a fibre post is shown in Fig. 2. Three posts were included in each group. The IPN posts were light cured for 60 s using an Optilux 500 light curing unit (Demetron-Kerr, Romulus, MI, USA), the intensity of the light was 400 mW cm^{-2}; following the manufacturer's recommendation, no light curing was performed on cross-linked posts. Both post types were then immersed in bonding resins for the contact times of 1, 30 or 300 s. Light exposure was avoided during the contact time of the resin. Both bonding resins (Scotchbond Multi Purpose Plus, 3 M, St Paul, MN, USA and Stick Resin, Stick Tech Ltd) were labelled with Rhodamine B, to allow the confocal microscopic measurement of the penetration of the resin into the polymer matrix of the post (Mannocci *et al.* 2001). At the end of the predetermined immersion time, the bonding resins were cured for 60 s using an Optilux 500 light curing unit (Demetron-Kerr).

The FRC posts were embedded in autopolymerizing acrylic resin (Palavit 55 VS, colour A35; Haereus Kulzer, Werheim, Germany) and then sectioned in three positions, perpendicularly to the long axis of the post at a distance of 3, 5 and 7 mm from the post tip using a low speed diamond saw. A total of nine sections were included in each experimental group. The tip of the cross-linked C Millennium Posts was conical and the remaining part of the post cylindrical. The posts were sectioned in the cylindrical part; in order to obtain sections of the same size and shape of those obtained from cylindrical everStick Posts (Fig. 3).

Measurement of the penetration of bonding resins into sections of the posts was performed using a confocal scanning type microscope (TSM; Noran Instruments, Madison, WI, USA), focusing below the surfaces damaged by sectioning. Samples were examined using ×20 and ×60 oil immersion objectives giving a final magnification of 200–600 times. For each post the deepest penetration depth (d) of the bonding resins along the diameter of the post was measured using the ×60 oil immersion objectives.

Statistical analysis

The experiment was designed as a factorial study. Table 3 lists the 12 combination of post, resin and contact time used. Analysis of the results was planned by two-way ANOVA, modelling the penetration depth of the bonding resin into the polymer matrix of the post by the type of post and the type of bonding resin. Contrary to our expectation, for eight of the 12 cells all results were zero. The resulting gross and irremediable heterogeneity necessitated use of nonparametric methods for the main analyses.

There was no evidence whatever for the difference between the two adhesives – differences between groups H and K and between I and L were both very small and in opposite directions. Consequently the main analysis related to the six merged pairs of groups, AD, GJ, BE, HK, CF and IL, of which all apart from HK

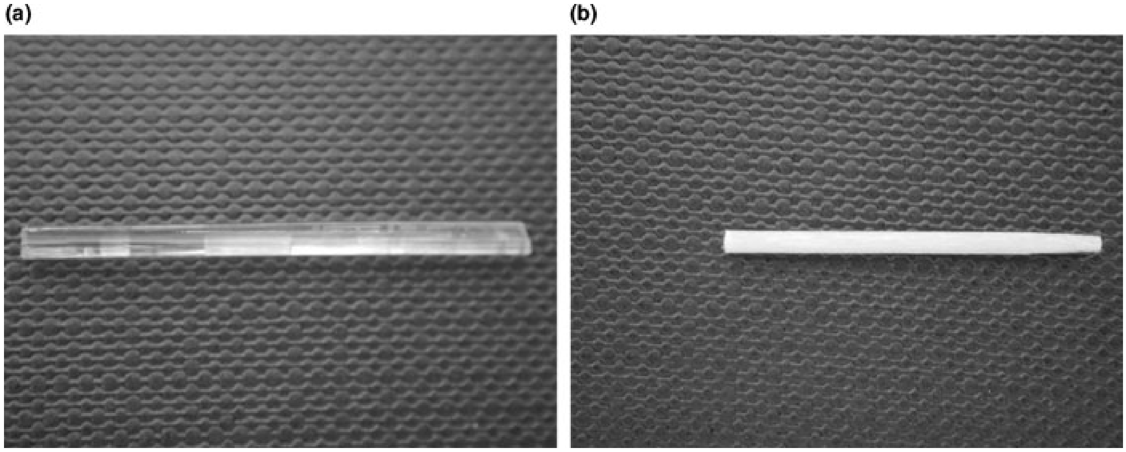

Figure 1 (a) EverStick Post with interpenetrating polymer network polymer matrix. (b) C Millennium post with cross-linked polymer matrix.

