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UV irradiation improves the bond strength of resin cement to fiber posts

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The purpose is to evaluate the effect of UV irradiation on the bond strength between epoxy-based glass fiber posts and resin cement. Twelve epoxy-based glass fiber posts were randomly divided into three groups. Group 1 (Cont.): No surface treatment. Group 2 (Low-UV): UV irradiation was conducted from a distance of 10 cm for 10 min. Group 3 (High-UV): UV irradiation was conducted from a distance of 1 cm for 3 min. A resin cement (CLEARFIL SA LUTING) was used for the post cementation to form resin slabs which contained fiber posts in the center. Microtensile bond strengths were tested and the mean bond strengths (MPa) were 18.81 for Cont. group, 23.65 for Low-UV group, 34.75 for High-UV group. UV irradiation had a significant effect on the bond strength ($p<0.05$). UV irradiation demonstrates its capability to improve the bond strength between epoxy-based glass fiber posts and resin cement.

Keywords: UV irradiation, Glass fiber post, Resin cement, Microtensile bond strength

INTRODUCTION

Prefabricated fiber-reinforced composite (FRC) posts are widely accepted today as a viable alternative to cast posts for the restoration of endodontically treated teeth[1)]. Luted inside root canals, the fiber posts provided retention to a core material, which is directly built up on it. Having an elastic modulus similar to dentin[2)], fiber posts provided a better distribution of loads to the teeth than metal posts and induced low rate of root fracture[3,4)]. However, prospective and retrospective clinical studies have reported that in restorations with fiber posts luted in the root canal, failures were commonly found in debonding of the posts[1,3,5,6)]. Therefore, enhancement of bond strength at the post/cement and cement/root dentin interface is needed in clinical practice[7)].

Most of the matrix component of FRC posts is epoxy resin which has a high degree of conversion and highly crosslinked structures. As a result, no functional groups of the fiber posts would react with the methacrylate group, which is the major component of dental composite resin. This results in the absence of chemical bonding between these two substrates[8)].

Many studies have tested ways of improving the interfacial strength between fiber posts and resin based materials. These methods can be mainly divided into three categories: (1)treatments that result in chemical bonding between composite and posts; (2)treatments that intend to roughen the surface or (3)the combination of above-mentioned two methods[8)]. Treating the post surface with a silane coupling agent has been widely practiced for enhancing fiber post/composite interfacial adhesion[7,9-12)]. Silane agent is able to chemically bridge –OH covered inorganic substrates and composite materials, at the fiber post/composite interface. In this condition, chemical bond is possible only between the composite and the exposed fibers of the post[9)]. However, challenges on the effectiveness of this approach still remain[12-14)]. In this sense, chemical adhesion alone still may not achieve a satisfactory fiber post/composite interfacial bond strength[8)]. Hydrofluoric acid has been applied to etching fiber posts surface, and this procedure statistically increased the interfacial bonding strength between the posts and the composite[11,15,16)]. However, this technique could lead to substantial damage to the fibers and resin matrix, affecting the integrity of the posts[10,15,17)]. Furthermore, several studies investigated that blasting with alumina particles on the fiber posts surface resulted in an increased roughness of the posts surface that induce interlocks at the post/composite interface, and enhanced the micromechanical retention[11,18-23)]. In addition, application of the Co-Jet system could weld a silicate layer onto the fiber posts surface which was often followed by silanization. This approach could significantly improve the interfacial adhesion through achieving a chemical combination and micromechanical retention[16,19,24)]. Besides, immersing the fiber posts into hydrogen peroxide could remove the epoxy resin of the surface layer, so that a greater surface area of exposed fibers is available for silanization and the space between these exposed fibers could provide additional micromechanical retention[25-28)]. Despite the above trials, there still has not been an absolutely ideal solution to achieve a satisfactory adhesion between fiber posts and composite materials.

In the field of industry, UV irradiation has been used to improve the adhesive property of coatings as well as wettability and printability of polymers, due to its capability to change the morphology and chemical properties of the polymer surface[29)]. Loyaga-Rendon *et*

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al. recently reported that a short period of UV irradiation on composite resin teeth could improve the bonding efficacy of composite resin artificial teeth to autopolymerizing resin[30]. This phenomenon could be explained by the fact that the energetic ultraviolet photons can break molecular bonds referred to as photolytic chain scission and produce free radicals which are reactive to other substance.

