THE EFFECT OF POLYMERIZATION METHODS AND FIBER TYPES ON THE

MECHANICAL BEHAVIOR OF FIBER-REINFORCED

COMPOSITE RESIN

by

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INTRODUCTION

When an anterior tooth is lost because of trauma, endodontic issues, periodontal diseases, or non-restorability, the dental professional is exposed to a myriad of complex esthetic, restorative and functional challenges. Often, for esthetic and functional purposes, dentists need to provide a temporary low-cost interim restoration before a permanent restoration such as 3-unit bridge, single dental implant, or Maryland bridge.[1] Traditionally, interim restorations are made by polymethyl methacrylate (PMMA), polyethyl methacrylate (PEMA), bis-acryl composite, or epimine resin. Due to the limited strength of these interim restoration materials, several materials have been used for reinforcement like metal wire, lingual cast metal, carbon fibers, polyethylene fibers, and glass fibers.[2]

PURPOSE OF THE STUDY

The purpose of this study was to evaluate the effect of different polymerization methods and fiber types on the mechanical behavior of fiber-reinforced interim restorations.

The null hypothesis was that 1) The two-step polymerization groups would have the same mechanical behavior as the one-step groups, and 2) The mesh fiber groups would have the same mechanical behavior as the strip fiber groups. The alternative hypothesis was that 1) The two-step polymerization groups would have greater mechanical behavior than the one-step groups, and 2) The mesh fiber groups would have greater mechanical behavior than the strip fiber groups.

REVIEW OF LITERATURE

FIBER-REINFORCED INTERIM RESTORATIONS

Lemongello[3] introduced fiber-reinforced framework with porcelain laminate veneer in a case of congenitally missing lateral incisors and advocated using the material as a conservative esthetic choice that requires minimal time. Kermanshah and Motevasselian[4] suggested that combining fiber-reinforced composite and natural tooth is a cost-effective method of immediate tooth replacement. Bejamine and Kurtzman[5] proposed that a fiber-reinforced fixed partial denture (FPD) can serve as a long-term temporary restoration or an interim restoration while an implant osseointegrates. In addition, this technique was considered a reversible procedure because the adjacent teeth were not prepared. While numerous clinicians use fiber-reinforced FPD to restore anterior missing teeth, a few clinicians have noted the same design is also applicable in the posterior area.[6]

FIBER-REINFORCED FIXED PARTIAL DENTURES

Although multiple clinical studies have advocated using fiber-reinforced FPDs as an alternative option to conventional FPDs, van Heumen et al.[7] systematically reviewed clinical studies of fiber-reinforced resin bonded FPDs and found the overall survival rate was 73.4 percent at 4.5 years. Furthermore, the review concluded the delamination of the veneer material, the wear, and the debonding to be the main reasons for failure of fiber-reinforced resin bonded fixed partial dentures. Meanwhile, in a clinical study, van Heumen et al.[8] described that failure of surface retention may be the main reason for crack formation compared to inlay-retained design. Jokstad et al.[9] pointed out that poor

adhesion between veneer material and fibers seems to be the general reason for debonding. Consequently, researchers have investigated the methods to increase the bonding between those two materials.

From the literature, it is known that the effectiveness of fiber reinforcement is dependent on many variables, including the quantity of fibers, length of fibers, form of fibers, orientation of fibers, adhesion of fibers to the polymer matrix, and impregnation of fibers with the resin.[10-16]

FIBER TYPES

The first important factor on the survival of the fiber-reinforced restorations is related to the fiber. Solnit[14] reported silane-treated fiber makes the mixture more homogeneous and has better reinforcement. In addition, Kolbeck et al.[17] assumed preimpregnated fibers showed better connections than nonimpregnated fibers, which have to be impregnated manually, depending on the skillfulness. Traditionally, strip fibers are used for reinforcing interim restorations. Rashidan et al.[18] suggested that the effectiveness of glass-fiber reinforcement is most evident in long-span interim FPDs. Hamza et al.[2] used four different fiber strips to reinforce PMMA interim FPDs and found all can increase fracture resistance like metal wire. Moreover, Geerts et al.[19] reported that strip fibers can increase both PMMA and bis-acryl composite interim FPDs fracture toughness. Mesh fibers, on the other hand, have been used in denture reinforcement or repair. Kanie et al.[20] tested four different thicknesses of woven fibers to reinforce denture base resin and recommended that woven glass fiber is an effective reinforcement in denture base resin. Furthermore, Hedzelek and Gajdus[21] concluded that both mesh fiber and strip fiber can increase the mechanical strength of the acrylic resin palatal denture bases. Due to the

weaving pattern in the mesh fibers that prevents the lateral displacement of the fibers, the

mesh fibers typically provide a better reinforcing effect then the strip fibers. Hence, it is

possible that we can fold the mesh fibers into strip to use for interim restoration

reinforcement. For example, Fahmy and Sharawi[22] theorized that both mesh and strip

fibers can alter specific provision resins fracture strength and modulus. No experimental

proof, however, has been provided.

POLYMERIZATION METHODS

Another important factor on the survival of the fiber-reinforced restorations is the

polymerization method. Dentists can use the one-step method or two-step method to

apply the fibers. In the one-step method, the dentist adapts the fibers on patient's teeth

right next to the space of the missing tooth. The dentist then uses a matrix to apply

composite resin to build up the restoration, followed by polymerization. In the two-step

method, the dentist firstly takes an impression, pours cast, and adapts the fibers on the

cast, followed by polymerization. The polymerized fibers are then moved to the patient's

mouth to continue the restoration build-up step as described above. The advantage of the

one-step method is efficiency and time-saving. The drawback is the challenge to control

the intra-oral environment to be moisture-free and to provide a good adaptation of the

fiber to the tooth. The advantage of the two-step method is the ease of adapting the fiber

to provide a better fit to the cast. The drawback is that it is more time-consuming.

Although it is well-documented that many factors influence the fiber reinforcement, little

information exists about the effects of different polymerization methods. Bertassoni et

al.[23] compared the effects of the two-step polymerization method and the one-step

polymerization method on the flexural strength and elastic modulus of a reinforced auto-

polymerized and a heat-polymerized acrylic resin reinforced by preimpregnated fibers.

The results showed that the two-step method improved the overall mechanical behavior

of reinforced auto-polymerized acrylic resins more significantly than the one-step method.

