Mechanical properties and radiopacity of flowable fiber-reinforced composite

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The aim was to evaluate the effect of different zirconia discontinuous fiber fractions on radiopacity and other selected properties of glass discontinuous fiber-reinforced flowable composite (Exp-SFRC). Exp-SFRC was prepared by mixing 30 wt% of resin-matrix and 45 wt% of particulate-fillers to 25 wt% of various weight-fractions of E-glass/zirconia discontinuous fiber-fillers (25:0, 20:5, 15:10, 10:15, 0:25 wt%). Flexural strength and fracture toughness were determined for each experimental material. Radiograph of each Exp-SFRC and aluminum step wedge were taken to determine the radiopacity. Degree of conversion and light-transmission were also measured. Scanning electron microscopy was used to evaluate the microstructure of the Exp-SFRC. Analysis of variance (ANOVA) revealed that fractions of E-glass/zirconia discontinuous fiber-fillers had significant effect ($p<0.05$) on radiopacity and other tested properties of the Exp-SFRCs. Replacing low fraction of E-glass fiber with zirconia fiber-fillers can increase the radiopacity of the fiber-reinforced composite without deteriorating the mechanical properties, although, degree of conversion was decreased.

Keywords: Fiber reinforced flowable composite, Fracture toughness, Radiopacity

INTRODUCTION

The use of light-cured resin composites for restoring cavities in stress-bearing posterior teeth has increased rapidly in recent years9-11). Especially the optimized handling characteristics cause an increased popularity of flowable conventional and flowable bulk-fill resin composites9). Beside the ability to bond to hard tooth tissues, mediated by adhesive systems, they feature the advantage of good esthetics and are less expensive compared with cast gold and ceramic inlays. However, insufficient material properties limited the success of resin composite restorations in high stress bearing areas6-8). Fracture within the body and margins of restorations and polymerization shrinkage have been cited as major problems regarding the failure of posterior composites9-11). The fracture related material properties, such as fracture resistance, deformation under occlusal load, and the marginal degradation of materials have usually been evaluated by the determination of the basic material parameters of flexural strength and fracture toughness9). Fracture toughness values are dependent on the physical properties and chemical composition of the individual component of restorative material. A material which has high fracture toughness has the ability to better resist crack initiation and propagation. Consequently, the property of fracture toughness and flexural strength become important criterions in a dental materials’ longevity9-11).

The requirement to strengthen resin composite has led to an ever increasing research effort into reinforcement techniques. Several former approaches dealt with incorporation of ceramic particle reinforced (random orientation), whisker (single or multi-layer) or fiber reinforced (continuous or discontinuous fibers in various orientations)9-11). Recently, a new experimental discontinuous fiber-reinforced flowable composite (SFRC) was introduced as a restorative resin composite9). It consists of a combination of a resin matrix, randomly orientated E-glass fiber and inorganic particulate fillers. This resin composite was reported to exhibit improved mechanical properties regarding flexural strength and fracture toughness and showed more favorable (repairable) type of failure behavior in comparison to particulate filler composite9). Ideally, the restorative resin composites should present, among other properties, sufficient radiopacity, to be distinguished from the adjacent anatomical structures, such as dentine and enamel12). Several factors may affect the radiopacity of resin composites, but the composition seems to be the most important factor. The higher the atomic number of the metal chemical elements present in the composition of the materials, higher is their capacity to absorb X-rays12). E-glass fiber has limited radiopacity because of the low concentration of radiopaque elements in the composition. Selected radiopacifying fillers, e.g., BaO, BaSO4, TiO2, SrO, or ZrO2 are incorporated into resin composite by physical blending. Unfortunately, incorporation of radiopaque particulate fillers mentioned above into resin composite has some adverse effects on the mechanical and chemical properties of composite materials even at a small concentration13,14). Based on this knowledge, radiopacifying fillers in form of discontinuous fibers instead of particles were used in this study. To the author’s knowledge, very little research exists in this field.

Specifically, the intent of this study was to replace a fraction of E-glass fiber with zirconia fiber that has same
length and diameter, in order to improve the radiopacity of an experimental fiber-reinforced flowable composite without deteriorating the mechanical performance. Thus, the effect of different zirconia discontinuous fiber fractions on radiopacity and selected properties (i.e., flexural strength, fracture toughness, degree of conversion and light transmission) of E-glass fiber-reinforced flowable composite was investigated.

