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Effects of Both Fiber Post/Core Resin Construction System and Root Canal Sealer on the Material Interface in Deep Areas of Root Canal

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Abstract: This study aimed to examine the resin polymerization of fiber post/core resin construction system and the interface between resin and root canal sealers, which are important for root canal sealing. We used i-TFC Luminus fiber post and i-TFC Luminus LC flow (i-TFC-L), GC fiber post and Unifil Core EM (GCF), and FiberKor post and Build-It FR (FKP), as core construction systems, and Nishika Canal Sealer BG (CS-BG), Metaseal Soft (META), and Nishika Canal Sealer EN (CS-EN) as sealers. The light transmission of fiber posts (n=5) the polymerization of core resin (n=5), and the adhesion between the sealer and core resin (n=10) were evaluated. i-TFC Luminus fiber post light transmission was significantly higher than that of other posts. Without shielding, i-TFC-L showed a significantly greater amount of polymerized resin than other systems. With shielding, although i-TFC-L showed a significantly greater amount of polymerized resin immediately after light irradiation, polymerized resin was significantly greater in GCF and FKP after 10 min. All systems adhered to CS-BG and META, but not to CS-EN. These results indicate that resin polymerization in the cavity differ among fiber post/core resin construction systems, and that adhesion of resin and sealer depends on the property of the sealer.

Keywords: Fiber post core construction system; Composite resin; Root canal sealer

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1. Introduction

The sealing of endodontically treated teeth is affected by material properties of root canal filling and fiber post/core resin construction system [1].

Endodontically treated teeth were commonly filled with gutta-percha and root canal sealer. Traditionally, eugenol-based sealers with antibacterial and sterilizing properties are used as major root canal sealers, but effective sealing ability in the root canal is not guaranteed because of its poor dentin adhesion. In the 2000s, a resin-based sealer containing 4-methacryloxyethyl trimellitic anhydride (4-META) was introduced for root canal wall adherence [2]. In recent years, bioceramics-based sealers with high biocompatibility that bond to the root canal wall by inducing hydroxyapatite formation have emerged [3,-5]. In modern root canal filling, resin-based and bioceramics-based sealers play an important role in root canal sealing through adhesion/bond to the root canal wall [6-9].

Fiber posts, instead of metal posts, are now used in the restoration with composite resin because of their elastic moduli similar to dentin, resulting in a reduction in root fractures [10-13]. For successful core construction using fiber posts, polymerization of the composite resin in the deep areas of the root canal is important for successful core construction using fiber posts. However, fiber post structure has been reported to affect the polymerization of composite resin in deep areas of the root canal [14]. Furthermore, complete polymerization of composite resin for construction does not occur in the deep areas

of root canal [15-18], and the unpolymerized layer remains in the post cavity, even with the use of dual-cure type composite resin for chemical polymerization [19, 20]. Recently, a construction system combining a light cure composite resin and fiber post with high translucency has been developed and clinically applied to increase resin polymerization rate in deep areas of the root canal [21-25]. Root canal filling materials and core construction materials have improved the sealing effectiveness in each development process.

In clinical practice, the root canal sealer and core resin are used without considering the difference in the properties of each material. In addition, the adhesion of fiber post/core resin construction system or root canal sealer to dentin has been reported extensively in previous research. However, there are few reports on the material interface between the composite resin used for construction and the sealer for the root canal filling, which is an important factor in achieving a root canal monoblock and complete root canal closure after core construction [26-30].

This study aimed to investigate the adhesion between core resin of the construction system and the root canal sealer in the context of material-property differences between the fiber post/core resin construction system and root canal sealer. This study was based on two null hypotheses. The first null hypothesis was that core resin of each core construction system polymerizes completely immediately after light irradiation. The second null hypothesis was that the core resin cannot adhere to each root canal sealer. We investigated the light transmittance of fiber posts, the extent of polymerization of construction composite resin, and the interface between the core resin and root canal filling sealer in the deep areas of the root canal.

2. Materials and Methods

Three types of core construction systems (i-TFC Luminus fiber post and i-TFC Luminus Fiber LC Flow [i-TFC-L], Sun medical Corp., Japan; GC fiber post N and GC Unifil Core EM [GCF], GC, Japan; and FiberKor post and Build-It FR [FKP], Pentron Corp., USA), and three types of root canal sealers (Nishika Canal Sealer BG [CS-BG], Nishika, Japan; Metaseal Soft [META], Sun medical Corp., Japan; and Nishika Canal Sealer EN [CS-EN], Nishika, Japan) were investigated (Table 1).

