Effect of ultraviolet light irradiation period on bond strengths between fiber-reinforced composite post and core build-up composite resin

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The aim of the present study was to characterize the effects of the ultraviolet light (UV) irradiation period on the bond strength of fiber-reinforced composite (FRC) posts to core build-up resin. Three types of FRC posts were prepared using polymethyl methacrylate, urethane dimethacrylate, and epoxy resin. The surfaces of these posts were treated using UV irradiation at a distance of 15 mm for 0 to 600 s. The pull-out bond strength was measured and analyzed with the Dunnett's comparison test (α=0.05). The bond strengths of the post surfaces without irradiation were 6.9 to 7.4 MPa; those after irradiation were 4.2 to 26.1 MPa. The bond strengths significantly increased after 15 to 120-s irradiation. UV irradiation on the FRC posts improved the bond strengths between the FRC posts and core build-up resin regardless of the type of matrix resin.

Keywords: Fiber-reinforced composite, Matrix resin, Ultraviolet light irradiation, Composite resin, Bond strength

INTRODUCTION

The use of fiber-reinforced composite (FRC) posts has increased due to their good esthetic appearance and suitable mechanical properties, which are close to those of dentin11. Clinical studies have indicated that the failure rate of FRC post application ranged from 7 to 11% after 7 to 11 years of service29. The most frequently observed reasons for FRC post failure were endodontic problems, post fractures, and loss of post retention2,3). Several studies reported the usage of UV irradiation for dental appliances, such as artificial teeth13, FRC posts14, and dental implants21. A short period of UV irradiation improved interfacial bonding strengths between composite resin artificial teeth and auto-polymerized resin15. Moreover, the bond strength between the FRC post and the core build-up resin was improved by UV irradiation14, which is considered to improve the surface characteristics of several dental appliances. However, the effects of UV irradiation on dental appliances might vary due not only to the types of irradiated materials but also the UV irradiation period. The application of UV irradiation on FRC posts is an easy process and is applicable as a chairside...
treatment, although the irradiation conditions for the
to posts have not been clearly elucidated.

The purpose of the present study was to investigate
the effects of the UV irradiation conditions on various
matrix resin-based FRC posts to improve the bond
strength. The null hypothesis of this in vitro study was
that the UV irradiation period did not influence the
bonding between the FRC post and the core build-up
resin regardless of the type of matrix resin in the FRC
post.

MATERIALS AND METHODS

FRC post preparation

The materials used in the present study are listed in
Table 1. Three types of experimental FRC posts using
different matrix resins were prepared as in a previous
study. The unidirectional fiberglass of alumino-
borsilicate glass (E-glass) (Glassron roving, Asahi
Fiber Glass Co., Tokyo, Japan) was prepared without
sizing agents. The fiberglass surfaces were silanized
with 3-glycidoxypropyl trimethoxysilane for the EP-
based FRC posts and 3-methacryloxypropyl
trimethoxysilane for the PMMA- and UDMA-based
FRC posts. Three matrix resins (methyl methacrylate
(MMA)-, UDMA-, and EP-based resin) were prepared
for the FRC posts; the MMA-based resin was composed
of 97% MMA and 3% benzoyl peroxide; the UDMA-based
resin consisted of 89.1% UDMA, 9.9% triethylene glycol
dimethacrylate, and 1.0% benzoyl peroxide; and the EP-
based resin was composed of 89.3% epoxy based resin
and 10.7% epoxy hardener. A bundle of fiberglass and
matrix resin was packed into a polytetrafluoroethylene
(PTFE) tube (1.5-mm-inner diameter) at a volumetric
fiberglass fraction of 40%. The MMA- and UDMA-
based resins were heat-polymerized with a continuous
heated molding method in which the temperature in

UV irradiation

A low-pressure mercury UV lamp (10 W; GL-10, NEC
Lighting, Tokyo, Japan) was used for UV irradiation.
The UV lamp was 25.5 mm in diameter and 330 mm
long. The main wavelength of the lamp was 254 nm, plus
a small amount of 185 nm wavelength (approximately
10%). One UV lamp was equipped with an ultraviolet
sterilizing oven (DM5, Daishin Kogyo Co., Osaka, Japan).
The distance between the material examined and the
UV lamp was 15 mm in air at ambient temperature.
The UV lamp intensity at 15 mm distance was 11.5 mW/
cm², which was confirmed by a UV illuminometer (UIT-
150-A, Ushio Inc., Tokyo, Japan) with a UV detector
(VUV-S172, Ushio Inc., Tokyo, Japan; the detectable UV
range was 150–400 nm).