The purpose of this study was to evaluate the effectiveness of UV irradiation on epoxy-based glass fiber posts in promoting the bond strength between the posts and resin cement. The null hypothesis was that UV irradiation does not affect the bond strength between epoxy-based glass fiber posts and resin cement.

MATERIALS AND METHODS

Post surface conditioning

Twelve glass fiber-reinforced composite posts (POPO, Shidelong, Beijing, China) with a diameter of 1.50 mm and length of 19.0 mm were used for this study. The POPO posts are made of unidirectional pre-tensed glass fibers (60 wt%) that are bound in an epoxy resin matrix (40 wt%). The fiber posts were randomly divided into 3 groups to be processed with different surface treatments (n=4). Before the surface treatments, an additional rinsing procedure was performed as follows. All the posts were ultrasonically cleaned for 10 min in distilled water, then immersed in 95% ethanol for 20 s, then dried with air stream.

Group 1 (Cont.): No surface treatment.

Group 2 (Low-UV): UV irradiation was conducted in an ultraviolet sterilizing oven (GD63-389, Midwest, Beijing, China) from a distance of 10 cm beneath the UV lamp at room temperature. The UV lamp emitted UV light with mostly a wavelength of 253.7 nm at a power of 15 W. The glass fiber posts were exposed to the UV irradiation for 10 min, and then rotated for 90° for another 10 min irradiation. Such procedure was repeated for four times in order to have the surface irradiated fully and evenly.

Group 3 (High-UV): UV irradiation was conducted in the same ultraviolet sterilizing oven from a distance of 1 cm beneath the UV lamp. The glass fiber posts were exposed to the UV irradiation for 3 min, and then rotate for 90° for another 3 min irradiation. The procedures were the same as group 2 (Low-UV).

Luting procedure

Each post was cemented in a translucent polyethylene film mould immediately after UV irradiation. The size of the mould is 16 mm in length, 10 mm in width and 1.5 mm in thickness. First, prepare the resin cement (CLEARFIL SA LUTING, Kuraray, Okayama, Japan) according to the instructions of the manufacturer and insert it into the mould with a syringe (STERILE EO 2, Suyun, Jiangsu, China). Then, the post was inserted into the center of the mould which contained the resin cement. Finally, the cement was evenly cured for 60 s with a LED light-curing unit (Free Light 2, 3M ESPE, Seefeld, Germany) with an output of 1200 mW/cm^2. After the polymerization process of the cement material was completed, the polyethylene film mould was removed gently, leaving behind a resin slab which contained the post in the center (Fig. 1a). The slabs were then stored in distilled water at 37°C for 24 h.

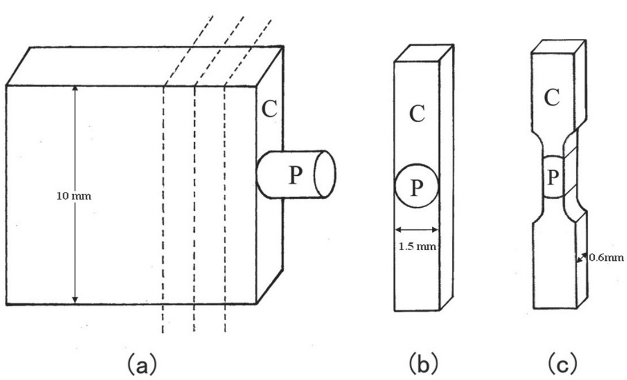

Fig. 1 Schematic illustration of specimens making for microtensile test (C: cement, P: fiber post). (a) The resin slab which was 10 mm in width contained the fiber post in the center. (b) The resin slab was sectioned vertically to the post to obtain resin beams, diameter of the fiber post is 1.5 mm. (c) The resin beam was trimmed into dumbbell-shaped specimens to expose the post surface, thickness of the specimens is about 0.6 mm.

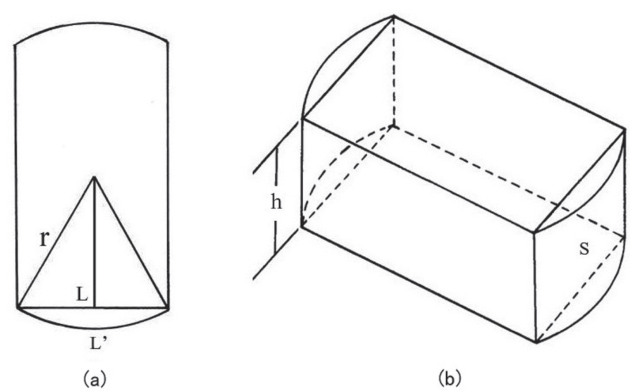

Fig. 2 Schematic illustration of the fiber post in the center of the microtensile test specimen. (a) The cross section of the fiber post in the center of the specimen (r=radius of the fiber post, L=chord length, L'=arc length). (b) The stereogram of fiber post in the center of the specimen. (h=thickness of the specimen, S=square measure of adhesion area). The square measure of adhesion area can be calculated according to the following formulas. L'=2r*arcsin(L/2r), S=L'*h

