

The Effect of Fiber Position and Polymerization Condition on the Flexural Properties of Fiber-Reinforced Composite

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Abstract

The aim of this study was to investigate the influence of the position of the fiber rich layer on the flexural properties of fiber-reinforced composite (FRC) construction. In addition, the total residual monomer content of FRC was quantitatively determined to find out the difference of the effectiveness of two types of light-curing units using liquid chromatography (HPLC). Unidirectional continuous E-glass FRC and hybrid particulate filler composite resins were used in the fabrication of test specimens. Four different positions of the FRC layer were used: compression, neutral, tension, and vertical side position. A three-point bending test (ISO 10477) was performed to measure the flexural properties of the specimens. Position of the FRC layer had a significant effect on the flexural strength ($p < 0.001$, ANOVA). Also, the type of light-curing device had an effect on flexural strength ($p < 0.001$). Specimens with FRC positioned on the compression side showed flexural strength of approximately 250 MPa, whereas FRC positioned on the tension side showed strength ranging from 500 to 600 MPa. Mean flexural modulus with FRC placed horizontally ranged between 9-12 GPa; no significant difference was found between these groups. However when fiber reinforcement was positioned vertically, the flexural modulus raised up to 16 GPa. Specimens with 24 vol% glass fibers contained 52% less residual monomer than specimens without glass fibers. The monomer content was lower in specimens polymerized with the curing device with higher polymerization temperature. In order to optimize flexural strength of low fiber volume fraction, the fibers should be placed at the tension side of the specimen.

Keywords: Fiber-reinforced composite, flexural properties, residual monomer

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Introduction

Investigation regarding fiber-reinforced composites (FRC) in dentistry has continued over 30 years.¹ During the last few years many types of FRC-materials for various dental applications have been introduced. Mechanical properties of FRCs have been reported, and it has been shown by many investigators^{2,3} that a FRC structure with continuous unidirectional fibers achieves the best mechanical properties compared to reinforcement with short fibers of random orientation. Another critical factor affecting the strength of FRCs is the adhesion between the fibers and the resin matrix. Without adequate adhesion the fiber acts as an inclusion in the resin matrix, which actually weakens the composite.^{3,4,5} Also the void volume content between the fibers decreases the flexural properties of FRCs.^{4,6}



Continuous Unidirectional
Fibers



Random Short Fibers

A higher volume fraction of fibers in the resin matrix improves the mechanical properties.^{2,7-10} Generally, fiber volume fraction of FRCs is relatively high, up to 60 vol%. However, in dental applications fiber fractions are considerably lower. This is due to the fact fibers should be covered with a layer of unfilled polymer

or with a layer of particulate filler composite in order to obtain polishable and occlusal wear-resistant surfaces.¹¹ In order to optimize the mechanical properties of dental construction with a relatively low quantity of fibers, the position and orientation of fibers should maximize the stress transfer from matrix to fibers.

The light curing process has an influence on hardness¹² and flexural properties of a composite resin.¹³ The higher the degree of monomer conversion, usually the better the mechanical properties. A higher degree of conversion can be achieved by increasing either light intensity or polymerization temperature, which results in a more homogeneous polymer matrix. The effect of vacuum and pressure conditions during the light polymerization process has also been investigated.^{14,15}

The aim of this study was to investigate the influence of positioning relatively low quantity of fibers on the flexural properties of FRC structure and to determine the total residual monomer content of test specimens after different light polymerization conditions.

Materials and Methods

Test Specimen Preparation

The materials used in this study are listed in Table 1. Sinfony Activator is an unfilled light curing monomer resin, which was used to impregnate porous polymethyl methacrylate (PMMA) preimpregnated continuous unidirectional E-(electrical)-glass fiber (Stick™, StickTech, Turku, Finland) reinforcement. Further-impregnation was made with Sinfony Activator resin for 24 hours in a dark box. Fiber reinforcement placed between Mylar strips was inserted into the mold with a fiber orientation along the axis of the test specimens (2x2x25mm). Positioning of fibers in the test specimens is described in Figure 1a and Figure 1b in Table 2. The mold was filled with particulate filler composite resin (Sinfony Dentin Shade A1), covered with Mylar strips on both sides, and compressed by glass plates. Six specimens were fabricated for each group. There were also six test specimens made of Sinfony Dentin particulate filler composite resin as a control specimen.