The authors suggested that the structural damage occurred on the interaction of the

polymerizing acrylic resin mixture and that the unpolymerized impregnating resin was

the reason to decrease the mechanical properties. This conclusion was also supported by

the study of Ballo et al.[24]

Though the study of Bertassoni et al.[23] proved the different polymerized methods

affect the fiber reinforcement effect on the denture base resins, the effect of the

polymerization method on interim restorations is still unclear. Hence, the present study is

to understand the effect of different polymerization methods on the mechanical behavior

of fiber-reinforced composite resin.

MATERIALS AND METHODS

MATERIALS

A light-polymerized composite resin (Filtec Z250, 3M ESPE) was used as the restorative material. Commercially-available unidirectional glass fiber (eFiber, PREAT Corp.) and mesh glass fiber (Perma Mesh, PREAT Corp.) from the same manufacturer were used to reinforce the composite resin (Table I). The eFiber is a bis-GMA and PMMA pre-impregnated strip-type product with 60 wt% glass fiber with a dimension of 200-μm diameter x 100-mm length and the Perma Mesh is a non-impregnated mesh-type product with a diameter of 22 μm and 50 x 90 mm^2 in surface area. Due to the difference in the thickness between the as-received unidirectional fiber and mesh glass fiber, the mesh glass fiber was first stacked to produce a specimen of the same thickness as the unidirectional fiber as described below.

SPECIMEN PREPARATION

A control group (n = 15) and four experimental groups (n = 15) were fabricated, representing the effects of different parameters: type of fibers (strip fibers or mesh), and polymerization methods (one-step or two-step group) (Figure 1).

Mesh Fiber Strip Fabrication

The mesh fiber was cut into 25mm x 2mm using a sharp scalpel blade while maintaining the thickness as provided by the manufacturer. These fiber strips were wetted in a light-polymerized wetting agent (PREAT Corporation) for 10 minutes in the light-isolated bag for improved adhesion of the fibers with composite resin. After wetting, the

mesh fibers were layered to be an eight-layer thickness fiber strip with the dimension of 25mm x 2mm (Figure 2).

Group I: Control Group(C) – Composite Only without Fibers

According to ISO 4049:2009 (Dentistry -- Polymer-based restorative materials), fifteen rectangular bar shaped specimens (25mm x 2mm x 2mm) were fabricated using the customized aluminum molds (Figure 3). The composite resin (FILTEK Z250, 3M ESPE) was packed into the mold, which was positioned on the top of an acetate strip. An acetate strip was then placed on top of the mold, and gentle pressure was applied to extrude excess material and achieve a consistent surface finish (Figure 4). The resin was light polymerized using a dental curing unit (Demi Plus LED Light Curing System, Kerr, USA) with a wavelength of 450 nm to 470 nm at 1100 mW/cm^2 at both the top and bottom of the specimens. Six light polymerizing cycles of 5 seconds each were necessary to cover the entire length of the specimen (3 cycles on each side) [25].

Group II: Strip fiber/ One-step group (S/O)

Fifteen rectangular bar-shaped specimens (25mm x 2mm x 2mm) were fabricated using customized aluminum molds. One strip of eFiber was cut into 25-mm length using a sharp scalpel blade while maintaining the thickness as provided by the manufacturer and positioned on the bottom as the first layer. A single-component bonding agent (ADPER Single Bond 2, 3M ESPE) was applied in multiple coats with a scrubbing technique and allowed to penetrate the fibers for 20 seconds. The composite resin (FILTEK Z250, 3M ESPE) was packed above the fiber as the second layer into the mold. The rectangular bar shaped specimens therefore consist of two layers: a first layer of 0.2-

mm-thick eFiber and a second layer of 1.8-mm-thick composite resin (Figure 1b). An acetate strip was then placed on top of the mold and gentle pressure was applied to extrude excess material and achieve a consistent surface finish. The resin was light polymerized using a dental curing unit (Demi Plus LED Light Curing System, Kerr, USA) with a wavelength of 450 nm to 470 nm at 1100 mW/cm^2 at both the top and bottom of the specimens. Six light polymerizing cycles of 5 seconds each were necessary to cover the entire length of the specimen (3 cycles on each side).

Group III: Mesh fiber/ One-step group (M/O)

Fifteen rectangular bar-shaped specimens (25mm x 2mm x 2mm) were fabricated using the customized aluminum molds. One mesh strip was positioned on the bottom as the first layer. The composite resin (FILTEK Z250, 3M ESPE) was packed above the fiber as the second layer into the mold. An acetate strip was then placed on top of the mold and gentle pressure was applied to extrude excess material and achieve a consistent surface finish. The specimen was light polymerized using a dental curing unit (Demi Plus LED Light Curing System, Kerr, USA) with a wavelength of 450 nm to 470 nm at 1100 mW/cm^2 at both the top and bottom of the specimens. Six light polymerizing cycles of 5 seconds each were necessary to cover the entire length of the specimen (3 cycles on each side).

Group IV: Strip fiber/ Two-step group (S/T)

Fifteen rectangular bar-shaped specimens (25mm x 2mm x 2mm) were fabricated using the customized aluminum molds. One strip of eFiber was cut into 25-mm length using a sharp scalpel blade while maintaining the thickness as provided by the

manufacturer and positioned on the bottom as the first layer. The fibers were light

polymerized using a dental curing unit (Demi Plus LED Light Curing System, Kerr,

USA) with a wavelength of 450 nm to 470 nm at 1100 mW/cm^2 at both the top and

bottom of the specimens. Three light polymerizing cycles of 5 seconds each were

necessary to cover the entire length of the fiber. After polymerization, the single-

component bonding agent (Primer & Bond, 3M ESPE) was applied in multiple coats with

a scrubbing technique and allowed to penetrate the fiber for 20 seconds. The composite

resin (FILTEK Z250, 3M ESPE) was packed above the fiber as the second layer into the

mold. An acetate strip was then placed on top of the mold, and gentle pressure was

applied to extrude excess material and achieve a consistent surface finish. The specimen

was light polymerized using a dental curing unit (Demi Plus LED Light Curing System,

Kerr, USA) with a wavelength of 450 nm to 470 nm at 1100 mW/cm^2 at both the top and

bottom of the specimens. Six light polymerizing cycles of 5 seconds each were necessary

to cover the entire length of the specimen (3 cycles on each side).