2.1. Fiber Post Transparency

Each fiber post was cut into 18 mm pieces and irradiated using a light irradiator (Radii Plus; SDI, Australia) directly on the non-tapered side with the silicone-shielding around the edge. The amount of light transmission (units: counts, specific wavelength: 459.5 nm) at the post apex through fiber post (n=5) was measured by a multi-channel spectroscope (FLAME-S-XR1-ES; OptoSirius, Japan) (Figure 1).

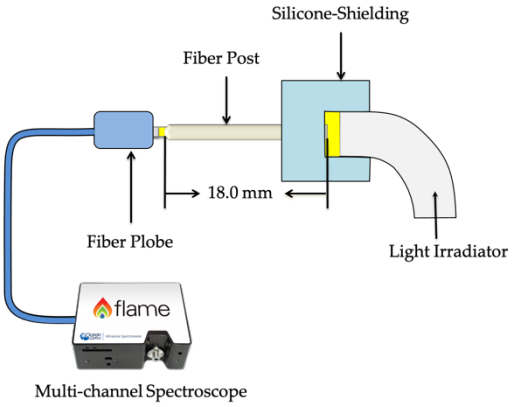

Figure 1. Schema of Fiber Post Transmission Test

Table 1. Investigated materials. Bis-MPEPP:2,2-Bis[4Bis-MPEPP:2,2-Bis[4-(methacryloxyethoxy)phenyl]propane, 4-META: 4-methacryloyloxy trimellitate anhydride, Bis-GMA: bisphenol A-glycidyl dimethacrylate, UDMA: urethane dimethacrylate , 4-MET: 4-[2-(methacryloyloxy)ethoxycarbonyl]phthalic acid, HDDMA: 1,6-hexanediol dimethacrylate, HEMA: hydroxyethyl methacrylate, MDP:10-methacryloyloxydecyl dihydrogen phosphate

Manufacturer	**Shape**	**Component**	**Lot**	**Code**
Sun medical Corp., Japan	φ 1.0 mm / tapered	Borosilicate glass, Barium oxide, Dimethacrylate and diacrylate copolymer	TW12	i-TFC -L
		Bis-MPEPP, Barium Silica Glass, Aromatic diol methacrylate	RW4	
		Bond: 4-META, Acetone, Water Catalyst: Aromatic amine, Aromatic sulfinate	RW13	
GC Corp., Japan	φ 1.0 mm / tapered	Slicate glass, Copolymer of methacryliate and Bis-GMA	2001291	GCF
		Fluoro-aluminosilicate glass, UDMA	1809041	
		4-MET, methacrlate, ethanol, water	1809041	
Pentron Corp., USA	φ 1.0 mm / straight	Glass fiber, filler, Bis-GMA, HDDMA, UDMA	7537776	FKP
		Bis-GMA, UDMA, HDDMA, barium borosilicate, Silica, Silane, Camphor quinone, Benzoyl peroxide	7558119	
		HEMA, Bis-GMA, MDP, Silica, ethanol	190031	
Nishika, Japan		Bioactive glass, Fatty acid, Bismuth subcarbonate, others	K36	CS-BG
Sun medical Corp., Japan		Powder:radiopaque filler, organic filler, hydrophilic chemical initiator Liquid:4-META, HEMA, di-methacrylates, water, photo-initiator	Powder:RM1 Liquid:SX1	META
Nishika, Japan		Eugenol, Rosin, Zinc oxide, Bismuth subcarbonate, others	K2F	CS-EN

2.2. Determination of degree of polymerization

The polymerization of core resin in the root canal was determined by the weight change (%) before and after light irradiation. Regarding polymerization activation, the i-TFC-L core construction system involves photopolymerization while the other two systems have a dual-cure mode.