Microtensile test

Each resin slab was mounted on the holding device of a slow-speed diamond saw (Isomet 1000, Buehler, Lake Bluff, USA) and then sectioned vertically to the post to obtain 7-8 beams of approximately 0.6 mm in thickness (Fig. 1b). The beams were then carefully trimmed with a high-speed dental airturbine handpiece and diamond bur into dumbbell-shaped specimens to expose the post surface (Fig. 1c). Following the trim, the thickness of each specimen was measured accurately with a digital caliper (SH100, SHAHE, Chengdu, China). In the next step, each specimen was secured to the flat grips of a holding device with cyanoacrylate adhesive (Loctite super glue, Henkel, Holthausen, Germany), and subjected to a tensile load at a cross-head speed of 1 mm/min until failure, using a universal testing machine (Model 3367, Instron Corp., Canton, MA, USA). Then the width of the failure interfaces was measured accurately by a stereo dynascope (TM-111, VISION, Surrey, UK). Then interfacial strength was calculated using the following mathematical formulas previously described by Bouillaguet *et al.* (Fig. 2a and 2b)[31]. $L'=2r*\arcsin(L/2r)$, $S=L'*h$, $\mu MBS=F/S$.

Examination of failure modes

The failure modes were evaluated with a stereomicroscope (SMZ10, Nikon, Tokyo, Japan) at 80× magnification and recorded in three categories: (1) adhesive failure (at the post/cement interface); (2) cohesive failure (within the cement or post) or (3) mixed failure (a combination of the two modes of failure in the same interface).

Statistical analysis

Having verified that data distribution was normal (Kolmogorov-Smirnov test, $p>0.05$) and that group variances were homogeneous (Levene's test, $p>0.05$), One-way ANOVA was applied to microtensile bond strength as the surface treatment as the factor. Tukey test was applied to post-hoc comparisons. The distribution of failure modes were analyzed by Chi-square test. Statistical significance was pre-set at $\alpha=0.05$ and calculations were performed by the SPSS 13.0 software (SPSS, Chicago, IL, USA).

RESULTS

The means and standard deviations of the microtensile bond strength are presented in Table 1. Statistical analysis showed significant difference between groups and post-hoc comparisons revealed that UV irradiation had a significant influence on the microtensile bond strength ($p<0.05$). In addition, comparing to Group 2 (Low-UV), the results of Group 3 (High-UV) demonstrated more obviously enhanced microtensile bond strength, which is statistically significant ($p<0.05$).

Stereomicroscope evaluation revealed modification of the specimen failure surfaces in all the three groups. Adhesive failure displayed solely the impression of the fiber post surface over failure area (Fig. 3a). Mixed failure displayed both the impression of the post and residual resin cement (Fig. 3b). There had no cohesive failure been tested. The distribution of failure modes are described in Table 2. As reflected, failure modes of specimens mostly concentrated in the category of mixed

Table 1 Detailed results of the microtensile test

Group	Surface Treatment	N	Mean microtensile strength and standard deviation (MPa)
1 (Cont.)	No treatment	31	18.81 (4.04)^{a}
2 (Low-UV)	UV irradiation, 10 cm, 10 min	30	23.65 (4.86)^{b}
3 (High-UV)	UV irradiation, 1 cm, 3 min	30	34.75 (5.54)^{c}

N: number of microtensile specimens. Different superscript letters indicate statistically significant differences ($p<0.05$).

Table 2 The distribution of failure modes

Group	Adhesive failure	Cohesive failure	Mixed failure
1 (Cont.)	7/31	0/31	24/31
2 (Low-UV)	7/30	0/30	23/30
3 (High-UV)	2/30	0/30	28/30

xx/XX: xx = numbers of specimen tested reporting the indicate failure mode; XX = numbers of total specimen tested. There was no significant difference between three groups (Chi-square test; $p>0.05$).