MATERIALS AND METHODS

Production of flowable fiber-reinforced composite

Experimental flowable discontinuous fiber-reinforced composite (Exp-SFRC) was prepared by mixing 30 wt% of dimethacrylate based resin [ethoxylated bisphenol-A-dimethacrylate (bis-EMA), triethyleneglycol dimethacrylate (TEGDMA), diurethane dimethacrylate (UDMA)] and 45 wt% of particulate fillers to 25 wt% of various weight fractions of E-glass/zirconia discontinuous fiber fillers (25:0, 20:5, 15:10, 10:15, 0:25 wt% respectively, Table 1). The discontinuous E-glass and zirconia fibers [as-received silanized, 3-(Trimethoxysilyl)propyl methacrylate, MPS) having length and diameter, in order to improve the radiopacity of an experimental fiber-reinforced flowable composite without deteriorating the mechanical performance. Thus, the effect of different zirconia discontinuous fiber fractions on radiopacity and selected properties (i.e., flexural strength, fracture toughness, degree of conversion and light transmission) of E-glass fiber-reinforced flowable composite was investigated.

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Flexural strength measurement

Three-point bending test specimens (2×2×25 mm3) were made from each tested resin composite. Bar-shaped specimens were made in a half-split stainless steel mold between transparent Mylar sheets. Polymerization of the resin composite was done using a hand light-curing unit (Elipar S10, 3M ESPE, St. Paul, MN, USA) for 20 s in five separate overlapping portions from both sides of the metal mold. The wavelength of the light was between 430 and 480 nm and light intensity was 1,600 mW/cm². The specimens from each group (n=8) were stored dry at 37°C temperature for 48 h before testing. Three-point bending test was conducted according to the ISO 4049 (test span: 20 mm, cross-head speed: 1 mm/min, indenter: 2 mm diameter). All specimens were loaded in material testing machine (model LRX, Lloyd Instrument, Fareham, UK) and the load-deflection curves were recorded with PC-computer software (Nexxygen 4.0, Lloyd Instruments).

Flexural strength (σf) was calculated from the following formula:

$$\sigma_f = \frac{3F_{\text{max}}I}{(2bh^3)}$$

Where \(F_{\text{max}}\) is the applied load (N) at the highest point of load-deflection curve, I is the span length (20 mm), b is the width of test specimens and h is the thickness of test specimens.

Fracture toughness measurement

Single-edge-notched-beam specimens (2.5×5×25 mm3) according to adapted ISO 20795-2 standard method (ASTM 2005) were prepared to determine the fracture toughness \(K_{\text{IC}}\). Custom-made stainless steel split mold was used, which enabled specimen’s removal without force. Accurately designed slot was fabricated centrally in the mold extending until its mid-height, which enabled central location of the notch and optimization of the crack length (x) to be half of specimen’s height. The resin composite material was inserted into the mold placed over a Mylar-strip-covered glass slide in one increment. Before polymerization a sharp and centrally located crack was produced by inserting a straight edged steel blade into the prefabricated slot. Polymerization of the resin composite was carried out for 20 s in five separate overlapping portions. The upper side of the mold was covered with Mylar strip and glass slide from both sides of the blade, before being exposed to the polymerization light. Upon the removal from the mold, each specimen was polymerized also on the opposite side. The specimens from each group (n=6) were stored dry at 37°C temperature for 48 h before testing. The specimens were tested in three-point bending mode, in a universal material testing machine at a crosshead speed of 1.0 mm/min.

The fracture toughness was calculated using the Equation: 

$$K_{\text{max}}=\frac{f(x)PL}{(BW^{3/2})}\sqrt{10-3},$$

where: 

$$f(x)=3/2x^{1/2}[1.99-x(1-x)(2.15-3.93x+2.7x^2)]/2(1+2x)(1-x)^{3/2}.$$ 

0<x<1 with x=a/W. Here P is the maximum load in Newton (N), L is the span length (20 mm), B is the specimen thickness (mm), W is the specimen width (depth) in mm, x is a geometrical function dependent on a/W and a is the crack length in mm.