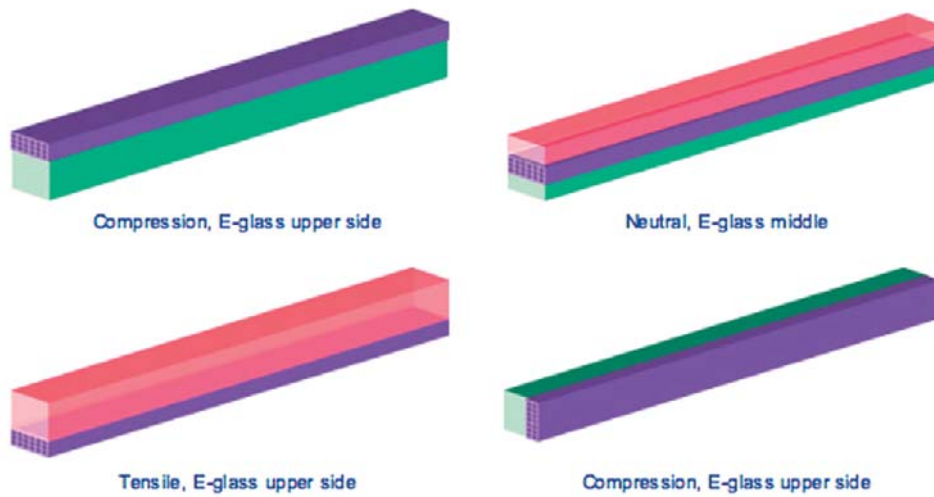


Figure 1a. Schematic diagram of cross sections of test specimens with different FRC (E-glass) locations.

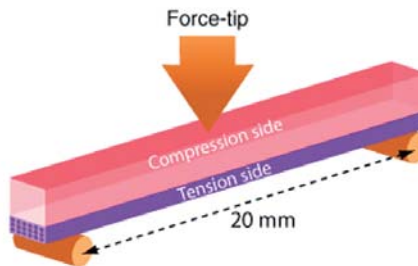


Figure 1b. Schematic diagram of test specimen with FRC on the tension side in three point bending test.

Table 1. Materials used in the investigation.

Product	Manufacturer	Chemical composition	Batch no
Sinfony Activator	Espe, Seefeld, Germany	*	03344609
Sinfony Dentin Shade A1	Espe, Seefeld, Germany	* + 50 vol% inorganic filler	FW0045230
Stick™	Stick Tech, Turku, Finland	E-glass + PMMA†	1980224-R-0037

* >90 wt% (octahydro-4,7-methano-1H-indenyl)bis(methylene)diacrylate monomers

† Polymethyl methacrylate

Table 2. Groups of the test specimens used in the investigation.

Group	Material	Light-curing oven	E-glass, volume%	Position of fiber-layer
1	Sinfony Dentin A1	Visio Beta vario	0%	-
2	Sinfony Dentin A1	LicuLite	0%	-
3	Sinfony Dentin A1	Visio Beta vario	24%*	up, middle, low†
4	Sinfony Dentin A1	LicuLite	24%*	up, middle, low†
5	Sinfony Dentin A1	Visio Beta vario	24%*	vertical†
6	Sinfony Dentin A1	LicuLite	24%*	vertical†

* Unidirectional E-glass fibers were further-impregnated with Sinfony Activator resin

† up = compression side, middle = neutral axis, low = tension side, vertical = along axial wall

