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Original article

Effect of short glass fiber/filler particle proportion on flexural and diametral tensile strength of a novel fiber-reinforced composite

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ABSTRACT

Purpose: To evaluate the effect of glass fiber/filler particles proportion on flexural strength and diametral tensile strength of an experimental fiber-reinforced composite.

Methods: Four experimental groups (N = 10) were created using an experimental short fiber-reinforced composite, having as a factor under study the glass fiber (F) and filler particle (P) proportion: F22.5/P55 with 22.5 wt% of fiber and 55 wt% of filler particles; F25/P52.5 with 25 wt% of fiber and 52.5 wt% of filler particles; F27.5/P50 with 27.5 wt% of fiber and 50 wt% of filler particles; F30/P47.5 with 30 wt% of fiber and 47.5 wt% of filler particles. The experimental composite was made up by a methacrylate-based resin (50% Bis-GMA and 50% TEGDMA). Specimens were prepared for Flexural Strength (FS) (25 mm × 2 mm × 2 mm) and for Diametral Tensile Strength (DTS) (3 × 6 Ø mm) and tested at 0.5 mm/min in a universal testing machine.

Results: The results (in MPa) showed significance (different superscript letters mean statistical significant difference) for FS ($p < 0.009$) and DTS ($p < 0.001$) – FS results: F22.5/P55: 217.24 ± 20.64^{B}; F25/P52.5: 245.77 ± 26.80^{AB}; F27.5/P50: 246.88 ± 32.28^{AB}; F30/P47.5: 259.91 ± 26.01^{A}. DTS results: F22.5/P55: 21.82 ± 4.42^{B}; F25/P52.5: 22.00 ± 7.40^{B}; F27.5/P50: 18.63 ± 4.41^{B}; F30/P47.5: 31.05 ± 2.97^{A}. In SEM analysis, areas without fiber reinforcement demonstrated to be more prone to the presence of bubbles and crack development. The group F30/P47.5 showed areas with a great quantity of fibers without empty spaces for crack propagation.

Conclusion: Increasing fiber content results in higher flexural and diametral tensile strength of an experimental composite reinforced with glass fibers.

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1. Introduction

Over the past 30 years, several studies have been undertaken to strengthen dental polymers with various types of fibers [1–3]. Glass fibers have documented reinforcing efficiency, high tensile strength and favorable esthetic qualities [4–9]. The effectiveness of fiber reinforcement depends on many variables including the length [4,10], architecture [4], diameter [1,11], the quantity of fibers in the matrix resin [1,10], location and position of fibers [4,5], as well as its adhesion to the polymer matrix [5,12].

The fiber reinforcing mechanism has been explained by the principle that a relatively soft ductile polymer matrix is fully capable of transferring an applied load to the fibers via shear forces at the interface [13]. In order to fibers act as an effective reinforcement for polymers, the stress transfer from the polymer matrix to the fibers is essential, and it is achieved when fiber length is equal to or greater than the critical fiber length, which for a BISGMA based resin ranges from 0.5 to 1.6 mm [14]. Based on this knowledge, studies have used 3 mm-short glass fiber as reinforcement to obtain isotropic multidirectional reinforcement [1,6,15–18].

Some studies have demonstrated the relationship between the quantity of fibers in the polymer matrix and the flexural and impact strength of fiber-reinforced construction [6,19]. According to law of mixtures, by increasing quantity of fibers the flexural strength increases linearly [20]. Garoushi et al. [6] evaluated the influence of volume fraction and fiber length on the flexural strength of a methacrylate based resin, and concluded that a 22% volume fraction improves mechanical properties of a short fiber reinforced construction.

A short glass fiber composite (22.5 wt% of glass fiber, 22.5 wt% of methacrylate matrix and 55 wt% of filler particles) had already been used in dental materials to reinforce composites [15–17], as prosthesis infrastructure [21], onlay restorations [22,23], and endodontic posts [1,21,24]. The association of inorganic fillers in the short glass fiber composite contributes to reduce the polymerization shrinkage [25], improve the mechanical properties [26] and increase the viscosity of the material, facilitating its handling.

