



Degradation of optical and surface properties of resin-based composites with distinct nanoparticle sizes but equivalent surface area



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ABSTRACT

Objective: To determine the optical and surface properties of model resin-based composites made with distinct nanoparticle sizes but equalized surface area, among them, before and after ageing.

Methods: Model resin-based composites based on BisGMA/TEGDMA (50:50 mol%) were loaded with three different fumed silica filler content (7 nm/examethyldisilazane, 12 nm/octylsilane, and 16 nm/dimethylchlorosilane), but with equivalent surface area (BET method). The optical and surface properties of the disk-shaped specimens were measured in 24 h after photoactivation (baseline) and repeated after water storage (90 days at 37 °C), and after abrasion (20,000 cycles of brushing). CIELAB individual parameters, color difference (ΔE^*_{ab}), translucency parameter (TP), gloss (G.U.) and roughness (Ra parameter) were measured. Data were statistically analyzed at $\alpha=0.05$ significance level.

Results: CIE L^* and TP were not affected by ageing in any group. CIE a^* was increased and CIE b^* decreased for the 12 nm and 16 nm groups and did not show any difference for the 7 nm group. After ageing, the lowest ΔE^*_{ab} was observed for the 7 nm group. Gloss and roughness were statistically equivalent for all groups, before and after ageing. The water storage didn't show any significant difference while the abrasion decreased gloss and increased the roughness.

Conclusion: The optical properties of the tested materials were not dependent of the fillers' surface area, while the surface properties were.

Clinical significance: Several factors influence the ageing resistance of the resin-based composites, including the surface area of the fillers and the amount and coupling treatment of them. The improvement of the material's physical stability could avoid the premature replacement of dental restorations.

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1. Introduction

Dental resin-based composites are constituted by organic and inorganic components. The organic phase comprises different molecular weight monomers that polymerize to produce a polymer matrix. The inorganic content is generally composed of ceramic oxides and/or glass fillers [1] and their morphology is directly associated with the material's physical properties as well

as handling properties [2]. A coupling agent (usually an organosilane) binds the polymeric matrix with the inorganic components.

The inorganic content has been purposely reduced in size to facilitate surface polishing and reduce wear damage [1,3]. It has been suggested that the dispersion of nanofillers into the polymeric matrix improves the material's physical properties after photo-activation [3–5]. However, nano-structured resin-based composites are very difficult to process with conventional techniques. There is a strong tendency for filler agglomeration due to stronger hydrogen bonding by hydroxyl groups and high surface free energy. The smaller the filler, the more difficult it becomes to break down these agglomerates to yield a homogeneous

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distribution within the polymeric matrix [6]. The particle agglomeration might also be influenced by the coupling agent type and fraction [7].

From a clinical perspective, a highly stable appearance can increase the longevity of restorations. The optical and the surface properties of resin-based composites can be modified by several factors such as wear, degradation of the polymeric matrix or filler components, and weakening of matrix–filler bonding. Several studies have focused on the effect of filler type, shape, and size on the resin-based composites' overall appearance as well as their ageing resistance [8–10]. In a previous study [9], nano-structured resin-based composites were made with three different silanized fumed silica fillers (7 nm, 12 nm, and 16 nm average sizes) dispersed into a monomeric matrix with the same mass content. The filler size had a significant influence on the optical and surface properties of the composites. The smallest filler size material showed improved color stability and gloss retention. However, this resulted in different handling characteristics among the groups due to the surface energy of the different fillers.

Though the size of fillers in dental resin-based composites has been the subject of many articles, the surface area of the fillers has not been accounted for, with respect to the overall appearance. Therefore additional studies are required to analyze the optical and surface properties of resin-based composites when the filler surface area is kept constant. The objective of this study was to evaluate the optical properties, by the CIELAB individual parameters, the CIELAB color difference, and the translucency parameter, as well as surface properties, by the gloss and the roughness, of model resin-based composites formulated with different filler size but having equivalent surface area, before and after ageing procedures. The hypotheses were that the model resin-based composites with different filler sizes but equivalent surface area would show (i) similar effects on optical properties after ageing, and (ii) similar effects on surface properties after ageing.

2. Materials and methods

2.1. Experimental design

This *in vitro* 3 × 3 factorial-designed study (n = 6) evaluated two factors: filler content (7 nm, 12 nm, and 16 nm) and ageing conditions (baseline, water storage and surface abrasion). The optical and surface property measurements were performed at the three ageing conditions: a) 24 h after a curing period in dry and dark storage (baseline); b) after the immersion of all specimens in distilled water at 37 °C for 90 days, and c) after toothbrush abrasion of the top surface of the specimens. The sample size was based on previous study [9]. The collected response variables were CIELAB individual parameters (CIE L^* , CIE a^* , and CIE b^*), translucency parameter, CIELAB color difference (ΔE^*_{ab}), gloss, and roughness.