Table 1 Components of the bonding resins used

Scotchbond Multi-Purpose Plus	bis-GMA, HEMA
Stick Resin	bis-GMA, TEGDMA

bis-GMA: 2,2-bis(4-2-hydroxy-3-methacryloylxypropoxy)phenyl]-propane; HEMA: hydroxyethylmethacrylate; TEGDMA: triethylene glycoldimethacrylate.

Table 2 Components of the fibre post-systems used

Stick posts	E-glass fibres, PMMA, bis-GMA
C-post Millennium white	Glass fibres, epoxy polymer

PMMA: polymethylmethacrylate (MW 220.000); bis-GMA: 2,2-bis(4-2-hydroxy-3-methacryloylxypropoxy)phenyl]-propane.

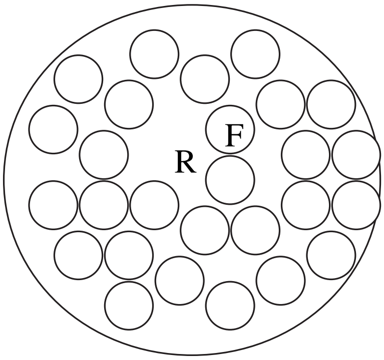

Figure 2 Schematic drawing of an horizontal section of a fibre post. F, fibres; R, resin matrix.

and IL showed zero penetration in all 18 specimens. For each of groups H, J, K and L, Table 3 shows results for three posts each at three sites. Two-way analysis of variance for 30- and 300-s data separately did not show clear evidence of differences by post or segment of post, so the nine readings in each cell can be regarded as independent.

Nonparametric tests were then performed for two types of comparisons:

1. Differences between three contact times, for the everStick Post: compare IL versus HK versus GJ using Kruskal–Wallis one-way nonparametric ANOVA, followed by Mann–Whitney tests comparing each pair of groups.

2. Differences between two post types, at 30 and 300 s contact time: Mann–Whitney tests comparing groups HK versus BE and IL versus CF.

Mann–Whitney tests comparing groups are presented both as *P*-values and also effect sizes expressed as U/mn with 95% confidence intervals calculated using method 5 of Newcombe (2004a,b,c).

Results

Table 3 summarizes the results for penetration depth by type of post, type of resin and contact time.

Table 4 shows the comparisons of outcomes between groups as described above, with confidence intervals for effect sizes.

For C Post Millennium, and for 1 s contact time, no penetration of bonding resins into the polymer matrix of posts occurred in any section of any post. For the everStick Post, penetration was always noted after 300 s contact (groups I and L, mean 22.8 μm) and nearly always after 30 s contact (groups H and K, mean 3.5 μm) with very little difference between the