A Teflon block with a semi-cylindrical cavity (diameter: 3 mm; depth: 15 mm) was made as a root canal post cavity model (Figure 2a). After filling the cavity with core resin, a fiber post of the same manufacturer was inserted, and light was irradiated from the crown side. The light irradiation for 30 s was carried out with or without shielding around fiber post at the entrance of the cavity. After 0, 5, and 10 min at 37°C and 100% humidity, each specimen was isolated from the model, then immersed into acetone to remove unpolymerized resin, washed and dried for 24 h. The weight of each specimen was measured using an electronic analytical balance (AUW220; Shimadzu, Japan) (Figure 2 b).

Weight change was calculated according to the following formula:

$$\text{Weight change (\%)} = (D-B)/(C-A-B)\times 100(\%), \quad (1)$$

A: overall mold weight with or without the use of shielding; B: fiber post weight; C: total weight after light irradiation; and D: specimen weight.

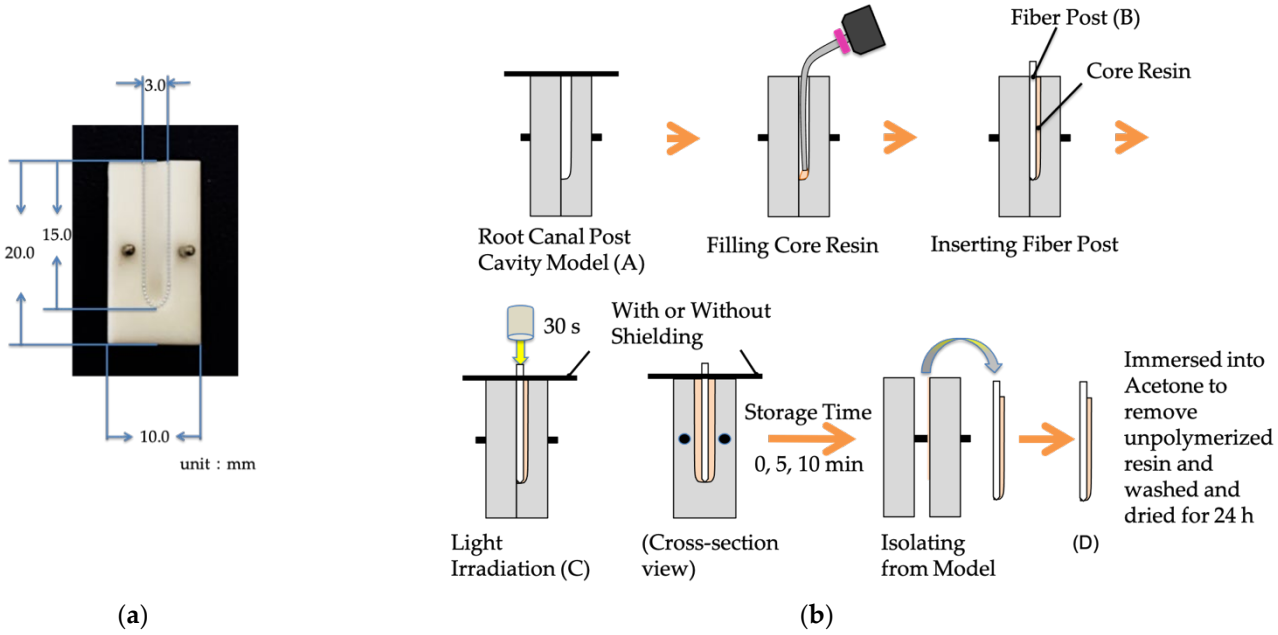

Figure 2. (a) The mold of the root canal cavity model. (b) Method of sample preparation.

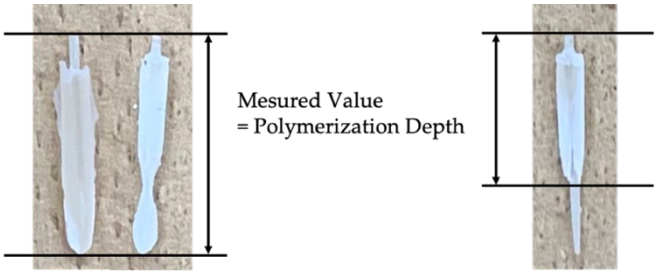

Figure 3. Polymerization Depth

Polymerization depth was evaluated by measuring the length from the coronal end of the fiber post to the most apical point of the fiber post covered with hardened resin. A

photograph and the measurement of the depth of polymerized resin are shown in Figure 3. Thirty samples were measured in these experiments (each group had 5 samples).