The test specimens for Groups 1, 3, and 5 were polymerized initially with a Visio Alfa (3M/ESPE, Seefeld, Germany) hand-curing unit for 10 s on both sides. Subsequently, the polymerization was completed in a light-curing oven (Visio Beta Vario 3M/ESPE, Seefeld, Germany) for 15 min. The test specimens in Groups 2, 4, and 6 were initially polymerized with a Elipar Highlight (Espe, Seefeld, Germany) hand-curing unit for 10 s from both sides. Subsequently, the polymerization was completed in a light-curing device (LicuLite, Dentsply DeTrey GmbH, Dreieich, Germany) for 15 min. After polymerization, the edges of the test specimen were finished with 1200 grit silicon carbide grinding paper. Specimen dimensions (width, height, and length) were measured at three different points of each side in order to ensure dimensions of the test specimens. The specimens were conditioned in air at room temperature for 2 days before mechanical testing.

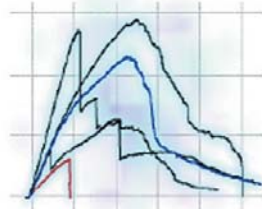
Mechanical Testing

A three-point bending test was performed to measure the flexural strength and modulus of the specimens according to the ISO 10477:92 standards¹⁶ using 20 mm span size and 1.0 mm/min crosshead speed. All samples were tested in a Lloyd material testing machine (model LRX; Lloyd Instruments, Fareham, England), and the load-deflection curves were recorded with a PC-computer software (Nexygen, Lloyd Instruments, Fareham, England).

Flexural strength (σ_f) and flexural modulus (E_f) were calculated from the formula.¹⁶

$$\sigma_f = 3 F_{\max} l / 2bh^2$$

$$E_f = S l^3 / (4bh^3)$$



Where F_{\max} is the applied load (N) at the highest point of load-deflection curve, l is the span length (20.0 mm), b is the width of the test specimen, and h is the thickness of the test specimens. S is the stiffness, $S=F/d$ (N/m), and d is the deflection corresponding to load F at a point in the straight line portion of the trace.

The mean quantity of glass fibers in the FRC test specimens was determined by combustion of the polymer matrix of the test specimens for 1 hour at

700 °C according to the following formula:

$$V_f = (W_f / r_f) / (W_f / r_f + W_r / r_r)$$

Where W_f is the weight proportion of E-glass, r_f (=2.54 g/cm³) is the density of E-glass, W_r is the weight proportion of resin and, r_r is the density of resin (= 1.22 g/cm³). The test specimens for the glass fiber quantity determination were made from resin without filler particles, i.e., from Sinfony Activator resin; the effect of particulate fillers of the Sinfony Dentine composite resin on the inorganic filler content was eliminated. Before and after combustion, the weights of the specimens were measured with a lab scale accuracy balance (Mettler A30; Mettler Instrument, Highstone, NJ, USA) with an accuracy of 0.1 mg. The particulate filler content of Sinfony Dentin composite resin was 50 vol% (according to the manufacturer's product information).

Residual Monomer Analysis

Total fraction of residual monomers was extracted from the crushed test specimens (2x2x25mm) for the quantitative analysis of the residual monomers.

The weight range of the polymer sample varied from 150 mg to 300 mg, which was then dissolved in solvent acetone for 3 days under a magnetic stirrer. High-pressure liquid chromatography (HPLC) was used to determine the total fraction of leachable residual acrylate.



A Hewlett Packard 1100 HPLC (München, Germany) equipped with an autosampler and a variable injection loop was used for the analysis of the monomers. A Hewlett-Packard diode array detector (HP 1090) was used to detect the residual acrylate monomers. External standard monomers in acetone with concentrations of 0.02 and 0.10 mg/ml were used. All standard monomers were commercially available and of HPLC grade purity. The water was distilled and then purified through a Millipore systemTM. Pure patch test substances (monomers of EGDMA, bis-GMA, bis-EMA, UDMA, BDMA, MMA, EMA, n-BMA, 2-HEMA, and TEGDMA) were used as external standards (Table 3).

Table 3. Monomer composition of products used as standard substances with HPLC.

