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Effect of nanofiller fractions and temperature on polymerization shrinkage on glass fiber reinforced filling material

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ABSTRACT

Objectives. The aim was to evaluate the effect of different nanofiller fractions and temperature on polymerization shrinkage strain and degree of monomer conversion of short glass fibers reinforced semi-interpenetrating polymer network (semi-IPN)-polymer matrix composite resin.

Methods. Experimental composite resin was prepared by mixing 22.5 wt% of short E-glass fibers (3 mm in length) to the 22.5 wt% of resin matrix with various weight fractions of nanofillers (0, 10, 20, 30, 40, 50 wt%) and then 55 wt% of silane treated silica filler were added gradually using high speed mixing machine. Another study group contained composite resin prepared by mixing 22.5 wt% of resin matrix (without nanofillers) to 77.5 wt% of filler particles (without fiber fillers). As control material, commercial particulate filler composite resin was used. The shrinkage strain of the specimens was measured using the bonded-disk technique at 26 and 37 °C with respect to time. Degree of conversion of the experimental composites containing different nanofiller fractions was measured using FTIR spectroscopy. **Results.** ANOVA revealed that fraction of nanofillers and polymerization temperature had significant effect ($p < 0.05$) on the shrinkage strain and degree of conversion of the composite resin. Shrinkage strain correlated with nanofiller fraction and polymerization temperature ($r^2 = 0.96$ and 0.95).

Significance. The use of high nanofiller fraction with short fiber fillers and IPN-polymer matrix yielded improved rate of shrinkage strain.

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1. Introduction

Dental restorative filling composite resins have been introduced to dental community in 1960s [1]. Since then, after many significant material improvements, restorative composite resins still suffer from two key shortcomings: deficiencies of mechanical strength and high polymerization shrinkage

[2]. Thus, advanced research have been undertaken to evaluate and improve composite resin in order to have a material with high strength and low polymerization shrinkage combined with advantages of esthetic properties. Attempts have been made to change the type of fillers or filler size and their surface silanization. By changing the polymerization kinetics of resins matrices and degree of monomer conversion has

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tried to be influenced [3–6]. Reinforcing the resin with glass fibers [7–9], with fiber-reinforced composite (FRC) substructure [10], whiskers [11], particulate ceramic fillers (dense and porous) [12] and optimization of filler content [3] are among the methods that have been studied. However, further significant improvements are still needed. The linear shrinkage of current composite resin ranged from 2 to 3% after curing [13]. Such shrinkage causes gaps to lead to secondary carries, which is a major problem in current restorative dentistry. Many factors affect the shrinkage of composites resins, including resin matrix composition, filler content, and polymerization method [14–16]. Filler technology has led to the development of composite resins characterized by containing zirconia or silica nanoparticle fillers of approximately 25 nm size and nanoaggregates of approximately 75 nm size.

Glass fibers have been investigated to reinforce dental polymers for over 30 years [17]. They have documented reinforcing efficiency and good esthetic qualities compared to carbon or aramid fibers [18–20]. The effectiveness of fiber reinforcement is dependent on many variables, including the resins used, the quantity of fibers in the resin matrix [21,22], length of fibers [22], form of fibers [23], orientation of fibers [24], adhesion of fibers to the polymer matrix [25], and impregnation of fibers with the resin [26]. Short random fibers provide an isotropic reinforcement effect in multidirections instead of one or two directions, as described by Krenchel [27].

Polymethyl methacrylate (PMMA) based semi-interpenetrating polymer network (semi-IPN) matrix has been established as a polymer matrix in denture base materials [28]. Also some products of fiber-reinforced composite use semi-IPN-polymer matrix [29]. However, dental restorative composite resins with semi-IPN-polymer matrix in combination with short glass fibers and particulate nanofillers have not been evaluated to the author's knowledge.

Therefore, the objective of the study was to provide an experimental material, which combines glass fiber, semi-IPN and nanofiller technologies.

Specifically, this study investigated the effect of nanofiller fraction and temperature on polymerization shrinkage on glass fiber reinforced filling material.

2. Materials and methods

2.1. Materials

Dimethacrylate (BisGMA 67% [bisphenol A-glycidyl dimethacrylate] and TEGDMA 33% [triethylenglycol dimethacrylate]) resin consisting nanofillers (SiO₂, 20 nm in size) with various weight fractions (Hanse Chemie, Germany) (Table 1) and E-glass fibers with BisGMA-PMMA [polymethylmethacrylate, Mw 220,000] resin matrix (everStick, StickTech Ltd., Turku, Finland). In addition, radio-opacity fillers of BaAlSiO₂ (3 ± 2 μm in size) (Specialty Glass, USA) were incorporated to the resin system. Before the BaAlSiO₂ filler particles were incorporated into the resin matrix, they were silane treated using previously defined technique [30]. Commercial particulate filler composite (Grandio Caps, VOCO, Germany) was used as a commercial control group.

2.2. Methods

Experimental fiber composites (FC) were prepared by mixing 22.5 wt% of short E-glass fibers (3 mm in length) to 22.5 wt% of resin matrix with various weight fractions of nanofillers (0, 10, 20, 30, 40, 50 wt%) and then 55 wt% of BaAlSiO₂-radio-opacity fillers were added gradually to the mixture). Classification of the test groups according to various filler content is given in Table 1. The mixing was carried by using high speed mixing machine for 5 min (SpeedMixer, DAC, Germany, 3500 rpm). The dimethacrylate based resin matrix consisting PMMA forms semi-IPN-polymer matrix for the composite of FC. Experimental control group of particulate filler composite resin was prepared by mixing 22.5 wt% of dimethacrylate resin (with 50 wt% nanofillers) to 77.5 wt% of silane treated BaAlSiO₂-fillers. Commercial particulate filler composite (Grandio) was used as a commercial control group. All groups used in this study are listed in Table 1.

The shrinkage strain was measured at two different temperatures (26 and 37 °C) using the Watt's bonded-disk technique [31] (Fig. 1). The specimens from each group (n = 5) were photo-polymerized for 40 s using a light source with an irradiance of 550 mW/cm² (Optilux-500, Kerr, CT, USA) and the shrinkage strain data was recorded with respect a time (1 h).

Table 1 – Classification of test groups used in the study according to their filler content and composition (n = 5, per group)

Groups	Fibers (wt%)	Nanofillers (wt%) in the resin matrix/resin matrix (22.5 wt%)	Micrometer scale fillers (wt% of the resin–nanofiller–fiber mixture)
A (control)	–	–	77.5
B	0	0/22.5	77.5
C0	22.5	0/22.5	55
C1	22.5	10/22.5	55
C2	22.5	20/22.5	55
C3	22.5	30/22.5	55
C4	22.5	40/22.5	55
C5	22.5	50/22.5	55

0: No fibers, no nanofillers; A: commercial particulate filler composite (Grandio); B: experimental particulate filler composite; C: experimental fiber composite.

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