2.3. Interface of Core Resin and Root Canal Sealer

To analyze the interface of core resin and root canal sealer at the deep area of the root canal cavity, shear bond test and microscopic analysis after the test were carried out. Figure 4 shows the schema of the experiment. The root canal sealer was hardened in the disk-shaped mold fabricated by acrylic resin (diameter: 10 mm, height: 2 mm). After one day, an artificial light blocking root canal cavity model fabricated by Teflon tube (inner diameter: 4 mm; height: 15 mm) was placed on the hardened root canal sealer, and filled with core resin. After injection of core resin in the tube, a silicone cover was used to block the light. A fiber post by the same manufacturer was inserted into the cavity and irradiated for 30 s, and stored for 1 week. The shear bond strength was measured at a crosshead speed of 1.0 mm/min using a universal testing machine (AGS-H; Shimadzu, Japan). The prepared specimen is mounted along horizontal axis and adding shear strength along vertical axis with 1.0 mm cross head speed [31].

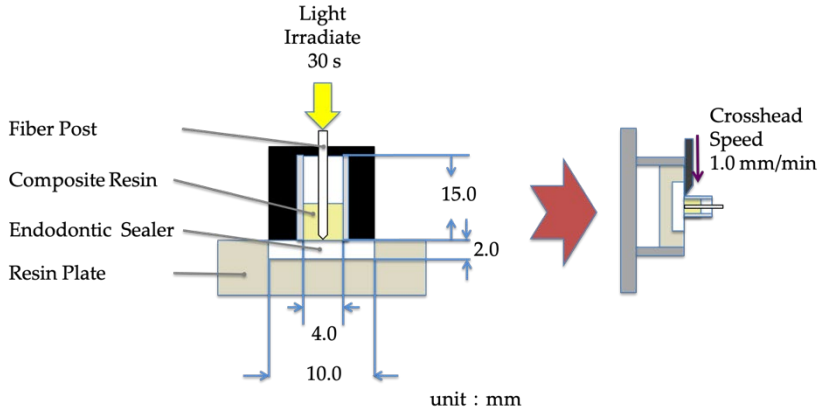

Figure 4. The schema of experiment sample.

After the shear bond test, specimens were embedded with acrylic resin and cut vertically. The cut surface was polished to #8000 and observed under a scanning electron microscope (SEM) (JCM-7000; JEOL, Japan).

2.4. Statistical Analysis

Results of fiber post transparency, core resin weight change, and shear bond test were analyzed using one-way analysis of variance (ANOVA) and Tukey's test ($P<0.05$).

3. Results

3.1. Light Transmittance of Fiber Posts

Table 2 shows each fiber post transparency. The i-TFC Luminus fiber showed significantly higher transparency values than the GC fiber post N and Fibrekor post. The Fibrekor post had a significantly lower transparency value than the other posts.

Table 2. Light Transmittance Test

Fiber Post	i-TFC-L	GCF	FKP
Counts	5195 ± 639[a]	2564 ± 667[b]	381 ± 11[c]

Different superscript letters indicate statistical differences in row (n=5, $p<0.05$)

3.2. Core Resin Weight Change

Table 3 shows the weight change of core resin before and after light irradiation. When the upper part of the root canal was unshielded from light irradiation, the weight change for i-TFC-L (0 min: 97.5±0.1%, 5 min: 96.5±0.4%, 10 min: 96.6±0.2%) was significantly higher than those of others. Regardless of the time after the light irradiation, the polymerization depth was 18.0 mm, which reached to the bottom of the cavity. GCF (0 min: 88.6±2.0%, 5 min: 91.3±2.4%) showed a significantly higher weight change than FKP (0 min: 44.7±2.0%, 5 min: 90.1±2.1%) at storage times of 0 and 5 min. At 0 min, the polymerization depth was 18.0 mm for GCF and 10.3±0.4 mm for FKP. At 10 min, the weight change of FKP (96.6±1.4%) was significantly higher than that of GCF (92.6±2.3%), and there was no significant difference between FKP and i-TFC-L. After 5 min, the polymerization depth was 18.0 mm for both GCF and FKP.