	Chemical Composition	Manufacturer	Country
EGDMA	ethyleneglycoldimethacrylate	Chemotechnique Diagnostics	Malmö, Sweden
bis-GMA	Bisphenol-A-glycidyl dimethacrylate	Chemotechnique Diagnostics	Malmö, Sweden
bis-EMA	bishydroxyethacryloxy-propoxyphenypropane	Chemotechnique Diagnostics	Malmö, Sweden
UDMA	urethanedimethacrylate	Chemotechnique Diagnostics	Malmö, Sweden
BDMA	butanediol-dimethacrylate	Chemotechnique Diagnostics	Malmö, Sweden
MMA	methyl methacrylate	Polyscience Corp.	Niles, IL, USA
EMA	ethylmethacrylate	Polyscience Corp.	Niles, IL, USA
n-BMA	n-butylmethacrylate	Polyscience Corp.	Niles, IL, USA
2-HEMA	2-hydroxyethylmethacrylate	Polyscience Corp.	Niles, IL, USA
TEGDMA	triethyleneglycoldimethacrylate	Fluka Chemie AG	Buchs, Switzerland

The leachable monomers of the test specimens were analyzed using gradient elution with a mobile phase of acetone and water. The gradient elution started after 3 min and ended after 20 min. The analysis time was 25 min and the flow-rate was 0.8 ml/min. The column was Waters Spherisorb S5 ODS2 (Capital HPLC Ltd, Broxburn, UK) (250 x 4.6 mm I.D.). A 10 µl volume of sample solutions and external standards solutions were injected for UV-detection at two different wavelengths, 210 nm and 275 nm, with a slit set of 4 nm. UV-spectra were recorded when a peak eluted. The signal at 210 nm was used to calculate the quantitative results.

Statistical Analysis

Data were statistically analyzed using analysis of variance (ANOVA) and with Scheffe´ post hoc test with SPSS software (Statistical Package for Social Science, SPSS Inc, Chicago, IL) to determine the differences among the groups.

Results

Mechanical Testing

Mean flexural strengths and modulus for each of the groups are shown in Tables 4 and 5. Two-way ANOVA revealed the position of the FRC layer had a significant effect ($p < 0.001$) on the flexural strength (Table 6). Also, the choice of light curing device had an influence on flexural strength ($p < 0.001$). An interaction between these variables was found. The unreinforced groups had mean strengths of 123.5 ± 13.7 MPa when polymerized with LicuLite device and 90.1 ± 13.3 MPa when polymerized with VisioBeta (Figure 2) (Table 4). The groups with the FRC layer positioned horizontally on the upper side (compression side) of the specimen showed a mean strength close to 250 MPa. When the FRC layer was positioned in the middle (neutral axis) of the specimen, flexural strength was approximately 400 MPa. When the FRC layer was positioned at the lower side of

Table 4. Flexural strength of FRC test specimens polymerized with the two different light curing devices.
For the explanation of FRC layer location, see Table 2.

Position of E-glass fibers	LicuLite Strength (MPa)	Difference %	Visio Strength (MPa)
Unreinforced control	123.5 ± 13.7 ^a	26%	90.1 ± 13.3 ^a
FRC Up	248.1 ± 31.2 ^b	1%	245.8 ± 26.7 ^b
FRC Middle	435.9 ± 48.6 ^{cd}	6%	408.4 ± 41.9 ^c
FRC Low	577.7 ± 25.5 ^{de}	12%	509.3 ± 33.0 ^{de}
FRC Vertical	585.4 ± 88.5 ^{de}	25%	445.6 ± 19.3 ^{cd}

Superscript letters indicate homogenous subsets, values marked with the same letter do not differ significantly ($p < 0.05$), determined by the 1-way ANOVA post hoc Scheffe test.

