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Short Fiber Reinforced Composite: The Effect of Fiber Length and Volume Fraction

Sufyan K. Garoushi, BDS; Lippo V. J. Lassila, DDS, MSc;
Pekka K. Vallittu, CDT, DDS, PhD, Prof

Abstract

Aim: The aim of this study was to determine the effect of short fiber volume fraction and fiber length on some mechanical properties of short fiber-reinforced composite (FRC).

Methods and Materials: Test specimens (2 x 2 x 25 mm^3) and (9.5 x 5.5 x 3 mm^3) were made from short random FRC and prepared with different fiber volumes (0%-22%) and fiber lengths (1-6 mm). Control specimens did not contain fiber reinforcement. The test specimens (n=6) were either dry stored or thermocycled in water (x10.000, 5 – 55°C) before loading (three-point bending test) according to ISO 10477 or statically loaded with a steel ball (Ø 3.0 mm) with a speed of 1.0 mm/min until fracture. A universal testing machine was used to determine the flexural properties and the load-bearing capacity. Data were analyzed using analysis of variance (ANOVA) (p=0.05) and a linear regression model.

Results: The highest flexural strength and fracture load values were registered for specimens with 22 vol% of fibers (330 MPa and 2308 N) and with 5 mm fiber length (281 MPa and 2222 N) in dry conditions. Mechanical properties of all test specimens decreased after thermocycling. ANOVA analysis revealed all factors were affected significantly on the mechanical properties (p<0.001).

Conclusions: By increasing the volume fraction and length of short fibers up to 5 mm, which was the optimum length, the mechanical properties of short FRC were improved.

Keywords: Fiber-reinforced composite, FRC, flexural strength, load-bearing capacity, critical length

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Introduction

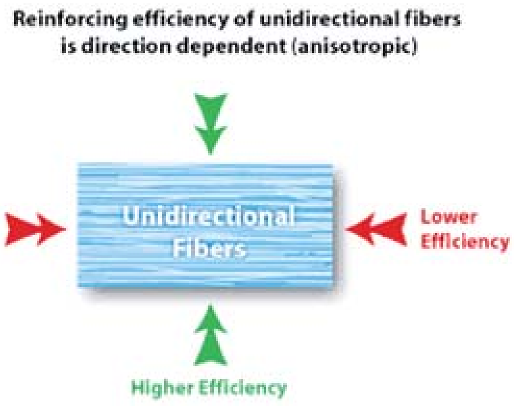
Attempts have been made to reinforce dental polymers with several types of fibers for various dental treatment modalities during the past 30 years. Polyethylene fibers,[1] carbon/graphite fibers,[2-4] or glass fibers have been tested.[5-8] At the moment, glass fibers are the fibers of choice to reinforce dental polymers because of their documented reinforcing efficiency and favorable esthetic qualities.[9-11]

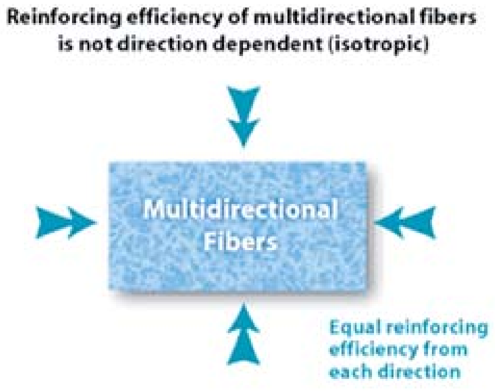
The effectiveness of fiber reinforcement is dependent on many variables including the resins used, the quantity of fibers in the resin matrix,[12,13] length of fibers,[13] form of fibers,[14-16] direction of fibers,[17] adhesion of fibers to the polymer matrix,[18] and the impregnation of fibers with the resin.[19] The location of the fiber-rich phase in a dental device also affects the strength of the device.[16,20,21]

It is known when the directional orientation of the fiber is with their long axis and perpendicular to an applied force the result is a high reinforcing effect.[22,23] However, forces that are perpendicular to the long axis of the fibers produce matrix-dominated failures and, consequently, result in low reinforcing efficiency. Multi-directional fiber reinforcement has been employed to minimize the anisotropic behavior of unidirectional fiber reinforcement. On the other hand, multidirectional fiber reinforcement is accompanied by a decrease in strength when compared with unidirectional fibers as described by Krenchel.[24]

