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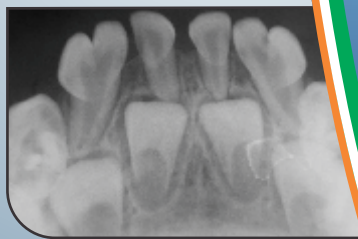
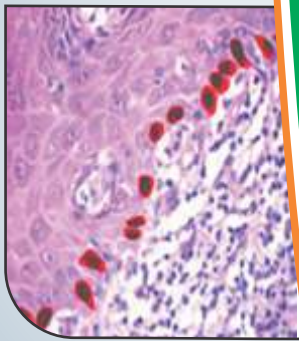


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Effect of fiber diameter on flexural properties of fiber-reinforced composites

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ABSTRACT

Background: Flexural strength (FS) is one of the most important properties of restorative dental materials which could be improved in fiber-reinforced composites (FRCs) by several methods including the incorporation of stronger reinforcing fibers.

Aim: This study evaluates the influence of the glass fiber diameter on the FS and elastic modulus of FRCs at the same weight percentage.

Materials and Methods: A mixture of 2,2-bis-[4-(methacryloxypropoxy)-phenyl]-propane and triethyleneglycol dimethacrylate (60/40 by weight) was prepared as the matrix phase in which 0.5 wt. % camphorquinone and 0.5 wt. % N-N'-dimethylaminoethyl methacrylate were dissolved as photoinitiator system. Glass fibers with three different diameters (14, 19, and 26 μm) were impregnated with the matrix resin using a soft brush. The FRCs were inserted into a $2 \times 2 \times 25 \text{ mm}^3$ mold and cured using a light curing unit with an intensity of ca. 600 mW/cm^2 . The FS of the FRCs was measured in a three-point bending method. The elastic modulus was determined from the slope of the initial linear part of stress-strain curve. The fracture surface of the composites was observed using scanning electron microscopy to study the fiber-matrix interface.

Statistical Analysis: The results were analyzed and compared using one-way ANOVA and Tukey's *post-hoc* test.

Results: Although the FS increased as the diameter of fibers increased up to 19 μm ($P < 0.05$), no significant difference was observed between the composites containing fibers with diameters of 19 and 26 μm .

Conclusion: The diameter of the fibers influences the mechanical properties of the FRCs.

Key words: Fiber diameter, fiber-reinforced composites, flexural modulus, flexural strength

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In recent years, materials with unusual blends of antithetic properties such as low density, high strength, rigidity, and wear resistance are more favorable in material science. Although these features are not accomplished by an individual material, composites, which are complex and multiphase materials, vastly present the characteristics of its phases leading to a more desirable combination of traits that could fulfill the aforementioned requirements.^[1,2] In dentistry, the term "resin composite" generally refers to a reinforced polymer used for restoring enamel and dentin.^[3]

Different methods have been used to improve the properties of these resins,^[4] among which the production of fiber-reinforced composites (FRCs), composed of reinforcing fibers surrounded by polymer matrix, has shoot up in last few years besides their growing application in many dental fields such as fixed partial dentures (FPD).^[5,6] Their popularity has an upsurge trend because of their aesthetic aspect (translucency of FRC frameworks) which is substantially superior to that of FPDs with a metal framework.^[7]

However, this new prosthesis has some blind spots, including the greater occurrence of debonding in long-span FPDs that could be explained by the higher tensile stress at the bonding interface as the result of transferring the occlusal loads.^[8] From this perspective, a stronger framework material would be beneficial.^[7]

The strength of the FRCs is dependent on different factors including: Impregnation of fibers within the resin matrix, fibers to the matrix adhesion phenomenon, and the quantity and orientation of fibers.^[9-12] Since the mechanical properties

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of FRCs are directly related to a well-organized fiber/matrix interface to guarantee a successful load transfer from one fiber to another through the matrix,^[13] decreasing this interface would be favorable because it would lead to decreasing the amount of weak bonding surface.

Theoretically, although smaller fibers produce higher surface area comparing to the same volume fraction of larger ones,^[14] it is believed that increasing the fibers diameter could be advantageous when the bonding interface of fibers and matrix is not a completely well adhesion.

This study has been carried out to evaluate whether the flexural strength (FS) and elastic modulus of the FRCs are influenced by the fiber diameter using three different fiber diameters at the same weight percentage.

MATERIALS AND METHODS

Materials

2,2-Bis-[4-(methacryloxypropoxy)-phenyl]-propane (Bis-GMA) and triethyleneglycol dimethacrylate (TEGDMA) were supplied by Röhm (Degussa group, Hanau, Germany). Camphorquinone (CQ) and N-N'-dimethyl aminoethyl methacrylate (DMAEMA) were obtained from Aldrich (Germany). E-glass fibers with different diameters (14, 19, and 26 μm) were obtained from Jushi Group Ltd. (Zhejiang, China).