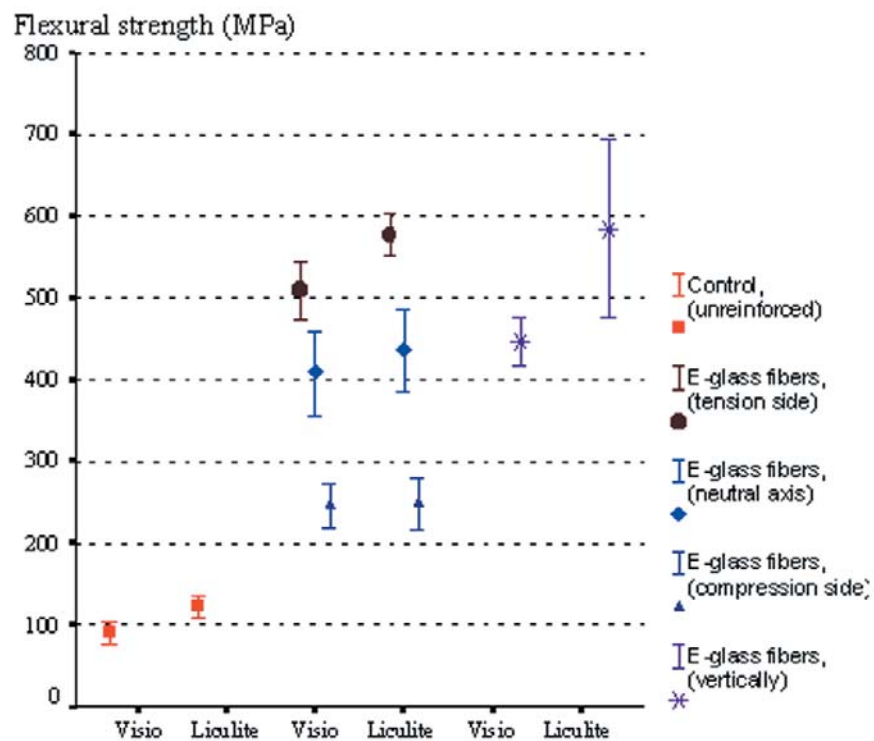


Figure 2. Flexural strength with different positions of FRC (E-glass) cured with Visio Beta vario and LicuLite light-curing devices. Vertical bars indicate standard deviations.

the specimen (tension side), the mean flexural strength was highest for both groups: 509.3±33.0 MPa when polymerized with VisioBeta device and 577.7±25.5 MPa when polymerized with LicuLite

device. If the FRC was positioned vertically, the flexural strengths were 585.4 MPa (polymerized with LicuLite device) and 445.6 MPa (polymerized with VisioBeta device).

The mean flexural modulus with the FRC layer positioned horizontally ranged between 9.1-12.0 GPa (Figure 3 and Figure 4), but no significant effects were found on the position of the FRC layer between these groups (Table 5). However,

when the FRC layer was positioned vertically, the flexural modulus was increased up to 16 GPa (Table 6 and Table 7). The mean percentage of E-glass fibers in the total volume of the test specimens was 24 vol%.

Table 5. Flexural modulus of FRC test specimens with two different light curing devices.

For the explanation of the FRC layer location, see Table 2.

Position of E-glass fibers	LicuLite Modulus (GPa)	Difference %	Visio Modulus (GPa)
Unreinforced control	6.4 ± 0.5 ^a	23.4	4.9 ± 0.5 ^a
FRC Up	12.0 ± 1.3 ^c	24.2	9.1 ± 0.8 ^b
FRC Middle	10.4 ± 0.7 ^{bc}	1.0	10.3 ± 1.4 ^{bc}
FRC Low	11.0 ± 0.4 ^{bc}	14.5	9.4 ± 0.7 ^{bc}
FRC Vertical	16.6 ± 0.2 ^d	1.2	16.4 ± 0.8 ^d

Superscript letters indicate homogenous subsets, values marked with the same letter do not differ significantly ($p < 0.05$), determined by the 1-way ANOVA post hoc Scheffe test.

Table 6. Effect of the position of the FRC layer and type of light-curing device on the flexural strength as analyzed by two-way ANOVA.