Composites that have randomly-oriented fibers are isotropic in their mechanical and physical properties. In other words, the strength of the fiber-reinforced composite (FRC) is not related to the direction of the stress.[25] It can be supposed the two fiber systems could be used in different clinical applications, where different properties are required.[5] Numerous studies have demonstrated the relationship between the quantity of fibers in the polymer matrix and the flexural and impact strength of fiber reinforced construction.[11,26,27]

It has been described by increasing the fiber content the flexural strength increases linearly according to the law of mixtures.[28] It is preferable to define the fiber quantity in the polymer matrix in volume percentage rather than weight percentage.[11] In short fiber composites the length and adhesion of fibers should provide load transfer from polymer matrix to the fibers. The shortest effective fiber length is called the critical fiber length. To the author's knowledge there are no studies demonstrating the influence of short glass fiber length on the flexural properties and load bearing capacity of FRC construction. It is





important to know the critical length of short fibers in polymer matrices are used in dentistry for the achievement of maximum reinforcing effect. The aim of this study was to determine the effect of different fiber lengths and volume fractions on the mechanical properties of short FRC.

Methods and Materials

A total of 264 test specimens were fabricated from short random E-glass fibers with various lengths (1 mm-6 mm) that had been pre-impregnated with porous PMMA (poly methyl methacrylate) (StickTeck Ltd, Turku, Finland, Lot no 1010321-R-0058). The fibers were further impregnated with light-polymerizable resin (BisGMA-TEGDMA) (60% bisphenol-A-glycidyl dimethacrylate-40% triethylen glycoldimethacrylate). Short random FRC test specimens were fabricated either to form bar-shaped specimens (2 x 2 x 25 mm^3) (Figure 1) or cubic specimens (9.5 x 5.5 x 3 mm^3) (Figure 2). Bar-shaped specimens were made in a half-split stainless steel mold between transparent Mylar sheets and cubic specimens in open silicon mold covered by Mylar. Study groups used in this study are described in Table 1.

In the first part of this study, the effect of fiber volume fractions on mechanical properties was determined using a fiber length of 5 mm. In the second part of the study, the effect of different fiber lengths with one fiber volume fraction (15 vol%) was evaluated. Control specimens were prepared from light-polymerizable resin without fiber reinforcement.

Polymerization of the composite was accomplished using a hand light-curing unit (Optilux-501, Kerr, CT, USA) for 40s from both sides of the metal mold and incrementally from the top of the silicon mold. The wavelength of the light was between 380 and 520 nm with maximal intensity at 470 nm and a light intensity of 800 mW/cm^2.

The specimens from each group (n=6) were either stored dry or additionally thermocycled in water for 10,000 cycles between (5°C - 55°C) with dwell time of 30s and then stored in distilled water for 24h before testing.

Three-point bending tests were conducted according to the ISO 10477 (test span: 20 mm, cross-head speed: 1.0 mm/min, indenter: 2 mm diameter). All specimens were loaded in a material testing machine (model LRX, Lloyd Instrument Ltd, Fareham, England), and the load-deflection curves were recorded using PC-computer software (Nexygen, Lloyd Instruments Ltd, Fareham, England). Static compressive fracture tests for determining the load-bearing capacity were performed using a universal testing machine, and the results were recorded using the same software as was used with the three-point bending test. Specimens were loaded with a steel ball (Ø 3.0 mm) under a static load until fracture.

Data of the fracture load values and flexural properties were statistically analyzed using SPSS (SPSS Inc, Chicago, IL, USA) using an analysis of variance (ANOVA). Fiber length, fiber

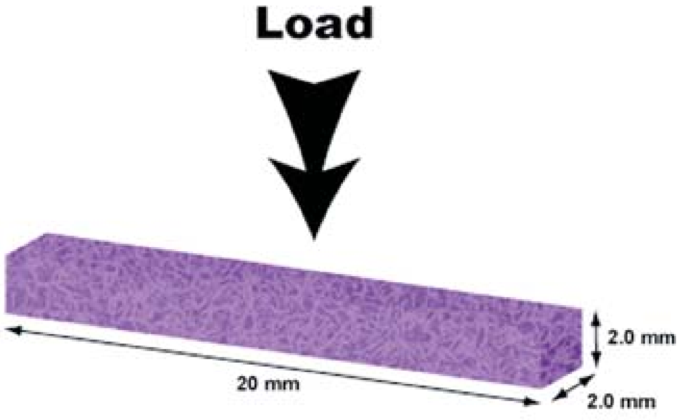


Figure 1. Schematic drawing of bar-shaped test specimen and the three-point bending test setup.