Bonding test specimen preparation

A pull-out bonding test was used to evaluate the
bonding efficiencies between the FRC post and the
core build-up resin, according to the previous study.

<table>
<thead>
<tr>
<th>Material</th>
<th>Product name</th>
<th>Lot No.</th>
<th>Manufacture</th>
</tr>
</thead>
<tbody>
<tr>
<td>E-glass*</td>
<td>Glass roving</td>
<td>—</td>
<td>Asahi Fiber Glass Co., Tokyo, Japan</td>
</tr>
<tr>
<td>3-methacryloyxpropyl trimethoxysilane</td>
<td>KBM 503</td>
<td>806553</td>
<td>Shin-Etsu Chemical Co., Tokyo, Japan</td>
</tr>
<tr>
<td>3-glycidoxyxpropyl trimethoxysilane</td>
<td>KBM 403</td>
<td>104419</td>
<td>Shin-Etsu Chemical Co., Tokyo, Japan</td>
</tr>
<tr>
<td>Methyl methacrylate</td>
<td>SAJ first grade</td>
<td>A8668</td>
<td>Sigma-Aldrich Japan, Osaka, Japan</td>
</tr>
<tr>
<td>Urethane dimethacrylate</td>
<td>Art resin</td>
<td>—</td>
<td>Negami Chemical Industrial Co., Ishikawa, Japan</td>
</tr>
<tr>
<td>Triethylene glycol dimethacrylate</td>
<td>3G</td>
<td>0919R</td>
<td>Shin-Nakamura Chemical Co., Tokyo, Japan</td>
</tr>
<tr>
<td>Epoxy-based resin</td>
<td>Epofix resin</td>
<td>6365-0782</td>
<td>Marumoto Struers K.K., Tokyo, Japan</td>
</tr>
<tr>
<td>Epoxy hardener</td>
<td>Epofix Hardener</td>
<td>6396-0136</td>
<td>Marumoto Struers K.K., Tokyo, Japan</td>
</tr>
<tr>
<td>Benzoyl peroxide</td>
<td>SAJ first grade</td>
<td>U1725</td>
<td>Sigma-Aldrich Japan, Osaka, Japan</td>
</tr>
</tbody>
</table>

* The chemical compositions of the unidirectional E-glass fiberglass were the same of the commercial product without sizing agent.
The FRC post was cleaned using a disposable cloth moistened with 80% ethanol, placed in an upright position in a TFE mold with a hole at the center. One end was exposed outside the mold by approximately 2 mm (Fig. 1A). The exposed FRC post was irradiated with the UV lamp. The UV irradiation period of one exposure was 0 (control), 5, 15, 30, 60, 120, 180, 300, and 600 s. The FRC post was exposed to UV radiation once, rotated at 90°, then exposed to another UV treatment. The UV process was repeated four times in order to evenly and completely irradiate the surface. The FRC posts were embedded with a core build-up resin within 10 min after irradiation. An acrylic tube (8 mm inner diameter, 2 mm high) was firmly fixed in the center of the mold (Fig. 1B). The acrylic tube was then filled with the core build-up resin (Clearfil DC Core Automix, Dentin, GC Corp., Tokyo, Japan), covered the height of the core build-up resin was adjusted and then a metal ring was placed under the core to support the bottom surface, E: FRC post and core build-up resin was placed upside down and placed in another split mold, F: prepared specimen for pull-out bonding test.

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Pull-out bonding test
The sizes of the FRC post diameter and core build-up resin thickness were measured using a digital micrometer (MDC-25M, Mitsutoyo, Tokyo, Japan) before the pull-out bonding test. Eight specimens of each group were tested using a pull-out bonding test at a crosshead speed of 0.5 mm/min until the core build-up resin separated from the FRC post using a universal testing machine (Model 1123, Instron, Canton, MA, USA). The bond strength was determined as the maximum load (N) divided by the bonding area of FRC post (mm²) and recorded in MPa.

Fracture surface observation
All the surfaces of the specimens after the bonding test were observed using a stereoscope (×25, SZ-Tr-1, Olympus Co., Tokyo, Japan) to determine the fracture mode. The fracture mode of the specimens was classified into three categories as follows: 1) interfacial fracture (between the FRC post and core build-up resin), 2) cohesive fracture (FRC post), and 3) mixed fracture (interfacial fracture and cohesive fracture of the FRC post).