Source	SS	Df	MS	F	Sig.
Curing device	39495.2	1	39495.2	21.0	<.001
Position of FRC	630224.5	3	210074.8	111.7	<.001
Interaction	28854.4	3	9618.1	5.1	.005

Table 7. Effect of the position of the FRC layer and type of light-curing device on the flexural modulus as analyzed by two-way ANOVA.

Source	SS	Df	MS	F	Sig.
Curing device	16.0	1	16.0	20.4	<.001
Position of FRC	291.7	3	97.2	124.0	<.001
Interaction	15.0	3	5.0	6.4	.001

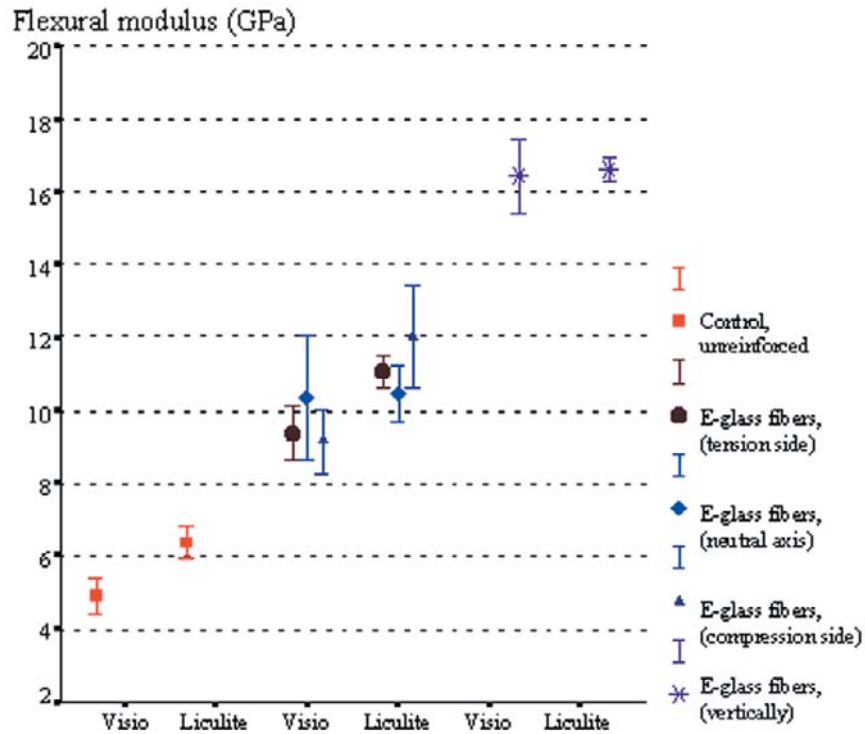


Figure 3. Flexural modulus with different positions of the FRC layer (E-glass) polymerized with Visio Beta and LicuLite light-curing devices. Vertical bars indicate standard deviations.