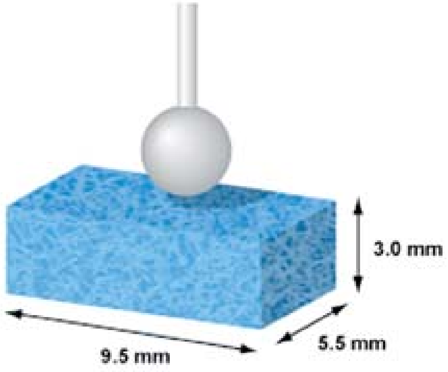
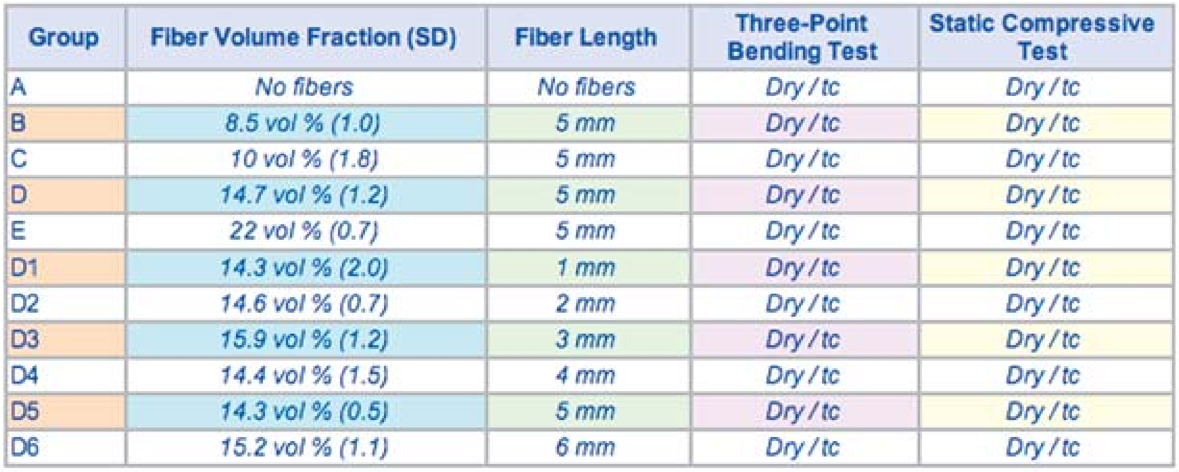


Figure 2. Schematic drawing of the test specimen and the compressive strength test setup.

Table 1. Test groups used in the study (n=6 per group) and measured fiber volume fractions with standard deviation (SD).



Group	Fiber Volume Fraction (SD)	Fiber Length	Three-Point Bending Test	Static Compressive Test
A	No fibers	No fibers	Dry / tc	Dry / tc
B	8.5 vol % (1.0)	5 mm	Dry / tc	Dry / tc
C	10 vol % (1.8)	5 mm	Dry / tc	Dry / tc
D	14.7 vol % (1.2)	5 mm	Dry / tc	Dry / tc
E	22 vol % (0.7)	5 mm	Dry / tc	Dry / tc
D1	14.3 vol % (2.0)	1 mm	Dry / tc	Dry / tc
D2	14.6 vol % (0.7)	2 mm	Dry / tc	Dry / tc
D3	15.9 vol % (1.2)	3 mm	Dry / tc	Dry / tc
D4	14.4 vol % (1.5)	4 mm	Dry / tc	Dry / tc
D5	14.3 vol % (0.5)	5 mm	Dry / tc	Dry / tc
D6	15.2 vol % (1.1)	6 mm	Dry / tc	Dry / tc

Note: tc refers to thermocycling for 10,000 times.

volume fraction, and storage conditions were used as independent variables. A linear regression analysis was used to determine the correlation between these independent variablesand flexural strength and load-bearing capacity of the test specimens. Flexural strength ($?_f$) and flexural modulus (E_f) were calculated from the following formula.[29]

$$?_f = 3F_m l / 2bh^2 \qquad E_f = Sl^3 / 4bh^3$$

F_m 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 test specimens.
h is the thickness of test specimens.
S is the stiffness (N/m) S=F/d.
d is the deflection corresponding to load F at a point in the straight-line portion of the trace.