Group V: Mesh fiber/Two-step group (M/T)

Fifteen rectangular bar-shaped specimens (25mm x 2mm x 2mm) were fabricated

using the customized aluminum molds. One mesh strip was positioned on the bottom as

the first layer. The fibers were light polymerized using a dental curing unit (Demi Plus

LED Light Curing System, Kerr, USA) with a wavelength of 450 nm to 470 nm at 1100

mW/cm^2 at both the top and bottom of the specimens. Three light polymerizing cycles of

5 seconds each were necessary to cover the entire length of the mesh. The single-

component bonding agent (Primer & Bond, 3M ESPE) was applied in multiple coats with

a scrubbing technique and allowed to penetrate the mesh for 20 seconds. The composite

resin (FILTEK Z250, 3M ESPE) was packed above the mesh strip as the second layer into the mold. An acetate strip was then placed on top of the mold, and gentle pressure was applied to extrude excess material and achieve a consistent surface finish. The specimen was light polymerized using a dental curing unit (Demi Plus LED Light Curing System, Kerr, USA) with a wavelength of 450 nm to 470 nm at 1100 mW/cm^2 at both the top and bottom of the specimens. Six light polymerizing cycles of 5 seconds each were necessary to cover the entire length of the specimen (3 cycles on each side).

Each sample was polished with the composite polishing kit (Diacomp Composite Polishing Kit, Brasseler, USA). Before testing, all specimens were immersed in distilled water at 37 ± 1°C for 24 hours.[19,26,27]

MECHANICAL TESTING

Three-Point Bending Test
(Figure 5 and Figure 6)

The fracture strength and flexural modulus were determined using the three-point bending test as specified by the ISO 4049:2009. The specimens were tested using a universal testing machine (Sintech Renew 1121, Instron Engineering Corp., Canton, MA). A standard three-point bending jig was attached to the machine and connected to a computer with a specifically designed program (Test-Works 3.0 MTS Systems Co., Eden Prairie, MN). This software controlled the testing machine and recorded the breakage load and beam deflection. Before each test, the specimen thickness and width were recorded with a digital micrometer and entered into the computer. The specimens were placed on the jig and the fiber layer was positioned in the bottom (tension side). All tests were carried out using a crosshead speed of 1 mm/min. and a span length of 20 mm.

The flexural strength (S) was calculated using the following formula:

$$S = 3FL / 2db^2$$

Where:

S   Flexural strength (MPa).

F   Load at break or yield (N).

L   Distance between supports (20mm).

b   Width of the strip (mm).

d   Thickness of the strip (mm).

The flexural modulus was calculated using the following formula:

$$E = 3F_1L^3 / 4bd^3D_1,$$

Where:

E   Flexural modulus (MPa).

F_1 Force at deflection (N).

L   Distance between supports (20 mm).

b   Width of the strip (mm).

d   Thickness of the strip (mm).

D_1 Deflection at linear region of load deflection curve.

Microhardness

One broken specimen from each group was randomly chosen for microhardness testing by using a Knoop microhardness testing machine (M-400 Hardness Tester, Computing Printer ACP-94, LECO[®], Knoop Diamond Indenter 860-538). Ten indentations were made on the fiber surface of experimental groups.

The load of the indenter was set at 100 g and an indentation time of 15 seconds dwell time was used.

The Knoop hardness number (KHN) is the ratio of the load applied to the area of the indentation calculated from the following formula: KHN = L/ l^2Cp.

Where:

L   load applied (kgf).

l length of the long diagonal of the indentation (mm).

Cp   constant relating l to the projected area of the indentation.

The units for KHN are also kg/mm^2. Higher values represent harder materials.

FAILURE MODE ANALYSIS AND
SCANNING ELECTRON MICROSCOPY

After the three-point bending test, all samples were observed for their failure modes, especially at the interface of fibers and composite resin.

The failure mode was categorized into three groups. In group A, both the fibers and composite were completely fractured into two pieces (Figure 7a). In group B, only the fibers or composite were fractured (Figure 7b). In group C, neither the fiber nor the composite were fractured (Figure 7c). Two samples from each group were randomly chosen for the cross-sectional surface observation by scanning electron microscopy (SEM).

THERMOGRAVIMETRIC ANALYSIS (TGA)

Three additional eFiber specimens were light polymerized using a dental curing unit (Demi Plus LED Light Curing System, Kerr, USA) with a wavelength of 450 nm to 470 nm at 1100 mW/cm^2 at both the top and the bottom of the specimens.

Thermogravimetric analysis (TGA) was performed with SDT-Q600 thermogravimetric analyzer (TA Instruments, USA) to determine the fiber weight content under a nitrogen atmosphere. Fiber specimens of 8 mg to 10 mg were heated from 18°C up to 650°C at a rate of 10°C/min with a holding time at 650°C for 30 min.

STATISTICAL METHOD

The data were first submitted to Levene's test to verify the normality of distribution and subsequently analyzed by analysis of variance (ANOVA). A one-way ANOVA and the Tukey's post hoc test were used to determine the significance of the flexural strength, flexural modulus, and microhardness between the control and testing groups. The effect of fiber types (mesh, strip) and polymerization methods (one-step, two-step) on flexural strength, flexural modulus, and microhardness was assessed using two-way ANOVA and the Tukey's post hoc test. All tests were performed at a significance level of 5 percent in SPSS 20.0 software (IBM, New York, USA).

RESULTS

FLEXURAL STRENGTH

Mean flexural strength (MPa) and the standard deviation for each of the five groups are presented in Table II (a) and Figure 8. The statistical analysis indicated that the flexural strength of strip fiber groups was significantly higher than for other groups ($p < 0.05$). However, there was no significant difference in the flexural strength between mesh fiber groups and the control group. The results of the present study revealed that fiber types affect the flexural strength of test specimens ($F = 469.48$; $p < 0.05$), but the polymerization methods have no significant effect on flexural strength ($F = 0.05$; $p = 0.82$). The interaction between these two variables was not significant ($F = 1.73$, $p = 0.19$) (Table II[b]).

FLEXURAL MODULUS

Mean flexural modulus (MPa) and the standard deviation for each of the five groups are presented (Table III(a), (Figure 9). The statistical analysis indicated that the value for flexural modulus of the two-step polymerization groups was significantly smaller than for other groups ($p < 0.05$). The results of this study revealed that both fiber types and polymerization steps affect the flexural modulus of test specimens ($F = 9.71$; $p < 0.05$ for fiber type; $F = 12.17$, $p < 0.05$ for polymerization method). However, the interaction between these two variables was not significant ($F = 0.40$; $p = 0.53$) (Table III[b]).