When the upper part of the root canal was shielded from light, the weight change of i-TFC-L at 0 min was the highest (66.9±4.7%), followed by GCF (41.5±4.5%) and FKP (0.1±0.1%). There was a significant difference among systems. The polymerization depth was 18.0 mm, 11.3±0.8 mm, and 0 mm for i-TFC-L, GCF, and FKP, respectively. i-TFC-L showed the same weight change regardless of time after the irradiation (0 min: 66.9±4.7%, 5 min: 72.7±2.7%, 10 min: 72.2±3.0%). At 10 min after the irradiation, GCF (82.9±1.4%) and FKP (93.3±1.4%) showed significantly higher values than i-TFC-L (72.2±3.0%) and FKP showed more than 93%, regardless of shielding (96.6±1.4% without shielding, 93.3±1.4% with shielding).

Table 3. Weight change.

Without shielding			
Storage time	**i-TFC-L**	**GCF**	**FKP**
0 min	97.5±0.1 ^{Aa}	88.6±2.0 ^{Ba}	44.7±2.0 ^{Ca}
5 min	96.5±0.4 ^{Aa}	91.3±2.4 ^{Bab}	90.1±2.1 ^{Cb}
10 min	96.6±0.2 ^{Aa}	92.6±2.3 ^{Bb}	96.6±1.4 ^{Ac}
With shielding			
Storage time	**i-TFC-L**	**GCF**	**FKP**
0 min	66.9±4.7 ^{Aa}	41.5±4.5 ^{Ba}	0.1±0.1 ^{Ca}
5 min	72.7±2.7 ^{Aa}	76.4±4.1 ^{Ab}	91.3±2.0 ^{Bb}
10 min	72.2±3.0 ^{Aa}	82.9±1.4 ^{Bc}	93.3±1.4 ^{Cb}

Same superscript capital letters indicate no significant differences (rows) for storage time. Same superscript lower-case letters indicate no significant differences between each post materials (columns). (n=5, $p<0.05$).

At 5 and 10 min after the irradiation, the polymerization depth was 18.0 mm for all systems.

3.3. Interface of the Core Resin and Root Canal Sealer

Table 4 shows the results of the shear bond test. CS-BG and META adhered to all composite resins. The interface of core resin and CS-BG showed material fractures (n=9) and interfacial fractures between the bonding material and CS-BG (n=1). The interface of

core resin and META showed material fractures occurred in all samples. On the other hand, no adhesion was observed in the interface of core resin and CS-EN.

Table 4. This table shows shear bond strength between core resin and root canal sealer.

Core resin	Sealer	MPa (Ave)	SD
i-TFC-L	CS-BG	0.22[b]	0.05
	META	4.66[a]	1.11
	CS-EN	0	0
GCF	CS-BG	0.21[b]	0.04
	META	4[a]	0.99
	CS-EN	0	0
FKP	CS-BG	0.2[b]	0.06
	META	4.41[a]	1.06
	CS-EN	0	0

Different superscript letters indicate statistical differences in vertical column (n=10, $p<0.05$)

Figure 5 shows representative results of the interface microstructure, showing adhesion of CS-BG and META to core resin via bonding layer.