The quantity of fibers in the test specimens was determined by combustion of the polymer matrix of the test specimens for 1h at 700°C in a furnace (Jelrus Radiance MSL, New York, USA). Before and after combustion, the weight of the specimens was measured with a balance (Mettler Toledo, GmbH, Switzerland) with an accuracy of 0.1 mg. The fiber content as percentage by volume (V_f) (vol%) was calculated with the following formula:[30]

$$V_f = [(W_f / r_f)/(W_f/ r_f + W_r/ r_r)] \times 100(\%)$$

Where W_f is the weight proportion of E-glass, r_f(=2.54g/cm^3) is the density of E-glass, W_r is the weight proportion of resin, and r_r (=1.238g/cm^3) is the density of resin.[25]

Results

The mean flexural strength, modulus, and load-bearing capacity for each of the groups are shown in Tables 2 and 3. ANOVA revealed fiber volume fraction had a significant effect (p<0.001) on the flexural strength and on the load-bearing capacity. Fiber length revealed also a significant effect (p<0.001) on the flexural strength, whereas no systematic effect of the fiber length on the load-bearing capacity was found (p>0.001) (Figure 6).

The regression analysis demonstrated significant correlation between fiber volume fractions and fiber lengths with flexural strength and load bearing capacity (Figures 3, 4, and 5).

Thermocycling decreased the flexural strength and the load-bearing capacity for all of the specimens (p<0.001). Some interaction existed between the factors (p<0.001). The specimens with 5 mm fiber length showed the highest flexural strength and load-bearing capacity. On the other hand, specimens with 6 mm of fiber length revealed lower load-bearing capacity than obtained with shorter fibers. The determined volume fractions of glass fibers of the specimens are shown in Table 1.

Discussion

This study demonstrated the effect of fiber volume fraction and fiber length on mechanical properties of short random FRC. Fiber reinforcement is

Table 2. Mean results with standard deviation (SD) of three-point bending test and static compressive test of specimens with different fiber volume fractions.

Group	Flexural Strength (MPa)		Flexural Modulus (GPa)		Compressive Fracture Load (N)	
	dry	tc	dry	tc	dry	tc
A 0 vol%	53(20)^{a}	27(5)^{a}	4.7(0.2)^{ab}	3.5(1.0)^{a}	971(201)^{a}	735(136)^{a}
B 8.5 vol%	192(24)^{b}	168(10)^{b}	9.2(2.7)^{bc}	7.2(0.8)^{ab}	1529(250)^{bcde}	1260(142)^{abc}
C 10 vol%	226(38)^{bc}	183(33)^{b}	9.4(2.0)^{bc}	9.1(1.1)^{bc}	1725(164)^{cde}	1406(310)^{bcd}
D 14.7 vol%	264(50)^{bc}	186(48)^{b}	9.3(2.2)^{bc}	8.9(1.6)^{bc}	2201(130)^{f}	1664(133)^{cde}
E 22 vol%	330(21)^{c}	242(31)^{bc}	11.7(2.1)^{c}	11.1(2.3)^{c}	2308(175)^{f}	1951(141)^{def}

Note: Superscript letters indicate data sets that are not statistically different ($p > 0.05$) and tc refers to thermocycling for 10,000 times.

Table 3. Mean results with standard deviation (SD) of three-point bending test and static compressive test of specimens with different fiber lengths.

