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Replacement of glass particles by multidirectional short glass fibers in experimental composites: Effects on degree of conversion, mechanical properties and polymerization shrinkage

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ABSTRACT

Objective. To test the null hypothesis that the replacement of a small fraction of glass particles with random short glass fibers does not affect degree of conversion (DC), flexural strength (FS), fracture toughness (FT) and post-gel polymerization shrinkage (PS) of experimental composites.

Methods. Four experimental photocurable composites containing 1 BisGMA:1 TEGDMA (by weight) and 60 vol% of fillers were prepared. The reinforcing phase was constituted by barium glass particles (2 μm) and 0%, 2.5%, 5.0% or 7.5% of silanated glass fibers (1.4 mm in length, 7–13 μm in diameter). DC ($n = 4$) was obtained using near-FTIR. FS ($n = 10$) was calculated via biaxial flexural test and FT ($n = 10$) used the "single edge notched beam" method. PS at 5 min ($n = 8$) was determined using the strain gage method. Data were analyzed by ANOVA/Tukey test (DC, FS, PS) or Kruskal–Wallis/Dunn's test (FT, alpha: 5% for both tests).

Results. DC was similar among groups ($p > 0.05$). Only the composite containing 5.0% of fibers presented lower FS than the control ($p < 0.001$). FT increased significantly between the control (1.3 ± 0.17 MPa $m^{0.5}$) and the composites containing either 5.0% (2.7 ± 0.6 MPa $m^{0.5}$) or 7.5% of fibers (2.8 ± 0.6 MPa $m^{0.5}$, $p < 0.001$). PS in relation to control was significantly reduced at 2.5% fibers (from $0.81 \pm 0.13\%$ to $0.57 \pm 0.13\%$) and further reduced between 5.0% and 7.5% (from $0.42 \pm 0.12\%$ to $0.23 \pm 0.07\%$, $p < 0.001$).

Significance. The replacement of a small fraction of filler particles with glass fibers significantly increased fracture toughness and reduced post-gel shrinkage of experimental composites.

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1. Introduction

In spite of the significant improvements in mechanical properties and wear over the last decades, bulk fracture remains one of the most prevalent causes of failure of composite restorations [1]. Thus, strategies to improve composite fracture toughness (FT) should be investigated. Glass fibers have been used to reinforce resin-based materials for many decades. The first reports, dating back to the 1960s, described the reinforcement of poly(methyl methacrylate) devices, such as full dentures, with long unidirectional fibers, short multidirectional fibers or glass fiber weaves [2]. In the 1990s, resin-impregnated unidirectional fiber bundles or weaves referred to as "fiber-reinforced composites" (FRC) became available for clinical use [3]. These materials have several uses, such as splints or frameworks for direct, semi-direct and indirect composite fixed prostheses [4], as well as for fiber posts [5]. Around the same time, silanized glass fibers with a relatively low aspect ratio (*i.e.* length: diameter) of 5 (25 μm in length and 5 μm in diameter) were tested in experimental dimethacrylate-based composites [6]. In that study, authors pointed out the difficulty of incorporating high fiber levels into the organic matrix due to incomplete wetting of the fibers (which favors air entrapment) and tendency to clustering.

Notwithstanding, light-cured restorative composites containing randomly oriented low aspect ratio glass fibers as part of their reinforcing phase became commercially available in 1990s. One of them (Restolux, Lee Pharmaceutical, South El Monte, CA, USA), no longer available, had 52 wt% of fibers (80–120 μm in lenght, aspect ratio: 5) and a total filler content of 85 wt% [7]. The second one (ALERT, Pentron, Orange, CA, USA) has 62 vol% of filler, including glass fibers 20–50 μm in length and 6 μm in diameter [8]. This material showed higher FT but lower flexural strength (FS) and modulus than microhybrid composites [9,10].