FTIR observation
The molecular structural changes of the UV-irradiated surface were observed with a Fourier transform infrared spectroscopy (FTIR). Approximately 1 mL of the MMA-, UDMA-, or EP-based monomers was put in a 2-mL vial. The MMA-based monomer was heat-polymerized in a thermostatically controlled dry bath (ε-Heating Bucket, TAITEC Co., Saitama, Japan) at 65°C for 480 min, and subsequently post-cured in the heating oven at 100°C for 30 min. The polymerization processes of the UDMA- and EP-based monomers depended on the FRC post preparation. After polymerization, the specimen was cut using a low-speed saw to approximately 1 mm, polished with #800 to #1,500 SiC paper, then finished with alumina powder (particle size: 0.3 µm) using a buffing. After ultra-sonic cleaning for 5 min, each specimen was dried in a desiccator for at least 7 days. The surfaces of the specimens were observed with the FTIR (FTIR-8300, Shimadzu, Kyoto, Japan) equipped with an attenuated total reflectance accessory (DuraSampIR, ASI Technologies, Danbury, CT, USA). A total of 64 scans, whose spectral range was 4,000–600 cm⁻¹ with a resolution of 4 cm⁻¹, were recorded before (as a control) and after UV irradiation at 60 s and 24 h. For each UV irradiation condition, the spectra were recorded within 1 min after the irradiation and analyzed using software (IR-Mentor Pro, Bio-Rad Japan, Tokyo, Japan) to assign the functional groups. Three specimens of each condition were investigated.

Wetting tension
The changes in wettability of the UV-irradiated surfaces were analyzed by wetting tension measurements.
according to JIS K 6768 199924. A PMMA film was prepared using the specimens for wetting tension observation. The PMMA specimen was immersed in acetone and completely dissolved. A polyethylene mold, 20 mm wide, 70 mm long, and 70 µm thick placed on a slide glass (S1225, Matsunami Glass Ind., Tokyo, Japan) was immersed in the PMMA mixture and pulled out. The polyethylene mold on the glass slide was left in a draft chamber for 24 h to let the acetone evaporate, and then the film was removed from the glass plate.

For UDMA and EP, unpolymerized resin was poured into the polyethylene mold on the glass plate, covered with another glass plate, and polymerized according to the FRC post preparation. After polymerization, the UDMA and EP films were removed from the glass slide. The surface of the film was smooth and flat due to reproducing glass plate surface. These films were stored in a desiccator for at least for 7 days. The surfaces of the film were cleaned with ethanol immediately before the wetting tension measurements. Various fluids with wetting tension from 22.6–73 mN/m were formulated in accordance with JIS K 6768 by mixing four chemical reagents, ethylene glycol monomethyl ether (2-ethoxy ethanol) (Nacalai 1st grade, Nacalai Tesque Inc., Kyoto, Japan), formamide (JIS special grade, Nacalai Tesque Inc., Kyoto, Japan), methanol (SAJ first grade, Sigma-Aldrich Japan, Tokyo, Japan), and deionized water. One of these fluids was painted on the film using a disposable brush to create a thin layer of the fluid and left for 2 s. The maximum surface tension of the fluid, which remained on the film without collapsing, was determined as the surface tension of the film. The surface tension was considered to be within one minute after UV irradiation, and three specimens of each condition were examined. Each specimen was measured before (0 s) and after UV irradiation at 5, 15, 30, 60, 120, 180, 300, and 600 s.

### Statistical analysis

The bond strength data were analyzed with the Dunnett’s comparison test, and the differences in the bond strengths of the before- and after-UV irradiation groups were evaluated ($\alpha=0.05$).

## RESULTS

### Bond strength

The bond strengths, which ranged from 4.2 to 26.1 MPa, are summarized in Table 2. The bond strengths for the PMMA-based FRC posts after UV irradiation significantly increased regardless of the irradiation period. The 120-s UV irradiation showed the greatest value. The statistical analysis showed the greatest bond strength for the PMMA-based FRC posts after UV irradiation. The bond strength was significantly higher than the control value (0 s) ($p<0.05$).