MICROHARDNESS

Mean Knoop hardness number (Kgf) and the standard deviation for each of the

five groups are presented (Table IV (a) (Figure 10). The statistical analysis indicated that the Knoop hardness number of the control groups was significantly greater than for other groups ($P < 0.05$). In addition, there was significant difference in the Knoop hardness number between strip fiber groups and mesh groups ($P < 0.05$). The results of this study revealed that both fiber types and polymerization steps affect the Knoop hardness number of test specimens ($F = 5.73$, $p < 0.05$ for polymerization method; $F = 349.99$, $p < 0.05$ for fiber type). The interaction between these two variables was also significant ($F = 5.73$, $p < 0.05$) (Table IV[b]).

SCANNING ELECTRON MICROSCOPY (SEM)

Figure 11 showed the low magnification SEM images of strip fibers. Each glass fiber in the strip fibers was compacted densely into the threads and then multiple threads were combined into a strip (Figure 11a). In addition, the image also showed each strip fiber was pre-impregnated with PMMA and bis-GMA. After using the solvent to dissolve the resin, the SEM image showed that the strip fiber was unidirectionally oriented (Figure 11b). In the higher magnification images (Figure 12), each fiber dimension was 16 μm to 17 μm.

Figure 13 and Figure 14 showed two different magnification images of the mesh fiber. In a low magnification image (Figure 13), the mesh fiber was oriented into the net type and the loose connection was noted between the fibers. Lots of defects were noticed. Furthermore, the high magnification image (Figure 14) showed the dimension of each fiber was 5 μm to 6 μm. Although the manufacturer claimed the mesh fiber was non-impregnated (Table I1), both images showed that a thin layer of resin was placed over the mesh fiber structure.

THERMOGRAVIMETRIC ANALYSIS (TGA)
FOR FIBER CHARACTERISTICS

Because the additional PMMA and Bis-GMA were pre-impregnated on the strip

fibers, the thermogravimetric analysis was done to verify the exact fiber content. The

TGA result revealed 57.93±1.64 wt% fiber content in the strip-type fibers (Figure 15).

FAILURE MODE

The failure modes of all specimens were listed in Table V. The control group

showed all complete fractures (15/15); the M/O group demonstrated both partial fractures

(9/15) and complete fractures (6/15); the M/S groups showed the similar pattern in both

partial fractures (8/15) and complete fractures (7/15) like the M/O group; the S/O group

showed mostly non-fracture (12/15) and few partial fractures (3/15); the S/T group also

demonstrated non-fracture (6/15) and partial fractures (9/15) without any complete

fracture. With fiber reinforcement, the fracture mode tends to change from complete

fracture to partial fracture or non-fracture. In addition, the polymerization methods did

not change the failure mode in the same fiber materials. However, the difference of the

partial fractures between the mesh and strip fiber groups was noted. The partial fractures

on the strip fiber group demonstrated the fracture lines were between the fibers and the

composite and that the bottoms of the fibers were still intact. Furthermore, some partial

fractures mode in mesh fiber groups were close to complete fracture mode and just

slightly connected with mesh fibers.

SEM OF FRACTURE SAMPLES

Figure 16 showed the low magnification SEM image of the fracture strip fiber

sample. SEM images revealed cohesive failure accompanied by the pullout and bending of the fiber strips, as well as the delamination of the composite resin from the fibers. Under the higher magnification, SEM images of strip fiber sample (Figure 17) showed the facture and deformation of the composite resin. Meanwhile, the cracks were also noticed on the composite resin but not obvious on the fiber strips. However, the bonding between the fracture fragment of the composite resin and strip fibers was still intact. In addition, both SEM images of fracture strip fiber samples showed good incorporation between the preimpregnated glass fibers and the composite resin.

The low magnification SEM image of the fracture mesh fiber sample (Figure 18) revealed interfacial failure followed by delamination of both the composite resin and the mesh fibers. The pullout of mesh fibers was also noticed and more fiber fragments existed over the sample surface. In addition, the high magnification SEM image (Figure 19) showed the fracture line over the mesh fiber as well as the composite resin. The cavity on the composite resin was the evidence of the pullout of mesh fiber under the force. However, some spacing between the mesh fibers revealed the incorporation between the mesh fibers and composite resin was not as good as the strip fibers.

In conclusion, all SEM images showed the deformation and dislocation of the fibers and composite under the impact. However, the different patterns of fracture and bonding on both fibers and composite resin demonstrated the impact of the difference between the strip and mesh fibers properties.

TABLES AND FIGURES

TABLE I

Materials used in the study

Material	Brand	Manufacturer	Chemical composition
Composite resin	FILTEK Z250	3M ESPE Dental Products	Matrix: bis-GMA, TEGDMA,EDMAB and UDMA Filler: 75-80 wt%
Pre-impregnated glass fiber	EFiber	PREAT Corporation	Glass fiber (13 μ m in diameter) 200 μ m thickness 100mm length Impregnating with bis-GMA and PMMA resin
Non-impregnated glass fiber	Perma Mesh	PREAT Corporation	22 μ m thickness 50mm*90mm surface area
Bonding agent	ADPER Single Bond 2	3M ESPE Dental Products	bis-GMA, UDMA, EDMAB, DMA 25-35wt% Ethyl alcohol 5-15wt% HEMA 10-20wt% Nanofiller silica

TABLE II (a)

Flexural strength and p value of specimens

Group	N	Mean±SD	P-value from ANOVA
Control	15	140.53 ± 13.67^b	<0.05
M/O	15	132.60 ± 35.30^b	
M/T	15	155.10 ± 35.30^b	
S/O	15	467.73 ± 75.40^a	
S/T	15	451.85 ± 73.74^a	

TABLE II (b)

Results of ANOVA for flexural strength

Source	df	Sum of Square	Mean Square	F ratio	P-value
Polymerization methods	1	5465712.71	163.98	0.05	0.82
Fiber type	1	1497236.50	1497236.50	469.48	<0.05
Interactions	1	5522.50	5522.50	1.73	0.19
Error	56	178590.18	3189.11		
Total	60	7147225.87			

TABLE III (a)

Flexural modulus and p value of specimens

Group	N	Mean±SD	P-value from ANOVA
Control	15	11571.64 ± 1504.79^a	<0.05
M/O	15	9757.68 ± 1028.67^b	
M/T	15	$8391.02 \pm 1061.92c$	
S/O	15	$10581.69 \pm 1613.90^{ab}$	
S/T	15	9634.03 ± 1345.01^{bc}	