Methods

Sample preparation

A mixture of Bis-GMA and TEGDMA (60/40 by weight) was prepared as the matrix phase in which 0.5 wt.% CQ and 0.5 wt.% DMAEMA were dissolved in the matrix as photoinitiator system. The glass fibers were impregnated with the resin using a soft brush. Care was taken to apply equal amount of resin to all groups of fibers. The impregnated fibers were then cut in 25 mm length to be inserted into the FS steel molds. The composites consisting of glass fibers with diameters of 14, 19, and 26 μm are, respectively, designated as GC14, GC19, and GC26. The fiber loading of all the groups was the same.

Flexural strength

FS of the FRCs was measured in a three-point bending method. The specimens with dimensions of $2 \times 2 \times 25$ mm were prepared in stainless-steel rectangular mold utilizing a light curing unit (Optilu \times 501; Kerr, Danbury, CT, USA) with an intensity of *ca.* 600 mW/cm². An overlapping regime was applied to irradiate the whole specimens on both sides (40 s for each irradiation). After 1 week storage in 37°C deionized water, the three-point bending test was performed using a universal testing machine (SMT-20; Santam, Tehran, Iran) at a cross-head speed of 1 mm/min. The FS in MPa was calculated as:

$$FS = \frac{3pL}{2bd^2},$$

where p is the load at fracture (N), L is the span length (20 mm), and b and d are, respectively, the width and thickness of the specimens in mm. The elastic modulus was also determined from the slope of the initial linear part of stress-strain curve.

Scanning electron microscopy

The fracture surface of the composites was observed using scanning electron microscopy (SEM; TESCAN, VEGAII, XMU, Brno, Czech Republic) to study the fiber-matrix interface. The samples were gold coated using a sputter coater before SEM observations.

Statistical method

The results were analyzed and compared using Kolmogorov-Smirnov, One-way ANOVA, and Tukey's *post-hoc* test. The significant level was considered as 0.05.

RESULTS

Diagram 1 shows the FS of three groups. Although the FS increases as the fibers diameter grows up ($P < 0.05$), there is no significant difference between the GC19 and the GC26 groups.

Diagram 2 presents the elastic modulus of the FRCs. The lowest and the highest elastic modulus are for the GC14 and the GC26 groups, respectively, whereas the GC19 and GC26 groups do not differ statistically.

Figure 1 illustrates SEM images of GC14, GC19, and GC26 groups. As can be seen, the fibers are not completely impregnated by resin matrix.

DISCUSSION

The results of this study revealed that the FS of FRCs increased with increasing fiber diameter except for the FRCs containing 26- μm fibers. Also, our findings demonstrated that the elastic modulus trend the same as the FS among three groups. Our FS outcomes are in agreement with Obukro *et al.* who investigated FRCs of 30Vol% fiber content, incorporating 7, 10, 13, 16, 20, 25, 30, and 45 μm diameter silanized E-glass fibers. They reported an escalating FS as the fibers diameter grows up, except for the FRC with 45 μm that was even lower than what is observed in 13- μm group. Similar to ours, in their groups the FS of 13 μm was significantly less than 20 and 25 μm diameter fibers, where as 25- μm and 20- μm groups were not statistically distinguishable.^[15]

Lassila *et al.* showed that the flexural properties of well-impregnated FRCs are significantly higher than poorly

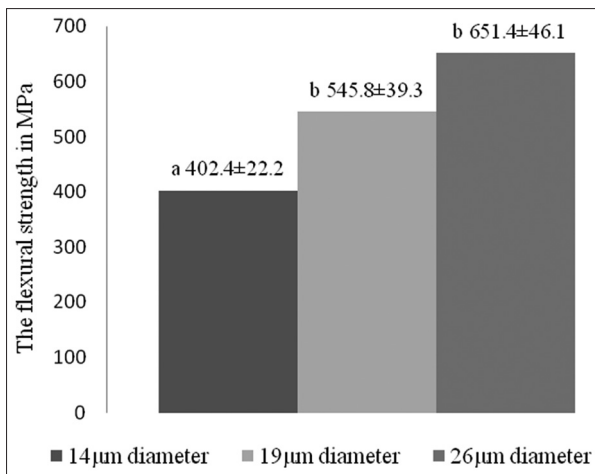


Diagram 1: Mean flexural strength of three groups (means followed by the same letter are not significantly differed by Tukey test)

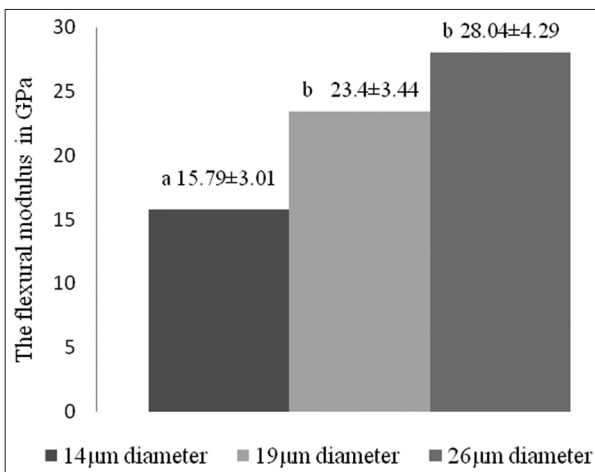


Diagram 2: Mean flexural modulus of three groups (means followed by the same letter are not significantly differed by Tukey test)