Table 1  Classification of the experimental discontinuous fiber reinforced resin composite test groups used in the study according to their fiber fillers content

| Experimental groups | E-glass fibers (wt%) | Zirconia fibers (wt%) | Resin matrix (wt%) | Particulate fillers (wt%) |
|---------------------|----------------------|----------------------|--------------------|--------------------------|
| Exp-SFRC1 (control) | 25                   | 0                    | 30                 | 45                       |
| Exp-SFRC2          | 20                   | 5                    | 30                 | 45                       |
| Exp-SFRC3          | 15                   | 10                   | 30                 | 45                       |
| Exp-SFRC4          | 10                   | 15                   | 30                 | 45                       |
| Exp-SFRC5          | 0                    | 25                   | 30                 | 45                       |
Radiopacity measurement

Five discs (2 mm) were prepared for every experimental resin composite, and irradiated with X-ray (63 kV, 8 mA, 0.08 s) by using Romex-program (Planmeca Oy, Helsinki, Finland) to get a digital radiograph. The object to focus distance was fixed at 30 cm and at 90° angle positions. The relative radiopacity was determined by comparison with the opacity exhibited by a standard aluminum step-wedge (1–8 mm) exposed on the same radiograph. A free image editing software Image J was used to measure the gray value of the sample and aluminum in the resulting image. The relative radiopacity (RXO) of disc was calculated by using the equation:

\[
RXO = \left[ \frac{(Gd - Gb)}{(Ga - Gb)} \right] \times 100\%
\]

where \(Gd\), \(Gb\), and \(Ga\) are the gray value of disc, background and aluminum at the same thickness, respectively.

Light transmission measurement

For each resin composite, 1 mm incremental thicknesses were considered (\(n=5\)). The specimens were prepared in cylindrical Teflon molds that are open at the top and the bottom sides and cured for 40 s by applying the curing unit with an irradiance of 1,600 mW/cm\(^2\) (Elipar S10, 3M ESPE) directly, perpendicular and centered on the specimen’s surface by using a mechanic arm. While the specimens were cured, a spectrometer (MARC Resin Calibrator, Blue Light Analytics, Halifax, Canada) measured in real time the transmitted irradiance at the bottom of the specimens. The MARC system contains a NIST-reference miniature spectrometer (USB4000, Ocean Optics, Dunedin, FL, USA) with a 3,648-element linear CCD array detector (TCD1304AP, Toshiba, Tokyo, Japan). The sensor is a CC3 cosine corrector (diameter 4 mm) designed to collect radiation (light) at around 180°, eliminating optical interface problem associated with the light collection sampling geometry. For data recording, irradiance at wavelengths of 360–540 nm was considered.

Degree of conversion measurement

Degree of conversion (DC%) during and after the photoinitiation of polymerization was monitored by Fourier transform infrared spectroscopy (FTIR) (Spectrum One, Perkin-Elmer, Beaconsfield Bucks, UK) with an attenuated total reflectance (ATR) accessory. Resin composites were analyzed in a mold that was 1 mm thick and 4.5 mm in diameter. First, the spectrum of the unpolymerized sample was placed in the mold and measured. Then the sample was irradiated through an upper glass slide for 40 s with a visible light-curing unit (Elipar S10, 3M ESPE) producing an average irradiance of 1,600 mW/cm\(^2\) (Marc Resin Calibrator, BlueLight Analytics). The sample was scanned for its FTIR spectrum after being irradiated. The DC was calculated from the aliphatic C=C peak at 1,636 cm\(^{-1}\) and normalized against the carbonyl C=O peak at 1,720 cm\(^{-1}\) according to the formula:

\[
DC = \frac{(AC = C/AC = O)_{0} - (AC = C/AC = O)_{t}}{(AC = C/AC = O)_{0}} \times 100\%
\]

where AC=C and AC=O were the absorbance peak area of methacrylate C=C at 1,636 cm\(^{-1}\) and carbonyl at 1,720 cm\(^{-1}\), respectively; \((AC = C/AC = O)_{0}\) and \((AC = C/AC = O)_{t}\) represented the normalized absorbency of the functional group at the radiation time of 0 and t, respectively; DC is the conversion of methacrylate C=C as a function of radiation time. For each resin composite, five trials were performed.

Microscopic analysis

Scanning electron microscopy (SEM, JSM 5500, JEOL, Tokyo, Japan) provided the characterization of the microstructure and fractographic surface examination of the Exp-SFRC materials. The specimens (\(n=3\)) were gold sputter coated before the SEM examination.