2.2. Surface area measurements and composite formulation

Three different silanized hydrophobic fumed silica fillers with average filler diameter of 7 nm (hexamethyldisilazane; Aerosil® R

812, Evonik Industries, Germany), 12 nm (octylsilane; Aerosil® R 805, Evonik), and 16 nm (dimethylchlorosilane; Aerosil® R 972, Evonik) were selected for this study (Table 1). The surface area of each filler type was determined by the BET method [11], with the use of a surface area analyzer (Quantasorb QS-9, Quantachrome Instruments FL, USA). The obtained BET surface area values for each filler particle, presented in Table 1, were in accordance to those reported by the manufacturer.

The model composites were prepared with a polymeric matrix composed of 2,2 bis[4-2(2-hydroxy-3-methacryloxypropoxy) phenyl] propane (BisGMA, Esstech Inc., Essington, PA, USA) and triethyleneglycol dimethacrylate (TEGDMA, Esstech Inc.) in a 50:50 weight ratio. Camphorquinone at 0.5 mol% (Esstech Inc.) associated with ethyl 4-dimethylaminobenzoate at 0.9 mol% (Sigma-Aldrich, Chemie, Steinheim, Germany) were added as the photoinitiator:co-initiator system, based on a previous study [12].

In order to equalize the total surface area among each of the different filler particles, the model resin-based composites were formulated with different weights of filler, as shown in Table 1. Filler loading for the higher average particle size (16 nm) was set at 45.5 wt% based on a previous study [9] where the same fillers were tested. Then the other filler fractions (7 nm and 12 nm) were proportionally reduced. All components were weighed with 0.0001 g accuracy using an analytical balance (AX 200, Shimadzu Corporation, Tokyo, Japan), and were mechanically mixed (DAC 150 Speed Mixer, Flacktek, SC, USA) for 1 min to produce a homogeneous paste.

Six disc-shaped specimens were made for each group in a cylindrical metal mold of 8-mm inner diameter and 2-mm thickness. After the composite insertion, the top surface was covered with a Mylar strip and made flat by pressing down with a glass slab. The specimens were light cured from the top surface for 40 s using a LED curing unit (Radii CAL, SDI, USA) operated in standard mode with irradiance of 1000 mW/cm², as measured with a hand-held radiometer (LED radiometer, Demetron, Kerr, WI, USA). Then, all specimens were mechanically polished on both surfaces with the use of a grinding/polishing machine (MetaServ 250, Buehler, IL, USA) with 2000 and 4000-grit SiC paper under continuous water cooling.

A toothbrush simulating machine (MEV2, Odeme, SP, Brazil) was used to abrade the specimens' top surface [9]. Soft toothbrushes (Colgate Professional Extra Clean, Colgate-Palmolive Company, SP, Brazil) were fixed in a toothbrush holder so that all the bristles were in contact with the specimen. The machine was adjusted to apply 2.5 N of vertical load on the specimens during horizontal movements of the toothbrush throughout the test. A popular dentifrice (Colgate Total 12 Clean Mint, Colgate-Palmolive Company) was used to form the slurry (2:1, water: toothpaste) according to ISO/TS 1469-1. Each "station" of the machine was filled with 5 ml of slurry. All specimens were brushed with 20,000 cycles, which corresponds to approximately 2 years of toothbrushing [13]. The toothbrushes and the slurry were replaced for each specimen every 2500 cycles. After the abrasion, the specimens were removed from the machine, rinsed with tap water, cleaned in an ultrasonic bath with distilled water for 5 min, and gently air dried.

Table 1
Characteristics of the filler particles and formulation of the dental composites tested.

Average filler size	Filler coupling agent	Surface area (BET method)	Filler fraction (wt%)	Polymeric matrix fraction (wt%)	Polymeric matrix formulation
7 nm	hexamethyldisilazane	246 m ² /g	15.5	84.5	BisGMA/TEGDMA (50:50 mol%), camphorquinone (0.5 mol%), ethyl 4-dimethylaminobenzoate (0.9 mol%)
12 nm	octylsilane	178 m ² /g	32.0	68.0	
16 nm	dimethylchlorosilane	135 m ² /g	45.5	54.5	

All fillers were from Evonik, Germany. BisGMA, TEGDMA, and CQ were from Esstech Inc., USA. EDMAB was from Sigma-Aldrich, Germany.

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