TABLE III (b)

Results of ANOVA for flexural modulus

Source	df	Sum of Square	Mean Square	F ratio	P-value
Polymerization methods	1	20085381.56	20085381.56	12.17	<0.05
Fiber type	1	16022123.13	16022123.13	9.71	<0.05
Interactions	1	658349.56	658349.56	0.40	0.53
Error	56	92393955.87	1649892.07		
Total	60	5648518285			

TABLE IV (a)

Knoop hardness value and p value of specimens

Group	N	Mean±SD	P-value from ANOVA
Control	10	81.82 ± 10.35^a	<0.05
M/O	10	13.36 ± 2.14^b	
M/T	10	13.36 ± 1.87^b	
S/O	10	3.66 ± 0.28^c	
S/T	10	5.86 ± 0.58^c	

TABLE IV (b)

Results of ANOVA for Knoop hardness value

Source	df	Sum of Square	Mean Square	F ratio	P-value
Polymerization methods	1	12.10	12.10	5.73	<0.05
Fiber type	1	739.60	739.60	350	<0.05
Interactions	1	12.10	12.10	5.73	<0.05
Error	36	76.08	2.11		
Total	40	4123.22			

TABLE V

Failure modes of the specimens categorized by the
fracture line's location and propagation

	Control	M/O	M/T	S/O	S/T
A: complete fracture	15	6	8		
B: Partial fracture		9	7	3	6
C: Non-fracture				12	9

FIGURE 1.   The diagrams of the sample used in this study (a) control
             group; (b) experimental groups.

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FIGURE 2.   Schematic diagram of the pre-impregnated mesh strip used in the study.

FIGURE 3.   The metal mold used for sample fabrication.

FIGURE 4.   The part of process of sample fabrication.

FIGURE 5.   The control samples being tested for flexural strength and modulus.

FIGURE 6. The breakage of a sample during flexural strength testing.

a.

b.

c.