Group	*Flexural Strength (MPa)*		*Flexural Modulus (GPa)*		*Compressive Fracture Load (N)*	
	dry	*tc*	*dry*	*tc*	*dry*	*tc*
D1 1 mm	79(18)^{ab}	45(10)^{a}	6.8(2.1)^{ab}	6.3(3.2)^{a}	1651(227)^{abc}	1278(190)^{a}
D2 2 mm	116(11)^{abcd}	79(28)^{ab}	6.9(1.6)^{ab}	11.0(5.2)^{abc}	2188(185)^{d}	1793(114)^{abcd}
D3 3 mm	146(34)^{bcd}	86(81)^{abc}	8.1(1.7)^{abc}	11.2(1.4)^{abc}	2163(245)^{cd}	2013(246)^{bcd}
D4 4 mm	171(32)^{cde}	116(22)^{abcd}	8.6(1.1)^{abc}	11.2(6.0)^{abc}	2227(372)^{d}	2069(175)^{bcd}
D5 5 mm	281(40)^{e}	184(43)^{cde}	12.3(1.9)^{c}	10.2(1.3)^{abc}	2222(312)^{d}	2059(344)^{bcd}
D6 6 mm	206(43)^{de}	196(50)^{de}	12.1(0.9)^{bc}	11.4(4.0)^{abc}	1586(387)^{ab}	1323(287)^{a}

Superscript letters indicate data sets that are not statistically different ($p > 0.05$).
tc refers to thermocycling for 10,000 times.

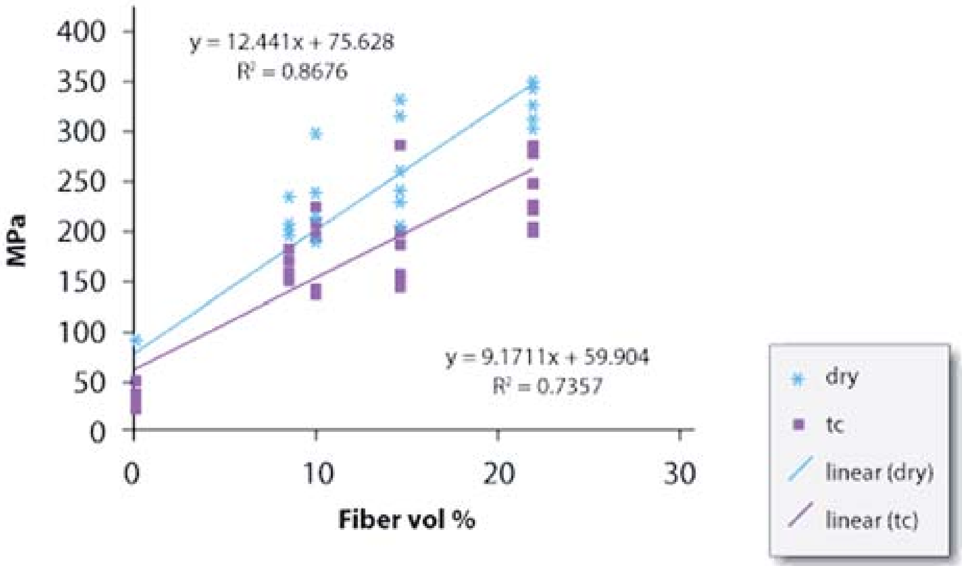


Figure 3. Linear regression (n=60) between different fiber volume fractions and flexural strength of test specimens. (tc refers to thermocycling for 10,000 times.)