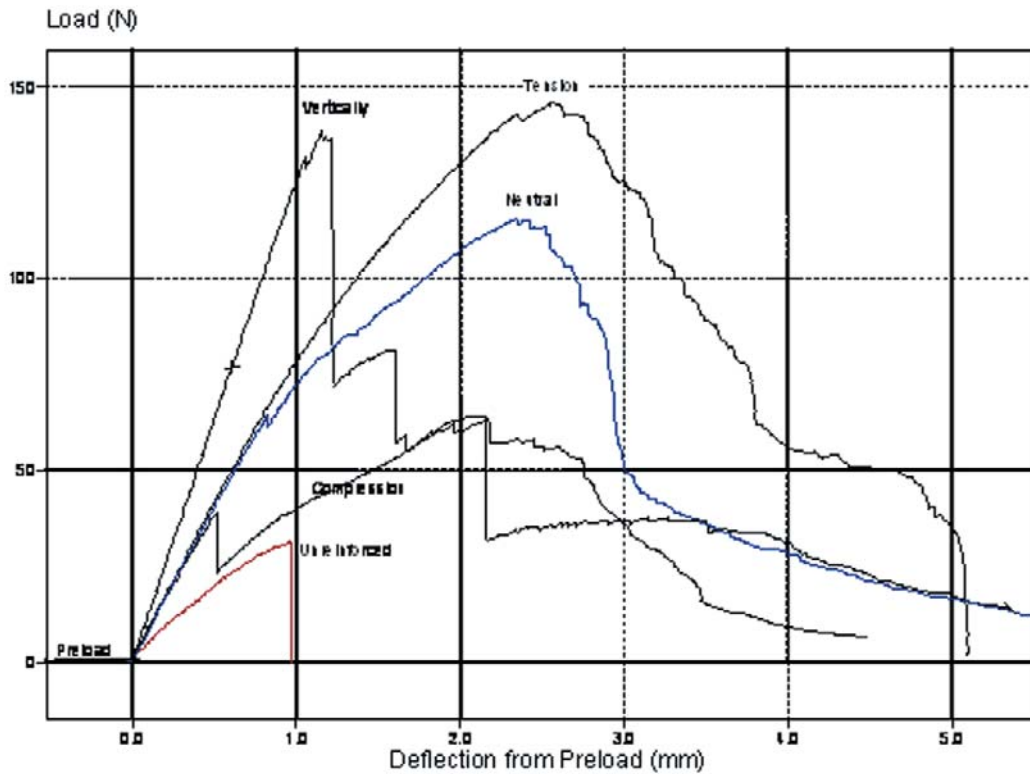


Figure 4. Typical load-deflection curves for specimens with the FRC layer positioned on tension or compression side, in neutral axis, or placed vertically along the wall of the test specimen.

Residual Monomer Content

HPLC analysis revealed total residual acrylate monomer content of the specimens polymerized in the Visio Beta light curing device was higher than those polymerized in the LicuLite light curing device. Specimens with 24 vol% glass fibers released 52% less residual monomers than specimens without glass fibers (Table 8).

Discussion

The position of the FRC layer had an effect on the flexural strength of the test specimen. The highest flexural strength was achieved when the FRC layer was located at the tension side of the test specimens. The particulate filler composite is the weaker phase of the test specimen. When it is located on the tension side, the fracture can easily initiate. The FRC structure benefits most when the tensile stresses can be transferred to the reinforcing fibers. The veneering particulate filler composite is strong in compression stress and, therefore, the FRC structure requires less reinforcement fibers on the compression side.¹⁷ When fiber reinforcement was placed in the middle of the test specimen, i.e., in the neutral axis, the highest possible flexural strength was not achieved. This is because the highest stress develops at the edges of the test specimen during loading. However, the maximum shear stress exists in the middle of the test specimen. It must also be noticed if a specimen with phases of different material properties is in flexion, the position of the neutral axis is located toward the phase with higher modulus of elasticity.¹⁸

If the FRC layer was positioned on the compression side, and the particulate filler composite resin layer was located on the tension side, the fractures proceeded across the specimens until it reached the FRC layer. The position of the FRC layer did not have a significant effect on the flexural modulus probably because the stiffness of the E-glass FRC was considerably higher than that of the polymer matrix. Thus, the stiffness of the test specimen was mainly influenced by the modulus of elasticity of the E-glass FRC layer.¹⁷ Usually, it is preferable to place the FRC laminates symmetrically relating to the FRC framework, to prevent polymerization shrinkage effect, thermal stresses, and possible deformation during polymerization.⁶ Nevertheless, it is often very

Table 8. Total residual monomer content of test specimens as analyzed with HPLC.