### Table 2

<table>
<thead>
<tr>
<th>FRC post</th>
<th>UV irradiation period (s)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>0</td>
</tr>
<tr>
<td>PMMA</td>
<td>7.4 (2.7)</td>
</tr>
<tr>
<td>UDMA</td>
<td>6.9 (1.6)</td>
</tr>
<tr>
<td>EP</td>
<td>7.3 (1.6)</td>
</tr>
</tbody>
</table>

(): s.d

The values with the asterisk were significantly different from the control value (0 s) ($p<0.05$).

### Table 3

<table>
<thead>
<tr>
<th>FRC post</th>
<th>Fracture mode</th>
<th>UV irradiation period (s)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>0</td>
</tr>
<tr>
<td>PMMA</td>
<td>1) interfacial fracture</td>
<td>8</td>
</tr>
<tr>
<td></td>
<td>2) cohesive fracture of the FRC post</td>
<td>0</td>
</tr>
<tr>
<td></td>
<td>3) mixed fracture of 1) and 2)</td>
<td>0</td>
</tr>
<tr>
<td></td>
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<td></td>
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<tr>
<td></td>
<td>3) mixed fracture of 1) and 2)</td>
<td>0</td>
</tr>
<tr>
<td>EP</td>
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</tr>
<tr>
<td></td>
<td>3) mixed fracture of 1) and 2)</td>
<td>0</td>
</tr>
</tbody>
</table>
strengths significantly increased after UV irradiation for 15 to 120 s. Moreover, the bond strengths for the 5-s and 180 to 600-s UV treatment were not significantly different from that of the control group (without UV irradiation). Sixty-second UV irradiation produced the greatest value. The bond strengths after UV irradiation for the EP-based FRC posts significantly increased regardless of the UV irradiation period. UV irradiation for 300 s produced the greatest value.

Fracture modes

The fracture modes of all groups are classified in Table 3. Only interfacial fracture was observed in the control groups for each matrix resin. Mixed fracture was mainly observed in the PMMA-based FRC posts after UV irradiation and the UDMA-based FRC posts after UV irradiation for the 60-s and 120-s time periods. Interfacial fracture was mainly observed in the UDMA-based FRC post after UV irradiation for the 5-s, 180-s, 300-s, and 600-s time periods and all EP-based FRC posts after UV irradiation.

FTIR observation

Representative FTIR spectra of the three types of matrix resins before and after UV irradiation are shown in Fig. 2. Obvious differences could not be detected after 60 s UV irradiation except for PMMA at 700 cm	extsuperscript{-1}, which was assigned the peak of benzoyl peroxide. After 24 h UV irradiation, changes of several peak intensities were detected. Regarding PMMA, the wave numbers (assigned function groups) of the intensities changed are as follows: decreases at 1485 (C-H), 1450 (C-H), 1260 (C-O), 1150 (C-O), and 750 (C-H) cm	extsuperscript{-1}, increases at 1220 (C-H), 1080, and 1040 cm	extsuperscript{-1}, and broad at 1720 (C=O) cm	extsuperscript{-1}. For UDMA, the peak intensities at 1470 (C-H), 1050 (C-O), 950 (C-O), 870, 750 (C-H) cm	extsuperscript{-1} decreased, and those at 1330 and 1090 cm	extsuperscript{-1} increased. Finally, the peak intensities for EP at 1580 (C-O), 1300 (C-O) cm	extsuperscript{-1} decreased. Those at 1720 (C=O), 1650 (C=O), and 1380 (C-H) cm	extsuperscript{-1} increased.

Wetting tension

The wetting tensions of the matrix resins before and after UV irradiation, which ranged from 34 to 42 mN/m, are summarized in Table 4. Regarding PMMA, the wetting tensions after UV irradiation were greater than that of the control group (without UV irradiation), except for 600 s. Regarding UDMA, the wetting tensions after UV irradiation were not different from that of the control group. For EP, the wetting tensions after UV irradiation increased regardless of the irradiation period.

DISCUSSION

The null hypothesis of this in vitro study that the matrix resins of the FRC post and UV irradiation period did not influence the bonding efficiency to the core build-up...
However, the wetting tension values of the matrix contact angle compared to that without ozone19). The 185-nm polymer15-20), which modifies the surface tension, polymer surface by removing the organic contamination in the present study might clarify the effects of the UV irradiation. Therefore, the bond strengths measured from 0 to 600 s post, and the period of UV irradiation were different. Moreover, the mixed fracture was observed after 600-s time period in spite of smaller bond strength. This result also suggested the degradation of PMMA surface after longer UV irradiation. The main structure of epoxy resin consists the phenol unit which is chemically stable. Consequently the bond strength of the EP-based FRC post did not dramatically change after UV irradiation. The thermoplastic and thermosetting polymers exhibited decreases of molecular weight after UV irradiation19,20). Therefore, it is possible that the matrix resins of the FRC posts dissolve and swell after UV irradiation which will create IPN bonding with the core build-up resin.