High aspect ratio fibers have also been tested as fillers in dental restoratives. In one study, the addition of 35–50 wt% random fibers (3 mm in length, 9 μm in diameter) to commercial particulate composites significantly increased their flexural properties [11]. Recently, a bulk-fill composite containing 54 vol% of fillers, part of them consisting of high aspect ratio E-glass fibers (1–2 mm in length) was introduced (EverX Posterior, GC Europe, Leuven, Belgium, formerly commercialized as Xenius base, Stick Tech Ltd., Turku, Finland). Indicated as a substructure material, it has shown a significantly higher FT compared to other bulk-fill composites [12]. Regarding its FS, however, the available reports are inconclusive when comparing the fiber-reinforced composite with other bulk-fill materials [13,14] and was not significantly different than that of the composite containing low-aspect ratio fibers [12]. A recent *in vitro* study did not find differences in strength of teeth restored with or without a layer of short fiber reinforced composite underneath a particulate composite [15].

The potential effects of fiber addition on composite post-gel polymerization shrinkage (PS) are another clinically relevant aspect. Fiber orientation has an important role on PS, as FRC with long continuous fibers were shown to shrink significantly more transversally than longitudinally to the fiber length [16]. Short fiber reinforced composites, on the other hand, should present an isotropic behavior unless fiber orientation is somehow influenced by specimen dimensions. For example, if specimen thickness is lower than fiber length, fibers will tend to be orientated perpendicularly to specimen thickness, resulting in a *quasi*-anisotropic behavior. EverX Posterior displayed lower PS (determined by the strain gage method) than particulated composites with higher overall filler content [12]. However, in a study using the "bonded-disk" method an experimental composite containing 22.5 wt% of 3 mm E-glass fibers and 55 wt% of silanized particles showed higher PS compared to a commercial composite containing 87 wt% of particles [17].

Studies systematically evaluating the effect of fiber content on dimethacrylate-based composite properties are scarce. Specifically regarding high aspect ratio fibers, it has been demonstrated that strength and modulus of dimethacrylate resins increased linearly with fiber content [18]. To our knowledge, however, there are no reports on the actual influence of replacing particulated fillers with randomly oriented fibers on composite mechanical properties or shrinkage. Therefore, the aim of the present study was to test the null hypothesis that FS, FT and PS of experimental composites are not affected by the replacement of silanized glass particles with high aspect ratio glass fibers.

2. Methods

2.1. *Composite preparation*

S-2 glass fibers, 7–13 μm in diameter (Advanced Glassfiber Yarns, Aikin, SC, USA) were cut into 1.4-mm long segments (standard deviation: 0.5 mm, Fig. 1) and silanized using 3-methacryloxypropyl-trimethoxysilane (3-MPTS). The silane content on the fibers was determined by thermogravimetric analysis as being 6%. Five experimental composites were formulated, all with a total filler content of 60 vol%, divided between fibers (0%, 2.5%, 5% or 7.5%) and silanized barium glass particles (2 μm). Maximum fiber percentage was limited to 7.5 vol% because at 10 vol% the composite mass had low cohesion, and it became impossible to produce specimens

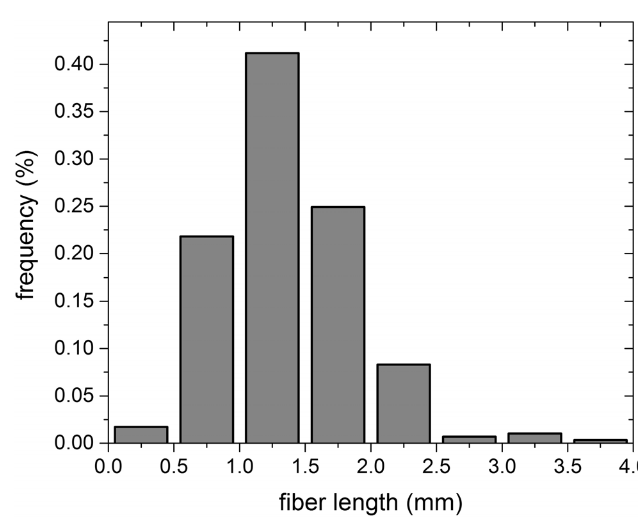

Fig. 1 – Histogram showing fiber length distribution.