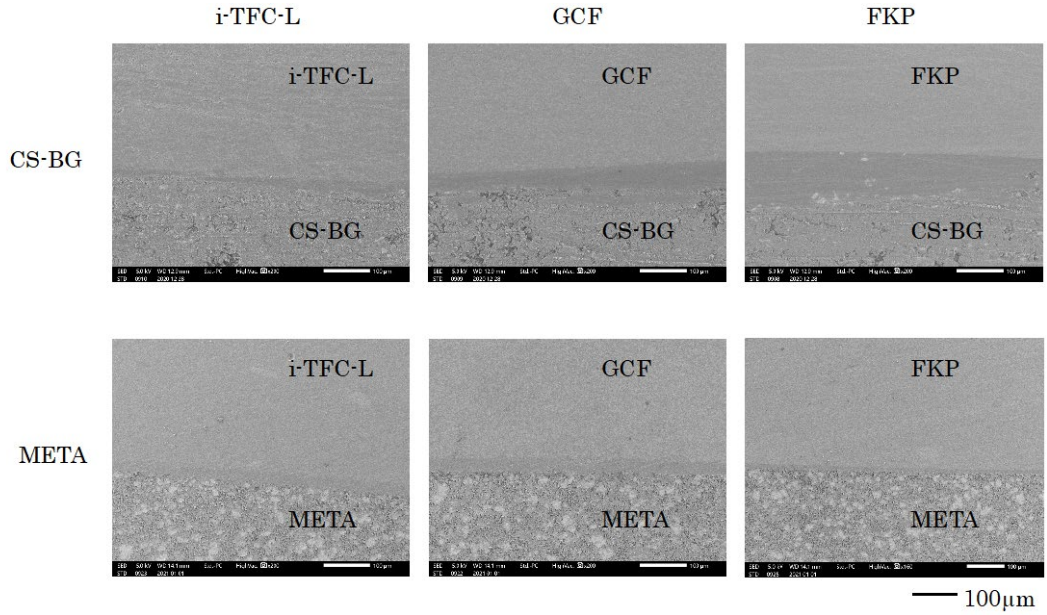

Figure 5. SEM micrographs of the surfaces of boned interface between core resin and root canal sealer (200×). Adhesive surface is observed in CS-BG group and META group regardless of type of core resin.

4. Discussion

In a core construction system using a fiber post and core resin, the core resin contacts the root canal filling material in the deep area of the root canal. Recently, single-point root canal obturation using a resin-based or bioceramics-based root canal sealer has received

acceptance [6-9]. In this obturation, main material in the root canal is root canal sealer, so the core resin mainly contacts to the root canal sealer. In the present study, the light transmittance of fiber posts, the polymerization of core resin, and the interface state between the core resin and root canal sealer, were analyzed to clarify the effects of material properties of the fiber post/core resin construction system and root canal sealer on the material interface in the deep areas of root canal, using three types of core construction systems with different fiber post structures and composite resin polymerization modes, and three types of root canal sealers with different compositions. A Teflon block with a semi-cylindrical cavity (diameter: 3 mm; depth: 15 mm) was fabricated and used as a root canal post cavity model in this study. Although various molds were used in the preliminary experiments, a Teflon block with a semi-cylindrical cavity was the easiest to separate from the hardened resin in the mold without breaking the sample.

First, the light transmittance of the fiber post and core resin polymerization of each system were examined. The examination of light transmittance showed that the transparency value of i-TFC-Luminus fiber was the highest and of FibreKor post was the lowest. For the examination of core resin polymerization, light irradiation was carried out with or without the shielding around fiber post at the entrance of the cavity to avoid direct irradiation of the resin. In this study, the degree of conversion (DC) in core resin after the irradiation was not measured to analyze the amount of polymerized resin. It is known that measuring device such as FT-IR can evaluate DC, but it measures only the outermost surface, not total amount. Therefore, hardened resin that remained after acetone immersion to remove uncured resin was considered as polymerized resin, and weight change (%) before and after acetone immersion was used to estimate core resin polymerization. Polymerization of the light-cure type composite resin of i-TFC-L was different in the presence or absence of shielding, whereas polymerization of both dual-cure type composite resins increased, regardless of shielding, in a time-dependent manner.

Without shielding, core resin polymerization of i-TFC-L was greater than the other systems for all periods. The fiber post of i-TFC-L consists of the optical fiber in the center and the glass fiber that covers the optical fiber. In this fiber post, irradiated light is scattered laterally via the glass fiber. The highest core resin polymerization without shielding may be the result of photopolymerization of the light-cure type composite resin, directly and indirectly via the light-transmitting fiber post.

With shielding, core resin polymerization of i-TFC-L was highest immediately after light irradiation, but after the irradiation, the resin polymerization of i-TFC-L did not increase. It was found that 10 min after the light irradiation, polymerization of both GCF and FKP was higher than that of i-TFC-L. Difference in the progress of core resin polymerization may have resulted from material-property differences of both fiber posts and core resin polymerization, among the three systems. With shielding, the light scattered via the glass fiber of the i-TFC-L fiber post may accelerate photopolymerization of the light-cured composite resin only during irradiation, after which the polymerization stops. On the other hand, chemical polymerization of both GCF and FKP may proceed after the irradiation. Interestingly, GCF showed higher polymerization than FKP immediately after the irradiation; however, 10 min after the irradiation, the polymerization of FKP was greater than GCF. The light transmission of the Build-it FR fiber post was very low, and photopolymerization was not accelerated with shielding. Chemical polymerization of FKP core resin may proceed even after irradiation. Fiber post of GCF has the light transmission, but it was less than half of i-TFC-L. According to previous studies, complete polymerization of the composite resin used for construction does not occur in the deep areas of the root canal. Further, these studies indicate that insufficient light irradiance for the polymerization of the dual-cure type composite resin prevents sufficient curing reaction, reduces viscosity, and hinders radical transfer, ultimately preventing chemical polymerization and resulting in an unpolymerized layer [15-18, 32]. The present results are consistent with previous reports, and may indicate that the insufficient light passing through the GCF