The maximum bond strengths after the UV irradiation of PMMA-, UDMA-, and EP-based FRC posts were 26.1 MPa, 21.0 MPa, and 13.5 MPa, respectively. In particular, the PMMA- and UDMA-based FRC posts were much improved compared to the EP-based FRC post. Moreover, mixed fracture was mainly observed in the PMMA and UDMA-based FRC posts after UV irradiation, which suggested an improvement of the surface and potential existence of IPN bonding by UV irradiation. The pull-out bond strengths of the core build-up resin to PMMA- and UDMA-based FRC posts after sandblasting22) are reported to be 16.7 and 18.5 MPa, respectively; those after dichloromethane treatment22) were 15.0 and 7.3 MPa, respectively. The bond strengths of PMMA- and UDMA-based FRC posts after UV irradiation in the present study were similar or greater than sandblasting and dichloromethane treatment. Moreover, the UV irradiation of the FRC post was effective regardless of the type of matrix resin as well as the sandblasting. Different fracture modes among PMMA-, UDMA-, and EP-based FRC posts could be influenced by their mechanical properties, which will be evaluated in a future study.

Improving the bond strength by UV irradiation was discussed based on the amount of UV irradiated energy on the FRC post surface14). The irradiated surface energy (E) on the post surface is calculated as the function of distance and period using the following formula:

$$E = w \times t / [2 \pi \times (r + D) \times L]$$

where w is the power of the UV lamp, t is the UV irradiation period, r is the radius of the UV lamp, D is the distance between the UV lamp and the post, and L is the length of the UV lamp. The UV irradiation energy of 4.5 J/cm² was more effective than that of 3 J/cm² for commercial EP-based FRC post bonding14). The UV irradiation values of 180 and 600 s in the present study are calculated to be 3.1 and 10.4 J/cm², respectively. However, the bond strength after longer UV irradiation decreased in the present study. On the other hand, the previous study13) reported that the bond strength of the composite resin teeth and auto-polymerized resin increased after UV irradiation until 1 h, but decreased after UV irradiation for 24 h. Thus, this tendency of the bond strength changes was similar to the present study. Moreover, the distance between the specimen and the UV lamp in the present study (15 mm) was shorter than that the previous study by Loyaga-Rendon et al.13) (60 mm) using the same type of UV lamp. As a
result, the UV irradiation period until the bond strength decreased in the present study was shorter than the previous study[13]. Moreover, the irradiation periods to obtain the maximum bond strength of PMMA, UDMA, and EP-based FRC post were different. However, the wetting tensions of the PMMA, UDMA, and EP-based resin after UV irradiation slightly changed during the UV irradiation period. An explanation of the poor correlation between the parameters found might be that the wetting tension observations, as e.g. contact angle determinations, describe a static situation, whereas insertion of a FRC post into core build-up resin is a dynamic process. Even at high contact angles or poor wetting tension, the post will be entirely wetted during seating in the resin paste.

This in vitro study evaluated only the initial stage of bonding after 24 h storage in water. The bonding durability between a UV-irradiated FRC post and the core build-up resin has not been examined. Moreover, the mechanisms of improving the bond strength by UV irradiation are not clearly elucidated. Future research is necessary to evaluate the long-term stability of the bond strength between UV-irradiated FRC posts and core build-up resin and the mechanisms for improving the bond strength by UV irradiation using surface analysis methods.

CONCLUSIONS

Within the limitations of the present study, the following conclusions can be made:

1. The bond strength of UV-irradiated PMMA- and EP-based FRC posts significantly increased regardless of the irradiation period. The bond strength between the UV-irradiated UDMA-based FRC post and core build-up resin significantly increased for a UV irradiation period from 15 to 120 s.
2. The fracture mode of all the FRC posts before UV irradiation was interfacial. After irradiation, the fracture mode of the PMMA- and UDMA-based FRC posts was mainly mixed, but that of the EP-based FRC posts was still mainly interfacial.
3. The changes of several peak intensity patterns of the matrix resin were observed after 24 h UV irradiation by FTIR.
4. The wetting tension of the matrix resins of FRC posts did not obviously change after UV irradiation.

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