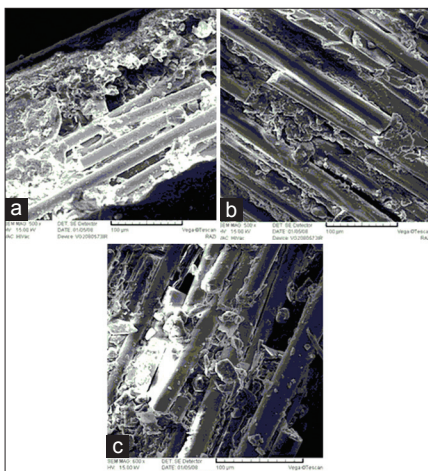


Figure 1: Scanning electron microscopy images of GC14 (a), GC19 (b), and GC26 (c) groups demonstrating the impregnation pattern of glass fibers by resin matrix. A poor bonding interface between the fibers and the matrix is observed

impregnated.^[16] Hence, transferring of the load from the matrix to fibers is strongly depends on the quality of the

interface. Not using any coupling agents for the surface treatment of the fibers in our study resulted in a poor adhesion between the fibers and matrix in the interface. Therefore, it would be expected that fibers with smaller diameter provide higher surface area and consequently more defects on the interface along with the fiber clustering which results in lower FS and modulus. Accordingly, it could be claimed that incorporating the larger fiber diameter produce less surface area comparing to the same volume fraction of smaller fibers^[14] and consequently, the FS would ascend.

This is in consistent with the SEM pictures, where fibers apparently are not well bonded to the matrix material, causing not well-organized stress transformation. Therefore, we recommend using coupling agents such as silanes in future studies.

In the case of elastic modulus, in contrast to the current research, Obukro *et al.* described no significant difference between the elastic modulus of groups containing fibers with different diameters.^[15]

Documenting by Callaghan *et al.*,^[17] when too many fibers are loaded in FRC, a cluster of fibers established with little matrix between them resulting in considerable interactions between fibers as well as poor bonding between fibers and matrix, leading to unfavorable stress distribution besides lower elastic modulus.

Flexural tests are routinely used for appropriate measurement of dental composite's strength in several studies.^[18-26] Direct measurement of the tensile strength is technically difficult while it does not reflect the flexural deformation in occlusal-loading situations, and it would be impossible to test a selected surface of the specimen in tension. The compressive strength is in a complex way related to a combination of tensile and shear failure modes. For precise measurement of the diametrial tensile strength, the material should not exhibit any plastic flow, which is impractical for most dental resin composites. Thus, the flexural test has been used in the current survey because it has been widely used to characterize the mechanical properties of dental restorative materials.^[18-30]

To date, various studies examined the flexural properties of commercially available FRCs. One of them investigating the flexural properties of commercially available light-cured FRCs^[31] show that the FSs of FibreKor (Preimpregnated Sglass FRC; Pentron Corporation, Wallingford, CT, USA) and Stick (Impregnated E-glass FRC; Stick Tech, Turku, Finland) are ranged from 367 to 405 MPa and from 430 to 460 MPa in that order; the elastic modulus of FibreKor and Stick were 23.8 GPa and 28.0 GPa, respectively. Another study^[32] demonstrated the FS and elastic modulus of FibreKor as 567 MPa and 26.5 GPa, respectively. In another study, the FSs of six commercially available FRCs are ranged

from 132 MPa to 764 MPa, whereby the highest was that of EverStick (Preimpregnated E-glass FRC; Stick-Tech, Turku, Finland).^[5]

Several studies report different values of the FS because the fabrication methods were in dissimilar conditions. On this ground, we could not compare our results with those found in published literature. Nonetheless, the FS of the 26- μm group in this study is on a par with the highest values found for the commercially available FRCs, and the elastic modulus values are also at an identical level.

Besides the mean diameter of fibers, several other factors such as type of matrix polymer and also its polymerization might influence the mechanical properties of FRCs.^[33] Usually, the modern FRC's matrix consists of synthetic resins or Bis-GMA polymers and hardly ever they contain PMMA (poly methyl methacrylate) chains of a high molecular weight (>220 kDa). Although fibers in FRCs are responsible for the high tensile strength, the matrix is the part which withstands compressive stresses.^[34] Nevertheless, the effect of the fibers on the matrix polymerization was not considered due to the primary interest in this research.

CONCLUSION

This investigation describes the FS and the elastic modulus of FRCs with different fiber diameters. Both of these variables increase by increasing the fiber diameter up to 19 μm , whereas there is no significant difference between the FS of the group containing 19 μm and the 26 μm diameter fibers.

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