FIGURE 7.   The failure modes of the samples after flexural strength testing
(a) Complete fracture; (b) Partial fracture; (c) Non-fracture.
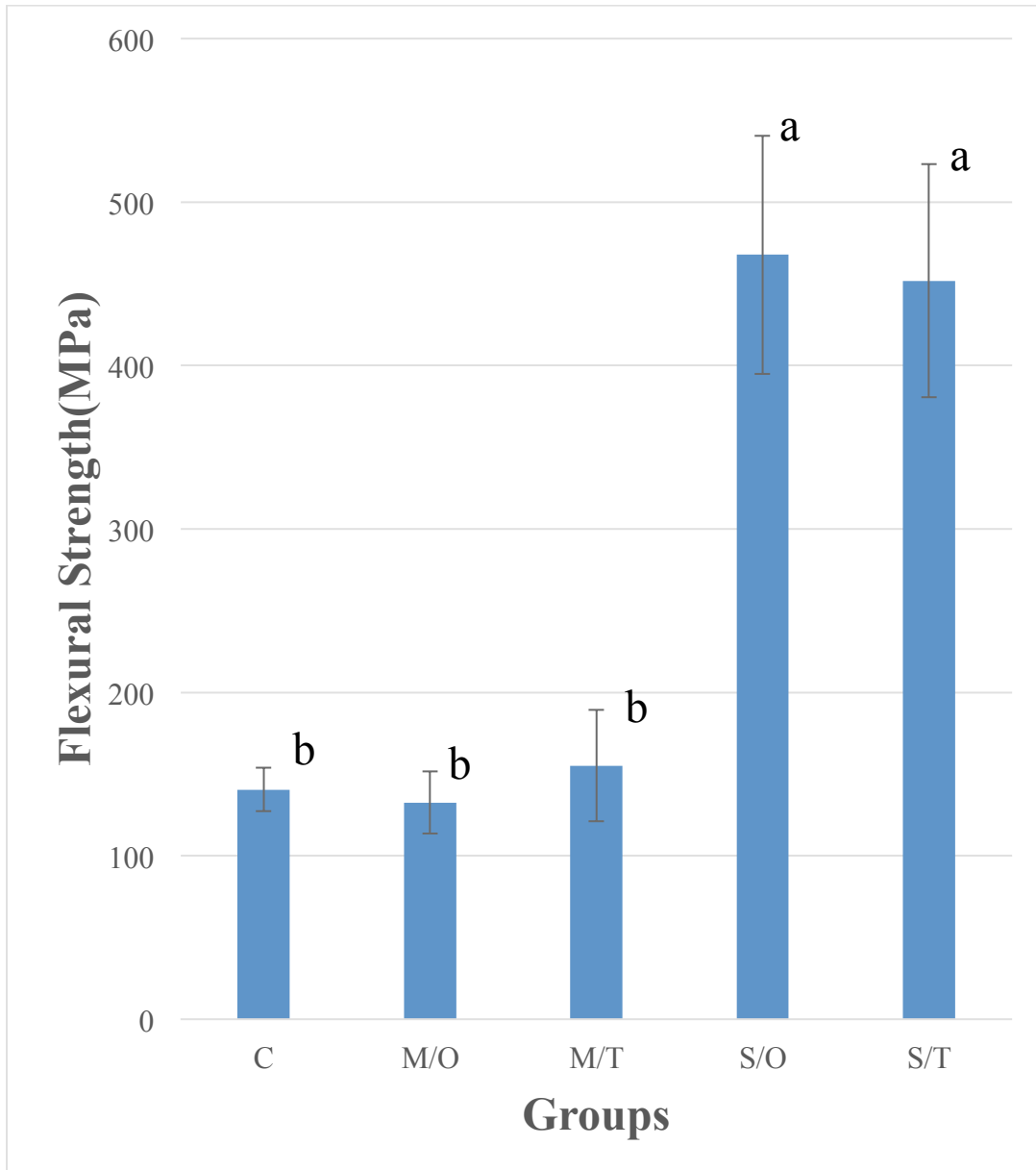
FIGURE 8. Mean flexural strength with standard deviations.
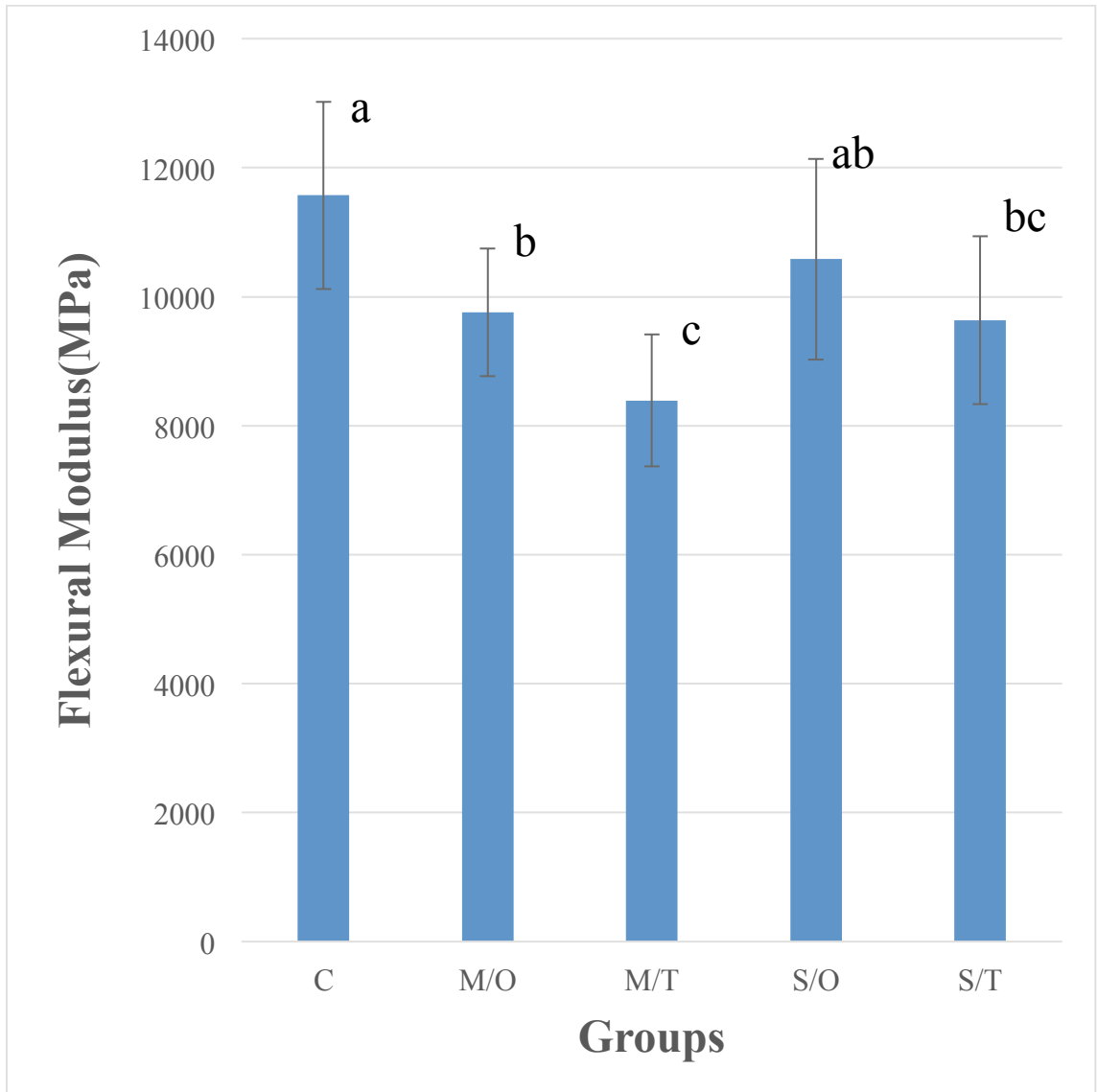
FIGURE 9.   Mean flexural modulus with standard deviations.
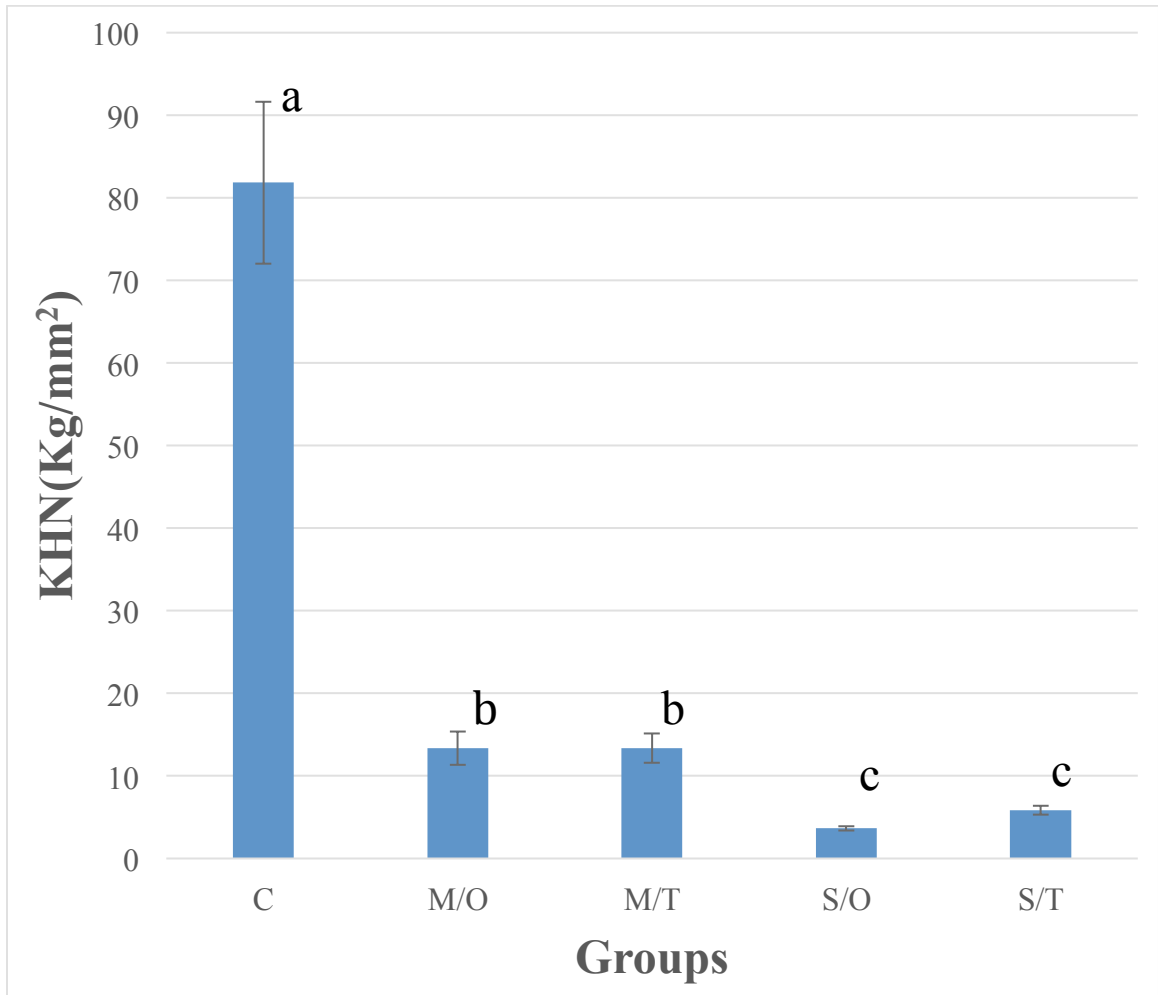
FIGURE 10.   Mean Knoop hardness number (KHN) with standard deviations.