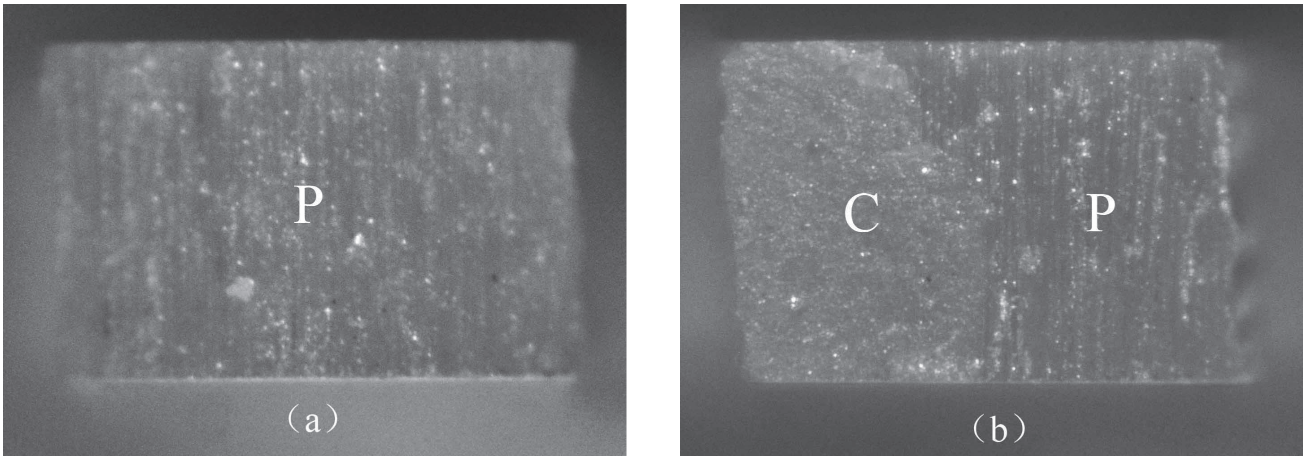

Fig. 3 Representative micrographs of the failure modes evaluation under the stereomicroscope at 80× magnification (C: cement, P: fiber post). (a). Adhesive failure displayed solely the impression of the fiber post surface over failure area. (b) Mixed failure displayed both the impression of the post and residual resin cement.

failure. There was no significant difference between the three groups (p>0.05).

DISCUSSION

The results of this study have confirmed that the microtensile bond strength of resin cement to epoxy-based glass fiber post was significantly enhanced when the posts were exposed to UV irradiation before cementation. The null hypothesis of this study has to be rejected.

During the past decade, a continuous effort has been made to improve the bonding strength between FRC posts and the composite materials, with methods of surface treatment being the frequently-used approach. However, previous methods of FRC posts surface treatment mostly focused on ways to increase surface roughness or enhance chemical bonding of the fiber component. Little is known on how resin phase of these FRC posts could be modified to improve the interfacial adhesion. In this field, the only effort was demonstrated in a recent study. Yavirach *et al*. reported that pre-treat the FRC posts by plasma could significantly improve the tensile-shear bond strength between posts and composite[32]. This enhancement might be explained by polymer chain scission caused by the bombardment of energetic gas molecule particles and polar functional groups can be induced on the surface of the polymer, resulting in higher surface wettability[32].

According to the result of this study, exposing glass fiber posts to UV irradiation in a relatively short period could significantly improve the microtensile bond strength of resin cement to epoxy-based glass fiber posts. UV irradiation led to activation of epoxy resin matrix on the surface of fiber posts, inducing chemical bonding with resin cement without destroying surface structure of fiber posts. It demonstrated stronger effect than using silane, and is much less destructive to the surface of fiber posts than hydrofluoric acid etching, air abrasion and tribochemical silica coating (Co-jet system) method. Furthermore, more appropriate parameter of UV irradiation which can achieve stonger bond strength of fiber posts may be detected in future studies.