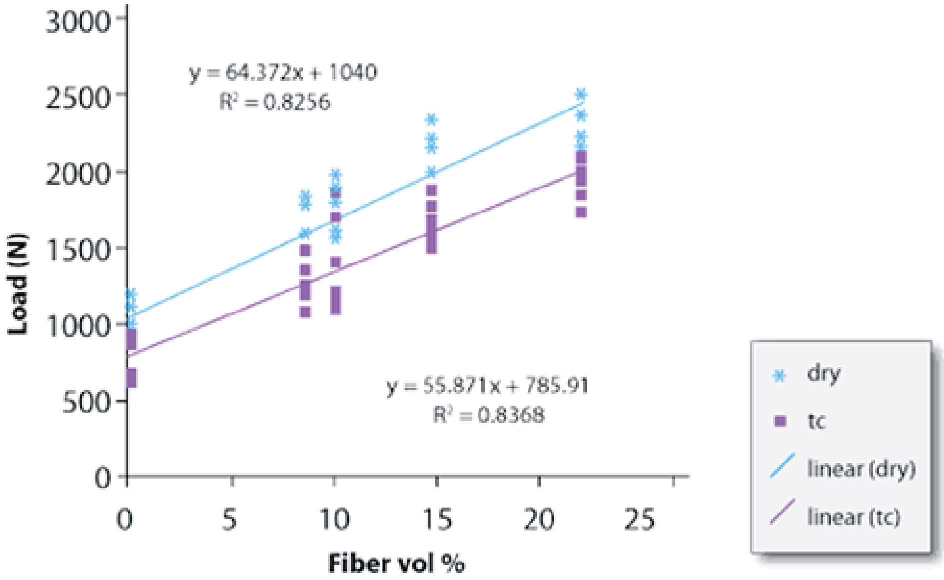


Figure 4. Linear regression (n=60) between different fiber volume fractions and compressive load of test specimens. (tc refers to thermocycling for 10,000 times.)

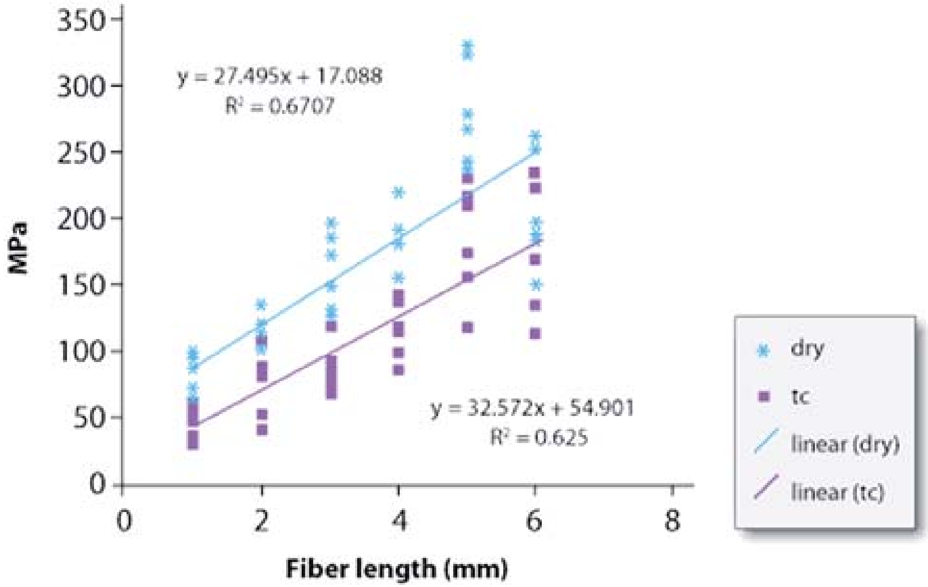


Figure 5. Linear regression (n=60) between different fiber lengths and flexural strength of test specimens. (tc refers to thermocycling for 10,000 times.)