Materials/light curing unit	Total residual monomer content
Sinfony/VisioBeta device	1.52%
Sinfony/LicuLite device	0.28%
Sinfony+FRC/VisioBeta device	0.93%
Sinfony+FRC/LicuLite device	0.12%

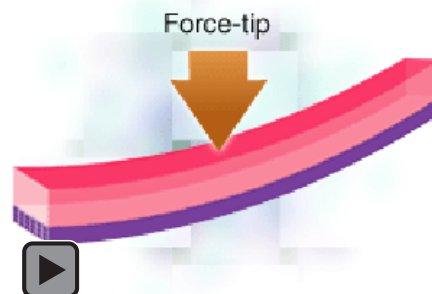
difficult to design the FRC framework to form an optimal design because of the abutment location and occlusal parameters.

If the FRC layer was placed vertically in the test specimen, the geometry of the FRC layer resulted in a higher flexural modulus of the specimen. From the following formula it can be seen the effect of the h (height) on the stiffness is in the power of three, whereas the effect of b (width) to the stiffness is linear.

$$E_t = S l^3 / (4b_i h_i^3) \Rightarrow S = 4 E_t b_i h_i^3 / l^3$$

(l is the span length, b_i is the width of the FRC layer in the specimen, h_i is the thickness of the test specimen, and S is the stiffness ($S=F/d$)).

The high stiffness of the FRC framework might be beneficial from the clinical perspective in reducing the risk of debonding of the framework from the abutments. Also, the risk of loosening veneering composite resin from the FRC framework can be diminished by the stiffness of the framework.



Visio Beta Vario is a light-curing unit, where the composite resins are polymerized in a vacuum chamber under light exposure of 400-500 nm wavelengths. The vacuum (< 0.04 mbar, according to the product information literature) minimizes the formation of oxygen inhibition layer during polymerization. The temperature in the Visio Beta vacuum chamber during the polymerization period raised up to 60°C. The LicuLite light-curing device had a 400 W high performance halogen lamp emitting a wavelength range of 350-550 nm (according to the product information literature). LicuLite polymerizes resins under normal atmospheric pressure while the temperature of the polymerizing chamber reaches 85 °C. The flexural properties were higher with the test specimens polymerized with the LicuLite light-curing device most likely due to the higher polymerization temperature and light intensity. Other investigators have previously reported similar results with composite resin filling materials.^{13,19} Generally, a higher polymerization temperature increases monomer movement resulting in a higher degree of conversion of C=C bonds of the functional groups of the monomers and lowers the residual monomer content.

The lower flexural modulus of Sinfony Dentine particulate filler composite resin polymerized in a Visio Beta device can be explained by the higher residual monomer content. This may have been caused by the lower polymerization temperature or by lower light intensity. Residual monomers act as plasticizers of polymer matrix, thus, reducing the flexural modulus. When the results of the residual monomer content of the test specimens are interpreted, it should be emphasized the total quantity of leachable acrylate monomers within the analytic region of the calibration monomers was determined. The HPLC analysis of the present study could, therefore, be considered only indicative to show the difference between the influence of two light curing devices, and between residual monomer content of unreinforced and the FRC reinforced test specimens.

It might be beneficial the polymerization of the FRC framework for the fixed partial denture is carried out with a light-curing device with high polymerization temperature, and the veneering particulate filler composite is polymerized in a vacuum at a lower temperature. Furthermore,

in order to optimize bonding of the veneering composite resin to the surface of FRC framework, it might be beneficial to polymerize the FRC framework in air to obtain an oxygen inhibition layer on the surface of FRC framework.²⁰ The oxygen inhibition layer allows veneering composite resin to bond to the frame with radical polymerization. Another approach to resolve the problem of adhering new resin to the FRC substrate is based on the interpenetrating polymer network (IPN) formation.^{21,22}



Conclusions

Within the limitations of the study, the following conclusions were made:

1. In order to optimize flexural strength of a FRC construction with low fiber volume fraction, the fibers should be placed on the tension side of the specimen.
2. In order to optimize stiffness of the construction, the fiber rich layer should be spread vertically.
3. Light polymerization of the polymer matrix of FRC at an elevated temperature decreased the quantity of leachable residual monomers and increased the flexural strength and modulus of elasticity. These findings can give general guidance to dental practitioners or technicians for designing FRC bridges.

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