Due to a potential benefit of having high fiber loading into a reinforced composite it seems necessary to evaluate mechanical properties of short fiber reinforced composited with higher fiber proportion. It is expected that increasing the quantity of fibers, by varying the relation between glass fibers and filler particles content, higher values of selected mechanical properties could be reached. Thus, the aim of this study was to evaluate the effect of glass fiber/filler particles proportion on flexural strength and diametral tensile strength in an experimental short fiber-reinforced composite.

2. Materials and methods

2.1. Experimental groups

The materials used and their respective manufacturers are listed in Table 1. Four experimental groups (N = 10) were created using an experimental short fiber-reinforced composite, having as a factor under study the glass fiber/filler particle proportion, on four levels, according to Table 2.

2.2. Experimental composite manipulation

The experimental composite was made up by a mixture of methacrylate-based resins, filler particles and E-glass fibers (original dimensions (length/diameter): 3 mm/12 μm). The methacrylate-based resin mixture was manipulated using: 50% Bis-GMA and 50% TEGDMA in a photoinitiator system mode with 1 mol% of camphorquinone, 2 mol% of dimethylaminoethyl methacrylate (DMAEMA), and 0.1 mol% butylated hydroxytoluene (BHT). All components were weighted on an analytical balance (HR-200, A&D Company Limited, Japan) and mixed in a high speed-mixing machine (SpeedMixer, DAC, Germany, 3500 rpm), as described by Fonseca et al. [27].

After that, 0.7 μm silanated $BaAlSiO_2$ filler particles was manually mixed into the resin until a visual homogeneous mixture was reached. The manual incorporation of filler particles followed the proportions, in weight, previously established in Table 2.

E-glass fibers were submitted to a surface treatment with a silane coupling agent before incorporation in the resin [27]. Fibers were completely wetted by silane and stored at room temperature for 24 h. After that, they were manually incorporated into the resin, according to Table 2. Both filler particles and glass fiber were weighted on an analytical balance.

2.3. Strength tests

2.3.1. Diametral tensile strength (DTS) test

A condensation silicon impression material mold was obtained from a stainless steel pattern to produce standardized cylindrical specimens with dimensions of 3.0 mm (±0.1) of height × 6.0 mm (±0.1) of diameter, according to ADA specification No. 27 [28]. The experimental composite was inserted into the mold and overlaid with a polyester strip, and then light polymerized (LED Seasky; Tosi Foshan Medical Equipment Company, Japan) at 850 mW/cm^2 for 40 s at the top and bottom surface. The specimens (n = 10) were stored in distilled water at 37 °C for 24 h before testing. Specimens were positioned in universal testing machine (Instron 5965, Canton, USA). A crosshead speed of 0.5 mm/min was applied at the diametrical surface of samples until fracture, and the maximum load recorded in Newton (N). The diametral tensile strength of each sample was obtained in MPa, according to the following formula: $DTS = 2F/\pi dL$, where "F" is the maximum load achieved in the test (kg), "d" is the diameter of the specimen (6.0 mm) and "L" (3.0 mm) is the height. The specimens' thickness and height were measured with a digital caliper (Mitutoyo, Japan) before testing.

2.3.2. Flexural strength (FS) test

A condensation silicon impression material mold was made from a stainless steel pattern to produce standardized rectangular specimens with dimensions of 25 mm (±0.1) × 2 mm (±0.1) × 2 mm (±0.1), according to ISO 4049/2000 [29]. The experimental composite was inserted into the mold and overlaid with polyester strip and then light polymerized (LED Seasky; Tosi Foshan Medical Equipament Company, Japan) at 850 mW/cm^2 for 40 s at top surface. The