Statistical analysis

The data were statistically analyzed with SPSS version 23 (SPSS, IBM, Armonk, NY, USA) using analysis of variance (ANOVA) at the \(p<0.05\) significance level followed by a Tukey HSD post hoc test to determine the differences between the groups.

RESULTS

In general, the data showed that by increasing the zirconia fiber weight ratios, the tested properties decreased except the radiopacity increased (\(p<0.05\)). Exp-SFRC1 resin composite with plain E-glass fibers (control) had significant higher fracture toughness (2.8 MPa m\(^{1/2}\)), flexural strength (146.5 MPa) and DC% (63) than other experimental SFRC resin composites (Figs. 1, 2 and 3). On the other hand, Exp-SFRC5 resin composite with plain zirconia fibers presented the lowest fracture toughness (1.6 MPa m\(^{1/2}\)), flexural strength (110 MPa), and DC% (59) values among the materials tested (\(p>0.05\)). Radiopacity of Exp-SFRC5 resin composite (4.6 mm Al) was found double of Exp-SFRC1 resin composite (2 mm Al) (Fig. 4).

The irradiance of penetrating light values

![Fig. 1 Bar graph illustrating means fracture toughness (Kmax, MP am\(^{1/2}\)) and standard deviation (SD). Groups joined by a horizontal line are not significantly different (\(p>0.05\)).](image-url)
significant correlated also with zirconia fiber weight ratios of resin composites specimens ($p<0.05, R^2=0.828$): as zirconia fiber weight ratio increase, the irradiance value decreased for each material (Fig. 5).

SEM analysis of the tested single-edge-notched-beam specimens showed random orientation and protruded (pullout) fiber ends at fracture surfaces of Exp-SFRC resin composite matrices (Fig. 6). In addition, it presented the random orientation of E-glass and zirconia discontinuous fibers.

**DISCUSSION**

This study verified that the radiopacity of experimental fiber-reinforced flowable resin composites containing E-glass and zirconia discontinuous fiber fillers could be easily controlled and improved by adjusting the zirconia fiber fraction in the matrix. As stated in the literature, the required level of radiopacity for the resin composite is, however, not standardized yet and is still controversial$^{16,17}$. A higher radiopacity of the resin composite provides easier distinction of the filling$^{16}$. On the other hand, Tveit and Espelid$^{18}$ reported that in posterior teeth, very radiopaque materials such as amalgam did not provide the best condition for radiographic detection of caries and defects adjacent to restorations, compared with posterior resin composite. Espelid et al. reported that the most accurate radiographic diagnosis was obtained with the resin composite having radiopacity slightly greater than that of enamel$^{19}$. In this research, Exp-SFRC1 (control) resin composite reinforced by discontinuous E-glass fiber and particulate fillers had radiopacity similar to enamel (2 mm Al). All zirconia fiber containing Exp-SFRCs had higher radiopacity than control Exp-SFRC1, and radiopacity increased with increasing of zirconia fiber fraction. It was great to notice that radiopacity of Exp-SFRC2 (5 wt% zirconia fiber) have already achieved the radiopacity of 2.8 mm aluminum. Zirconia is a high electronic density element with high radiopacity$^{20}$. However, ideal radiopacifier should provide material with a sufficient radiopacity without impairing other physical properties. Therefore, properties like flexural strength, fracture toughness, degree of conversion, and light transmission of Exp-SFRC containing zirconia discontinuous fiber fillers were also investigated.

The DC% and light penetration are a very important parameters for resin composites. A high
Fig. 6 SEM photomicrographs with different magnifications of fracture surface of Exp-SFRC3 single-edge-notched-beam specimen (A) showing pull-out of fibers (B); Random orientation of fibers (C); zirconia fiber (white arrow); E-glass fiber (red arrow).