FIGURE 11.   SEM images of eFiber. (a) Original (X150) (b) Solvent treated (X100).

FIGURE 12. SEM image of eFiber (X350).

FIGURE 13.   SEM image of Perma Mesh (X100).

FIGURE 14. SEM image of Perma Mesh (X500).

FIGURE 15.   Characteristic thermogravimetric analysis of the eFiber studied, indicating the amount of fiber left in weight%.

FIGURE 16. SEM image of eFiber fracture sample (X100).

FIGURE 17.   SEM image of eFiber fracture sample (X500).

FIGURE 18. SEM image of Perma Mesh fracture sample (X100).

FIGURE 19.   SEM image of Perma Mesh fracture sample (X500).

DISCUSSION

Z250: PREVIOUS STUDY

The control material studied in this project was 3M Filtek Z250 composite. Previous studies have reported the value of Z250 composite for flexural strength, flexural modulus, and microhardness. Blackham et al.[28] reported the mean values of the flexural strength (MPa) of composite Z250 were around 130 MPa, and Borba et al.[29] reported mean values of 135.4 MPa. Meanwhile, Blackham et al.[28] reported the mean values of the flexural modulus (MPa) of composite resin were around 8000MPa. Borba et al.[29] reported mean values of Knoop microhardness for Z250 of 98.12 (KHN number). In addition, Ceballo et al.[30] reported the values were 69.8 to 78.2 when using different curing appliances and depth. Similar values were reported in another study.[31] All the previous values for mechanical behaviors were in the same range of the ones obtained for the control group in the present study for all tests performed.

COMPOSITE REINFORCEMENT

Composite has been the object of many studies. Several materials have been used to reinforce composite mechanical behavior with more or less success. However, composite restorations still fracture at certain weak areas where stress is concentrated from the masticatory forces or impacts outside the oral cavity. Factors that contribute to stress concentration enable initiation of cracks.

Fiber reinforcement has been proposed for resin-based composite restorations to increase the resistance of materials to fracture especially in high stress-bearing cavities.[32] Different fiber materials like carbon fibers,[33] polypropylene fibers,[34, 35] polyethylene fibers,[36-38] and glass fibers[14, 39, 40] were introduced. However, the glass fibers have shown the best mechanical behavior and also showed the highest esthetic, especially for anterior

restorations.

In a fiber-reinforced composite, the fibers carry the load and effectively resist the stress on the tensile surface. The SEM image in the present study (Figure 16) was consistent with this observation. In addition, the higher magnification SEM images (Figure 17 and Figure 19) also showed the formation of stress crazes and fiber-composite debonding. Both can explain that stress was transferred from the fibers to the composite resin before the failure. The fracture line passes through the fibers, and composite resin was also evident.

FIBER TYPES

Dyer et al.[41] used polyethylene fiber and glass fiber to reinforce composites. The authors described that unidirectional glass fiber presented better outcomes than woven glass fiber and polyethylene fiber. In addition, Sharafeddin et al.[36] used two types of fibers to reinforce Z250 composite, and the results showed the glass fiber had a more significant influence than polyethylene fiber on flexural strength. The findings of the present study were in agreement with the previous studies demonstrating that strip glass fiber improves the mechanical behavior of composite resin.

In the present study, however, mesh fiber reinforcement did not show the significant differences when compared with unreinforced specimens. This result was not consistent with the previous studies.[21, 42-44]

Eronat et al.[32] investigated the effect of glass fiber layering on the flexural strength of microfill and hybrid composites. Their results showed the woven glass fiber significantly increased the flexural strength of the specimens. However, the data indicated the flexural strength of the specimens was less than 100 MPa. In the present

study, the flexural strength of the control group was 140.5 MPa. A possible explanation for our result is that new composite material has better mechanical behavior and that mesh fiber is not strong enough to provide further reinforcement. This conclusion is consistent with the conclusion of Ellakwa et al.[45]

Dikbas et al.[46] investigated three different E-glass fiber forms to reinforce PMMA and concluded that the woven-fiber-added group did not make a significant difference. The authors described that the direction of glass fibers is an important point regarding fiber-reinforced polymer. This was consistent with the conclusions of Dyer et al.[47] In addition, Kanie et al.[48] also demonstrated that woven fibers provided minor reinforcement even if it is two-direction reinforcement. These findings are consistent with the work of Loewenstein, who concluded that Krenschel's factor (the effectiveness of the woven fiber reinforcement) is smaller for woven fiber reinforcements than for unidirectional fiber reinforcements.[49] Furthermore, Polacek et al.[50,51] said that multidirectional E-glass fiber cannot be recommended for use in combination with the composites in their study.

In addition, the mesh fiber used in the present study was impregnated with the wetting agent according to the manufacturer's instruction. The air bubble and excess monomer may inhibit the adhesion between the mesh fiber and the composite.[52] In addition, Vallittu[43] suggested the use of a mixture of polymer powder and monomer liquid instead of a plain monomer liquid could avoid the creation of an excess of monomers inside the fiber reinforcement.

In this experiment, the higher magnification of SEM images (Figure 17 and Figure 19) showed the different bonding patterns on strip fiber and mesh fiber. The

incorporation of strip glass fibers and composite resin improved performance better than mesh fibers and composite resin, and less porosity was noticed between the strip glass fibers. This observation implied the wetting procedure of mesh fiber could be affected by the air bubble and excess monomer as mentioned by Tirapelli et al.[52] In addition, the SEM images showed more fracture for mesh fibers than strip fibers. These results demonstrate that the mechanical behavior between the strip and mesh fibers is significantly different and affects the resistance to fracture. The large differences between the glass fiber diameter could explain the differences in load-carrying capacity between strip glass fibers and mesh fibers.