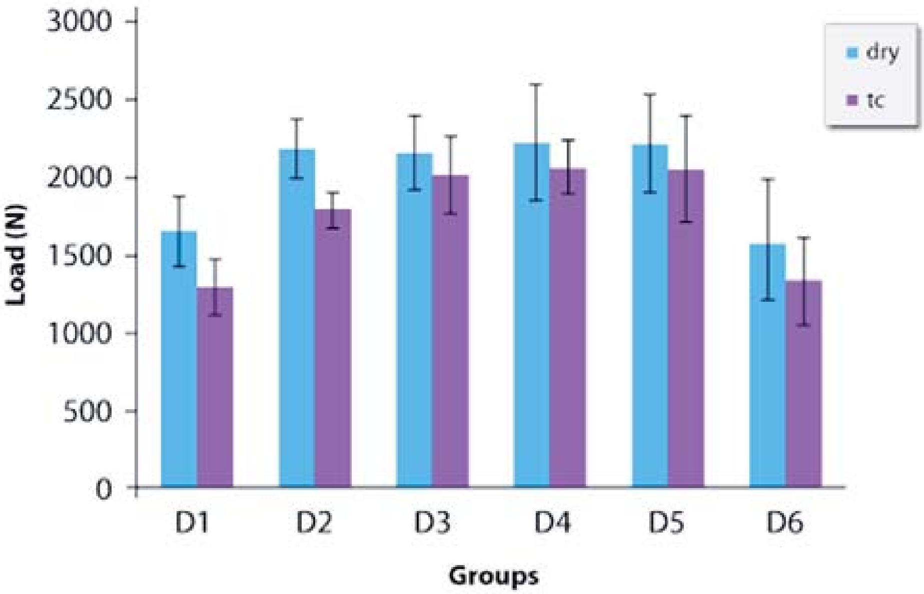


Figure 6. Compressive load of test specimens with different fiber lengths. Vertical lines represent standard deviations. (tc refers to thermocycling for 10,000 times.)

effective if the stress is transferred from the polymer matrix to the fibers.[20,31] This is achieved if the fibers have a length equal to or greater than the critical fiber length.[20,32] It has been estimated by using a fiber fragmentation test the critical fiber lengths of E-glass with bisGMA polymer matrix varies between 0.5 and 1.6 mm.[33] Critical length is a measure of minimum perfectly aligned fiber dimension required for maximum stress transfer within the polymer matrix.[32] Deteriorated or initially poor adhesion between the fibers and polymer matrix increase the critical fiber length.

In this case, the mechanical frictions of fibers to polymer matrix at the interface compensate for the poor adhesion.[34] The effect of fiber length of short random FRC has been demonstrated on flexural strength, flexural modulus, and impact strength of injection and compression-molded polymethyl methacrylate based denture base polymer. The critical fiber length in those studies was 6 mm.[34]

On the basis of the results of the present study, a 5 mm fiber length demonstrated significantly higher flexural strength and load-bearing capacity of test specimens than other fiber lengths. However, the effect of fiber length on the load bearing capacity was not significantly different between specimens containing fibers of 2 to 5 mm (Figure 6). The 6 mm fibers provided the lowest load bearing capacity and specimens could not resist the load, causing the specimens to split longitudinally to the fiber axis. This can be explained by the size of the mold (9.5 x 5.5 mm), which could have lead to orientation of fibers in one direction rather than a random orientation.

Thermocycling decreased the flexural strength and load-bearing capacity in all of the specimens and was likely due to water sorption of the polymer matrix which caused plasticization of the polymer. It has also been reported previously the interfacial adhesion of glass fibers to the polymer matrix through a silane coupling agent can be deteriorated by water storage.[30,35]

Conclusion

There have been numerous reports regarding the effect of fiber volume fraction on the mechanical properties of FRC. Based on the result of the present study, by increasing the fiber volume fraction improvement in mechanical properties of FRC was obtained. Within the limitations of this *in vitro* study, it was demonstrated chopped glass fibers of 5 mm length could be the optimum length for use with a resin system of BisGMA-TEGDMA along with currently used coupling agents for bonding fibers to polymer.

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About the Authors

Sufyan K. Garoushi, BDS

Dr. Garoushi is a graduate of Garyounis University in Benghazi, Libya, and he is currently a doctoral student in Prosthodontics and Biomaterials at the Institute of Dentistry, University of Turku, Finland. His current research is focused on fiber-reinforced composite in dentistry.

e-mail: sufgar@utu.fi

Lippo V. J. Lassila, DDS, MSc

Dr. Lassila graduated from Kuopio University and is now a doctoral student in Prosthodontics and Biomaterials at the Institute of Dentistry, University of Turku, Finland where he also serves as a laboratory engineer. His current research is focused on fiber-reinforced composites in dentistry.

e-mail: liplas@utu.fi

Pekka K. Vallittu, CDT, DDS, PhD, Prof

Dr. Vallittu is a Professor of Prosthodontics and Chairman of the Institute of Dentistry at the University of Turku, Finland. He received both his DDS and PhD from Kuopio University in 1994. He has served as a Visiting Scientist at the Scandinavian Institute of Dental Materials (NIOM) and has presented scientific lectures in 10 countries, including the USA, Canada, China, UK, Netherlands, and Japan. Dr. Vallittu has served as a referee to a number of international journals, edited 4 books, and published over 100 peer-reviewed articles. His primary research areas are fiber-reinforced composites in dentistry and in other biomedical applications.