DC% is a prerequisite when good performance of the resin composite restoration is desired. The DC% and light penetration of photopolymerizable methacrylate-based resin composites depend on several factors, like monomer formulation, photo-initiator formulation, characteristics of fillers and fillers content, and light-curing sources. In this study, the DC% and irradiance of penetrating light values significantly correlated with zirconia fiber fillers weight ratios of resin composites specimens: as zirconia fiber fillers weight ratio increase, the irradiance value decreased for each material (Figs. 3 and 4). This may be attributed to the light scattering at interface between zirconia fiber fillers and resin matrix, which may hinder the transmission of visible light into the deep zone of the resin composite. Zirconia discontinuous fiber fillers have high refractive index (≈2.1) comparable to that of resin matrix (≈1.5). Because of the refractive index of the zirconia fiber fillers is not matching well with that of the resin matrix, the light-cured resin composite appears opaque with decreased depth of cure and light penetration. Consequently, the results of mechanical properties showed that the resin composite group (Exp-SFRC1) without zirconia fiber fillers had the highest fracture toughness (2.8 MPa m$^{1/2}$) and flexural strength (146.5 MPa) in comparison to groups with different fractions of zirconia fiber fillers (Figs. 1 and 2). The mechanical properties of Exp-SFRC are significantly influenced by resin matrix composition and quantity of fibers. Decreasing the DC% and light penetration in resin matrix can decrease cross-linking density of cured resin composite, leading to decrease of mechanical properties. However, in this research, though DC% and light penetration decreased in the Exp-SFRC resin composites while zirconia fiber fillers ratio was increased, fracture toughness of zirconia fiber containing Exp-SFRC2 was still higher than common trade resin composite products (Fig. 1). This should be due to the higher reinforcing effect of discontinuous fiber fraction of Exp-SFRC2, which still can compensate the slight decrease in DC% and light penetration of the material. This is in accordance with Lassila et al. and Shouha et al. studies, which showed superior fracture toughness and flexural properties of discontinuous E-glass fiber reinforced flowable resin composite compared to conventional particulate filler resin composites. Reinforcing effect of the fiber fillers is based on stress transfer from polymer matrix to fibers but also behavior of individual fiber as a crack stopper. Previous study of Garoushi et al. showed how discontinuous fiber fillers could stop the crack propagation and provided increase in fracture resistance of resin composite. The fracture toughness of a material is a measure of how well that material hinders the progress of a crack or flaw under load. Fiber impedes the extension of a crack and develops interlocking bridges behind the progressing crack dissipating energy by fiber pullout resulting in graceful rather than catastrophic failure. This might be due to the random orientation of fibers in resin matrix and forming fiber network (Fig. 6), which seemed to have enhanced the ability of the material to resist the fracture propagation, as well as to reduce the stress intensity at the crack tip from which a crack propagates in an unstable manner. Aspect ratio, critical fiber length, fiber loading and fiber orientation are the main factors that could improve or impair the mechanical properties of Exp-SFRC. In this study, E-glass and zirconia
discontinuous fiber fillers have a similar length and diameter that give aspect ratio of more than 30.

Sufficient adhesion between fiber and matrix provides good load transfer between the two ingredients, which ensures that the load is transferred to the stronger fiber and this is how the fiber actually works as reinforcement. However, if the adhesion is not strong and if any voids appear between the fiber and the resin matrix, these voids may act as initial fracture sites in the matrix and facilitate the breakdown of the material. Wang et al. stated in their work that the interfacial bonding between the zirconia fibers and the resin matrix might not be optimal and therefore, there may possibly be weak adhesion as a result. From this point of view, the great increase in mechanical properties of discontinuous E-glass fiber-reinforced resin composites, could be also explained by the good chemical adhesion between the E-glass fibers and matrix. This seems to have enhanced the ability of the material to resist the fracture crack propagation by increasing the behavior of individual fiber as a crack stopper.

According to the data obtained in this work and despite the drawback, zirconia discontinuous fiber could be utilized as radiopacity adjustable fillers for light-cured fiber-reinforced resin composites. There are still unclear issues that need to be known regarding the use of zirconia discontinuous fiber fillers, like the type of interfacial bond between zirconia fibers and resin matrix and its effect on the light absorption and scattering. Therefore, further research is needed and an assessment of optimizing the formulation of this novel experimental flowable resin composite is now in progress.

CONCLUSION

Replacing low fraction of E-glass fiber with zirconia fiber fillers can increase the radiopacity of the fiber-reinforced flowable resin composite without deteriorating the mechanical properties, although, degree of conversion was decreased.

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CONFLICTS OF INTERESTS

Author PV consults for Stick Tech —Member of GC Group in R&D and training.

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