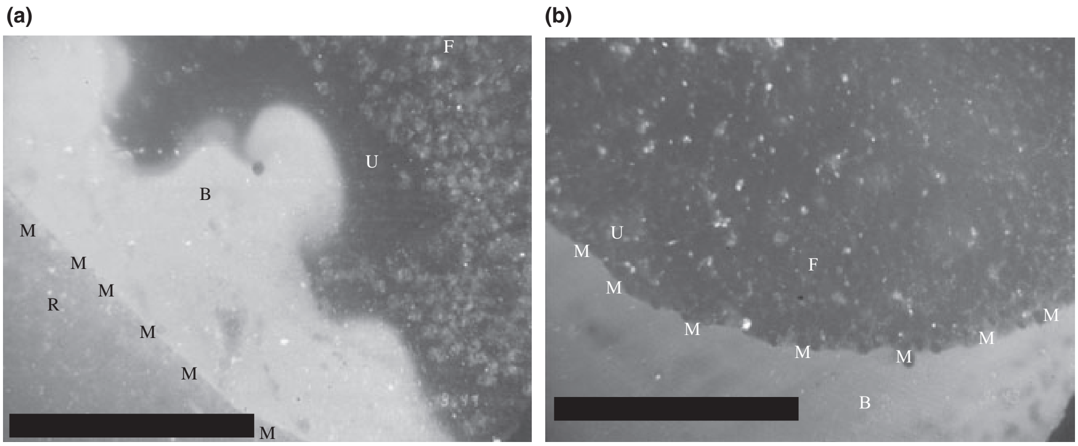

Figure 3 (a) Confocal micrograph of the cross-section of everStick Post showing penetration depth of the bonding resin into the polymer matrix of the post. (b) Confocal micrograph of the cross-section of C Millennium post showing penetration depth of the bonding resin into the polymer matrix of the post. R, self-curing resin; M, margin of the post; B, bonding resin; U, resin matrix of post; F, glass fibres. Bar = 20 μm.

Table 3 Univariate summary statistics

Group code	Post	Adhesive	t (s)	d_m (μm)	SD	d (μm)
A	C Post M	SMPP	1	0	0	
B	C Post M	SMPP	30	0	0	
C	C Post M	SMPP	300	0	0	
D	C Post M	Stick R	1	0	0	
E	C Post M	Stick R	30	0	0	
F	C Post M	Stick R	300	0	0	
G	eStick Post	SMPP	1	0	0	
H	eStick Post	SMPP	30	3.0	2.0	5, 1, 2, 4, 2, 1, 1, 5, 6
I	eStick Post	SMPP	300	23.2	6.6	15, 24, 25, 35, 31, 21, 15, 20, 23
J	eStick Post	Stick R	1	0	0	
K	eStick Post	Stick R	30	4.0	2.6	6, 0, 5, 7, 1, 5, 1, 5, 6
L	eStick Post	Stick R	300	22.4	6.2	19, 25, 31, 21, 22, 10, 20, 25, 29

C PostM, C Post Millennium; eStick Post, everStickPost; SMPP, Scotchbond Multi Purpose Plus Adhesive; StickR, Stick Resin; t, contact time in seconds; d_m, mean diffusion distance in μm; SD, standard deviation of diffusion distance; d, diffusion distance in μm.

two adhesives. The degree of penetration into everStick Posts was invariably greater after 300 s contact than after 30 s ($P < 0.01$). Both sets of results were highly significantly greater than zero penetration recorded using C Post Millennium or 1 s contact time ($P < 0.001$).

Discussion

In the present investigation only three posts were included in each experimental group. The posts were sectioned in three parts, so that nine measurements of the penetration of the dentine bonding systems were performed in each group, the number of specimens, although limited, was sufficient to show statistically significant differences between the experimental groups.

The confocal microscope is able to produce thin subsurface optical sections of translucent specimens, such as the glass fibre posts observed in this study, with improved vertical and horizontal resolution. This is due to elimination of scattered, reflected and fluorescent light from out of focus planes, according to the confocal principle that the illumination and imaging of one spot in one focal plane at one time are combined (Minsky 1988). This system facilitates imaging below the surface of a bulk semi-transparent specimen, thereby avoiding the need for thin sectioning and fixing.

Fluorescence imaging is probably the most important readout mode in biological confocal microscopy, in this investigation a fluorescent probe traditionally used for biological microscopy, such as Rhodamine B, was used to label the bonding resins, in order to trace their penetration into glass fibre posts.