POLYMERIZATION METHODS

The advantage of the one-step method is efficiency and time-savings. In addition, some authors proposed the one-step method could decrease the formation of a resin-rich inhibited layer and increase the interfacial adhesion between each layer.[51,53] However, intra-oral fiber adaptation is difficult to apply, and intra-oral moisture also affects the material adhesion.[54]

Bertassoni et al.[23] compared both polymerization methods on the flexural strength and elastic modulus of the auto-polymerized and heat-polymerized acrylic resin reinforced by preimpregnated fibers. The results showed that the two-step method improved the overall mechanical behavior of reinforced auto-polymerized acrylic resins better than the one-step method. However, in the present study, there was no significant difference between different polymerization groups for flexural strength.

Polacek et al.[51] evaluated the effect of different polymerization sequences during application of two different composites on fiber-reinforced composite. The result pointed

out that different material combinations need different polymerization sequences. It was explained that the significant effects of the fiber types and interaction between the fiber types and polymerization methods were observed, but the significant effects of the polymerization methods was not found.

MICROHARDNESS

Microhardness was used as an indirect method for assessing the degree of conversion of composite in several studies. Some researchers found that microhardness values cannot be used to compare the degree of conversion among different materials.[55] In the present study, the microhardness values between the control group and the experimental groups were significantly different, because the microhardness values measured in the experimental groups represent the mechanical behavior of the unfilled impregnating resin between the fibers. As expected, those resins will have a much lower hardness than Z250.

In present study, the two-step polymerization groups showed higher microhardness values compared with the one-step polymerization group. This is because a higher degree of conversion was expected in the matrix of the specimens that were polymerized twice. Given that the matrix represented a very small portion of the overall material in the specimen, the increase hardness number was not reflected in the flexural strength. This result was also consistent with the findings of Graoushi et al.[31]

EXPECTED FUTURE APPLICATION

Although many researchers have stated that fiber-reinforced composite can be used as an optional material for interim or permanent crown fabrication,[7-9] the clinical application methods still have differences between articles[3,5] or manufacturers' guidelines,

especially regarding layering procedures. In the present study, there was no significant difference between different polymerization methods on the flexural strength. However, within the limitations of this study, the result of the present study did not show a significant difference between different polymerization methods. No specific clinical application procedure can be suggested to increase the mechanical behavior of the fiber-reinforced composite. Further tooth-mold samples and clinical studies should be done to evaluate the effect of different polymerization methods on the mechanical behavior of fiber-reinforced composites. Then, a reliable and applicable method will be developed to decrease the possible clinical complications and treatment difficulties. Furthermore, the manufacturers can improve their fiber products for better clinical application.

SUMMARY AND CONCLUSIONS

As mentioned previously, the null hypothesis that the mesh fiber groups would have the same mechanical behavior compared with the strip fiber groups was rejected. However, the null hypothesis that the two-step polymerization group would have the same mechanical behavior compared with the one-step group was accepted.

1. Fiber types affect the flexural strength of test specimens, but the polymerization methods have no significant effect on flexural strength. Mean flexural strength for the strip fiber groups was significantly greater than for mesh fiber groups and the control group.

2. Both fiber types and polymerization steps affect the flexural modulus of test specimens. The mean flexural modulus of the two-step polymerization groups was significantly smaller than for the other groups

3. Both fiber types and polymerization steps affect the Knoop hardness number of test specimens. The mean Knoop hardness number of the control groups was significantly greater than for other groups

4. With fiber reinforcement, the fracture mode tended to change from complete fracture to partial fracture or non-fracture. However, the polymerization methods did not change the failure mode within the same fiber materials.

In conclusion, both strip and mesh fibers improved mechanical properties of composite resin and made the fractured samples easily repairable.

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ABSTRACT

THE EFFECT OF POLYMERIZATION METHODS AND FIBER TYPES ON THE

MECHANICAL BEHAVIOR OF FIBER-REINFORCED

COMPOSITE RESIN

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Background: Interim restoration for a lost anterior tooth is often needed for temporary esthetic and functional purposes. Materials for interim restorations usually have less strength than ceramic or gold and can suffer from fracture. Several approaches have been proposed to reinforce interim restorations, among which fiber reinforcement has been regarded as one of the most effective methods. However, some studies have found that the limitation of this method is the poor polymerization between the fibers and the composite resin, which can cause debonding and failure.

Purpose: The purpose of this study was to investigate the effects of different polymerization methods as well as fiber types on the mechanical behavior of fiber-reinforced composite resin.

Material and Methods: A 0.2-mm thick fiber layer from strip fibers or mesh fibers embedded in uncured monomers w as fabricated with polymerization (two-step method) or without polymerization (one-step method), on top of which a 1.8-mm composite layer was added to make a bar-shape sample, followed by a final polymerization. Seventy-five specimens were fabricated and divided into one control group and four experimental groups (n=15), according to the type of glass fiber (strip or mesh) and polymerization methods (one-step or two-step). Specimens were tested for flexural strength, flexural modulus, and microhardness. The failure modes of specimens were observed by scanning electron microscopy (SEM).

Results: The fiber types showed significant effect on the flexural strength of test specimens (F = 469.48; p < 0.05), but the polymerization methods had no significant effect (F = 0.05; p = 0.82). The interaction between these two variables was not significant (F = 1.73; p = 0.19). In addition, both fiber types and polymerization steps affected the flexural modulus of test specimens (F = 9.71; p < 0.05 for fiber type, and F = 12.17; p < 0.05 for polymerization method). However, the interaction between these two variables was not significant (F = 0.40; p = 0.53). Both fiber types and polymerization steps affected the Knoop hardness number of test specimens (F = 5.73; p < 0.05 for polymerization method. and F = 349.99; p < 0.05 for fiber type) and the interaction between these two variables was also significant (F = 5.73; p < 0.05). SEM images revealed the failure mode tended to become repairable while fiber reinforcement was

existed. However, different polymerization methods did not change the failure mode.

Conclusion: The strip fibers showed better mechanical behavior than mesh fibers and were suggested for use in composite resin reinforcement. However, different polymerization methods did not have significant effect on the strength and the failure mode of fiber-reinforced composite

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