fiber post can interfere with chemical polymerization of the core resin in the deep areas of the canal cavity model.

Results on polymerization depth show that the extent of core resin polymerization finally reached the bottom of the post cavity in all fiber post/core resin construction systems, but the time taken for this was different between i-TFC-L and the other materials. There was polymerization of the i-TFC-L core resin to the bottom of the post-cavity 0 min after light irradiation, whereas other materials needed 5 min or more, suggesting that a sufficient storage time is necessary after light irradiation when fiber post core construction systems include dual-cure type composite resin. Based on our results, the first null hypothesis that core resin of each core construction system completely polymerizes immediately after light irradiation, was rejected.

Next, the interface of core resin and root canal sealer was analyzed. In this study, three root canal sealers were used. CS-BG is bioceramics-based, META is resin-based, and CS-EN is eugenol-based sealers. The shear bond test between the core resin and root canal sealers showed values of 0.2–0.22 MPa and 4–4.66 MPa for CS-BG and META, respectively. CS-EN did not adhere to any of the core resins. Regarding CS-EN, eugenol remained on the surface of the cured product, which inhibited the polymerization of the composite resin and prevented adherence [33]. Furthermore, SEM observation of the material interface for CS-BG and META revealed adhesion via a bonding layer with all composite resins. Calcium ions of CS-BG and the acidic monomer of the bonding agent may have bonded via a chemical reaction, and resins may have bonded to each other for META. Therefore, the second null hypothesis that core resin cannot adhere to each root canal sealer was rejected. Recently, it was reported that the interface between composite resin and calcium silicate-based cements showed enough shear bond strength at several restoration timing, suggesting that calcium silicate-based cements may allow restorative procedures with both immediate and delayed timing [34]. Results of the present study using CS-BG are consistent with the result of immediate polymerization, as seen in this study. We are now trying to clarify the shear bond strength of the interface between core resin and bioceramics-based sealers during several time frames, including delayed timing.

Overall, the present study indicates that the core resin polymerization of fiber post/resin core construction system in the root canal cavity is affected by the light transmittance of the fiber post and the polymerization type of core resin, suggesting that it is necessary to consider the properties of each material when fiber post core construction system is clinically used. Furthermore, regardless of resin polymerization type, core resin of all fiber post core construction systems adhered to the bioceramics-based and resin-based canal sealers, but not eugenol-based sealer, suggesting that the combination of root canal sealer and fiber post/core resin construction system is important to obtain the adhesion at the interface of materials, and that the use of bioceramics-based or resin-based canal sealer may be essential for the establishment of the root canal sealing. A limitation of this study is that the experiments were conducted *in vitro* and not on human teeth. Our future work will involve investigation of similar events in extracted human teeth and *in vivo*.

5. Conclusions

Within the limitations of this study, core resin polymerization in the root canal cavity differs among fiber post/core resin construction systems. In addition, adhesion of core resin and root canal sealer depends on the properties of the sealer. The use of bioceramics-based or resin-based root canal sealers that adhere to core resin is essential for root canal sealing after core construction.

Author Contributions: Conceptualization, C.K. and S.Y.; methodology, S.Y. and M.F.; validation, H.M. and S.Y.; formal analysis, H.M and S.Y.; investigation, H.M and S.Y.; resources, H.M..; data curation, H.M.; writing—original draft preparation, H.M. and S.Y. ; writing—review and editing,

C.K., T.M., A.W., H.I. and M.F.; supervision, C.K. and S.Y. ; project administration, S.Y. and C.K.; All authors have read and agreed to the published version of the manuscript.

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