The adhesion of resinous material to an already polymerized substrate can be obtained by the interdiffusion of monomers of the new resin into the polymer structure of the substrate or by free radical polymerization of

Table 4 Selected comparisons between groups of results

Contrast	Test	*P*-value	U/mn (95% confidence interval)
Differences between contact times, for eStick Post			
300 s versus 30 s versus 1 s	IL versus HKvGJ	Kruskal–Wallis <0.001	
300 s versus 30 s	IL versus HK	Mann–Whitney <0.001	324/324 = 1.0 (0.89–1)
300 s versus 1 s	IL vigil	Mann–Whitney <0.001	324/324 = 1.0 (0.89–1)
30 s versus 1 s	HK e.G.	Mann–Whitney <0.001	323/324 = 0.997 (0.88–0.9999)
Differences between eStick Post and C Post M, at 30 and 300 s contact time			
30 s	HK versus BE	Mann–Whitney <0.001	323/324 = 1.0 (0.89–1)
300 s	IL versus CF	Mann–Whitney <0.001	323/324 = 0.997 (0.88–0.9999)

the bonding resin to the unconverted double bonds of the remaining functional groups of the substrate. The likelihood for the latter to occur is minor with conventional FRC root canal posts whose polymer matrix is typically based on epoxy. No chemical reaction of methacrylate based luting cements to the well-polymerized epoxy polymer occurs. On the contrary, there are difficulties in the adhesion of methacrylate-based resin to the polymerized methacrylate composite substrates (Kallio *et al.* 2001, Lastumäki *et al.* 2002). The interdiffusion bonding phenomenon may occur if the polymer substrate is totally or partially a linear polymer (not cross-linked), as is the case in IPN polymers (Sperling *et al.* 1994). Another requirement for the interdiffusion bonding to occur is related to the ability of the monomers of the bonding resin to dissolve the linear or IPN polymer. The dissolving ability of solvent is determined by the term 'solubility parameter', which should be close to that of the substrate to be dissolved. For the PMMA the solubility parameter is 9.45 (cal $cm^{-3})^{1/2}$ and for MMA it is 8.8 (cal $cm^{-3})^{1/2}$ (Sperling 1986). To the authors' knowledge, there is no information available on the dissolving parameters of bis-GMA and TEGDMA monomers. The results of the present study proved that both the monomers of the bonding resins, i.e. bis-GMA, TEGDMA and HEMA were able to penetrate into the IPN polymer structure of everStick Post, whereas no penetration was observed into the cross-linked C Post Millennium posts. This suggests that the solubility parameters of those monomers are likely to be close to that of PMMA.

It is important to understand that not all of the monomers used in dental bonding resins are capable of dissolving PMMA-based IPN structures. For instance, a monomer octahydro-4,7-methano-1H-indenediyl) bis (methylene) diacrylate used in Sinfony Activator Liquid (3M-ESPE, Seefeld, Germany) is not able to dissolve PMMA and its IPN structures (Lastumäki *et al.* 2003). It was suggested that although the monomer contains two acrylate groups, the existence of an indenediyl group may have increased the rigidity of the monomer to such extend that no dissolving of PMMA was possible to obtain. The Sinfony Activator Liquid is intended to be used in dental laboratories. If the monomer resin or polymer structure of the substrate does not allow formation of the interdiffusion bonding, other means of improving adhesion could be used. These can include using composite adhesive primers or roughening the bonding surface (Tezvergil *et al.* 2003).

The achievement of a good attachment between FRC post and composite materials used for luting of posts and for the crown build-up of the core is of great importance in the restoration of endodontically treated teeth. The ability of bonding resin to penetrate into the structure of IPN posts may give the opportunity to establish an adhesion between FRC post, luting cements and composite cores superior to that obtained with the other post-types available. Further laboratory studies are needed to test this hypothesis. To the authors' knowledge the evidence of a penetration of a luting material into the structure of an endodontic post has not been demonstrated previously. Prospective clinical studies are also needed to evaluate if the improved interdiffusion of bonding resins into IPN polymer structure will result in clinical performances better than those obtained with the cross-linked carbon, quartz and glass FRC posts that are now extensively used in daily practice.

Conclusions

It can be concluded that the two bonding resins tested effectively penetrated into the IPN structure of FRC post, whereas no penetration was observed in posts with cross-linked polymer matrix. In addition, the penetration depth was increased by the prolonged contact time of the bonding resin. The research hypotheses were therefore confirmed.

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