In fact, the capability of UV irradiation to modify polymer surface characteristics has been intensively studied in polymer science. UV irradiation reportedly can induce surface radicals that can recombine to form various types of networks and terminal groups[29]. Beak-Su Lee *et al*. had tested the properties of epoxy/glass fiber laminate material after UV irradiation[33]. The result showed that UV irradiation could increase the surface energy, polarity and wettability, resulting in greatly reduction of contact angle of epoxy. Photon energy of short wavelength at the below region of 350 nm is strong enough to rupture most chemical bonds such as C-C and C-H on the epoxy surface to rapidly create many radicals and oxygen groups[33,34]. The above mentioned fact has greatly increased the possibility to form chemical bonding between epoxy and composite materials. The UV lamp typically has a wavelength range between 184.9 nm and 253.7 nm. UV light of 184.9 nm is capable of generating ozone from oxygen in air, and this ozone can be converted into atomic oxygen which is extremely reactive by UV light of 253.7 nm[30]. In this study, the equipment used was an ordinary UV sterilizer in the dental office. The UV light emitted by the lamp was mostly in a wavelength of 253.7 nm. Little ozone was created such that the effect of ozone was negligible in this study. In future studies, UV irradiation with ozone may be able to bring a stronger interfacial adhesion between FRC posts and composite materials.

Several factors in UV irradiation of fiber posts may affect the strength bonding to the resin based materials. Such factors include the distance between the UV lamp and the posts, wavelength of the UV light, power of the UV lamp, duration of irradiation, and absence or presence of ozone. When irradiating beneath the same

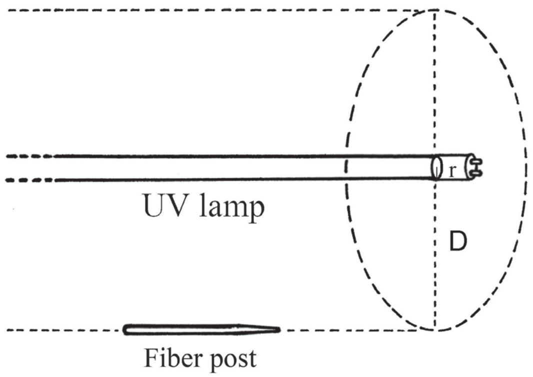

Fig. 4 Schematic illustration of UV irradiation. The UV irradiation energy on the post surface can be calculated according to the following formulas. I=w/[2π*(r+D)*L], E=I*t. (I=irradiation intensity on post surface, E=UV irradiation energy on post surface, w=lamp power, r=radius of the UV lamp, D=distance between lamp and fiber post, L=length of the UV lamp, t=irradiation period).

UV lamp, the distance between the UV lamp and the posts and the duration of irradiation seem to be the major factors that affect the interfacial bond strength in this study. Irradiation distance and duration lead to different extent that the interfacial bonding strength was improved. The reason for such difference might be the different amount of energy of irradiation on the post surface. As indicated in the result, the posts irradiated with a distance of 1 cm for 3 min demonstrated a stronger bonding strength enhancement. Under current condition, the UV lamp can be seen as a liner-shaped illuminate and the energy of irradiation on the post surface could be roughly calculated by the following formulas (Fig. 4). I=w/[2π*(r+D)*L], E=I*t. (I=irradiation intensity on the post surface, E=irradiation energy on the post surface, w=power of the UV lamp, r=radius of the UV lamp, D=distance between UV lamp and fiber post, L=length of the UV lamp, t=irradiation period) With such formula, the irradiation intensity of Group 2 (Low-UV) is approximately 5 mW/cm^2, and the irradiation energy is approximately 3 J/cm^2. The irradiation intensity of Group 3 (High-UV) is approximately 25 mW/cm^2, and the irradiation energy is approximately 4.5 J/cm^2. As a hypothesis, same irradiation energy might lead to the same effect of bonding enhancement. Such hypothesis needs to be supported with further research.

The UV sterilizer used in this study is a frequently-used and inexpensive equipment for disinfection. Compared to surface treatment methods such as sandblasting and etching followed by silanization, especially plasma, UV irradiation is a less expensive, more applicable way to improve the interfacial adhesion, without affecting the integrity of the FRC posts.

This study provides a new method for FRC posts surface treatment, improving the interfacial adhesion by applying UV irradiation. Several factors were excluded from this study, such as the thickness of the resin cement, the C-factor of the root canal and so on. The effect of these factors should be clarified in future clinical researches. In future *in vitro* studies, several questions should be involved, such as whether longer storage between UV irradiation and post cementation will reduce the efficacy of the interfacial adhesion; what is the best intensity of UV irradiation for clinical application; and if the interfacial bond strength will reduce during aging. Future researches should indeed be carried out to answer these questions.

CONCLUSION

In the limitation of the result of this study, UV irradiation of epoxy-based glass fiber posts significantly improved the microtensile bond strength of resin cement to the fiber posts. The results achieved with UV irradiation from the distance of 1 cm for 3 min demonstrated more obviously enhanced bond strength than that from the distance of 10 cm for 10 min.

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