Effect of Artificial Aging on the Roughness and Microhardness of Sealed Composites

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ABSTRACT

Statement of the Problem: The application of surface sealant could improve the surface quality and success of composite restorations; however, it is important to assess the behavior of this material when subjected to aging procedures.

Purpose: To evaluate the effect of artificial aging on the surface roughness and microhardness of sealed microhybrids and nanofilled composites.

Materials and Methods: One hundred disc-shaped specimens were made for each composite. After 24 hours, all samples were polished and surface sealant was applied to 50 specimens of each composite. Surface roughness (Ra) was determined with a profilometer and Knoop microhardness was assessed with a 50-g load for 15 seconds. Ten specimens of each group were aged during 252 hours in a UV-accelerated aging chamber or immersed for 28 days in cola soft drink, orange juice, red wine staining solutions, or distilled water. Data were analyzed by two-way analysis of variance and Fischer's test ($\alpha = 0.05$).

Results: Artificial aging decreased microhardness values for all materials, with the exceptions of Vit-l-escence (Ultradent Products Inc., South Jordan UT, USA) and Supreme XT (3M ESPE, St. Paul, MN, USA) sealed composites; surface roughness values were not altered. Water storage had less effect on microhardness, compared with the other aging processes. The sealed materials presented lower roughness and microhardness values, when compared with unsealed composites.

Conclusions: Aging methods decreased the microhardness values of a number of composites, with the exception of some sealed composites, but did not alter the surface roughness of the materials.

CLINICAL SIGNIFICANCE

The long-term maintenance of the surface quality of materials is fundamental to improving the longevity of esthetic restorations. In this manner, the use of surface sealants could be an important step in the restorative procedure using resin-based materials.

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INTRODUCTION

Currently, composite resins are widely used in dental practice as materials for esthetic restorations because of the increased esthetic demands by patients, simplification of bonding procedures, and improvement in their composition and characteristics.[1–4]

High-quality finishing and polishing of dental restorations are essential steps in the restorative procedure that enhances the aesthetics and clinical longevity of restored teeth.[3,5] As the resin matrix and filler particles differ in hardness, they do not abrade to the same degree.[3,6] Thus, various surface defects can appear in materials, such as microcracks and irregularities, because of the removal of some of the surface particles during finishing,[3,7,8] decreasing the wear resistance of the restoration.[7]

The surface degradation of composites caused by the erosion of the composite matrix and the exposure of filler particles after UV-accelerated aging has been reported in the literature.[9] In addition, exposure to low-pH drinks, alcoholic beverages, and even water could affect esthetic and physical properties, such as microhardness, surface roughness, and translucency of composites.[1] The chemicals in beverage formulations could lead to wear and surface degradation of composite materials,[1] increasing the risk for plaque accumulation and staining. A previous study[10] defined a critical surface roughness threshold of 0.2 μm (Ra), above which dental plaque accumulation may occur, thus favoring the development of both caries and periodontal inflammation.[10]

The main objective of surface sealants is to improve the sealing and marginal integrity of composite materials, increasing the smoothness and aesthetic quality of the restoration. According to Bertrand and colleagues (2000),[8] the surface sealant decreased the microhardness, improving the surface quality of all composites because of the disappearance of microfissures and slight surface irregularities.

However, few studies have assessed the effect of different aging processes on the surface roughness and microhardness of sealed composite resins with surface sealants. Based on this information, the following null hypotheses were tested: (1) the aging procedures would not cause alterations on the surface roughness and microhardness of composite restoratives whether sealed or not; and (2) there would be no difference among the different types of composites for the same factors evaluated.

MATERIALS AND METHODS

The materials used in this study are listed in Table 1. One hundred disc-shaped specimens were prepared for each composite (6 mm in diameter and 1.5-mm thick), totaling to 300 specimens. For the confection of the samples, a metallic matrix was filled with composite resin, then covered with a polyester strip and a microscope slide. To compress the material and prevent bubble formation, the glass blade was gently pressed to remove excess material. The composite was polymerized for 20 seconds, according to the manufacturer's recommendations, using a halogen light-curing unit (UltraLux; Dabi Atlante, Ribeirão Preto, São Paulo, Brazil) at 570 mW/cm^2, which was monitored by a radiometer (model 100; Demetron/Kerr, Danbury, CT, USA). Specimens were stored at 37°C and 100% relative humidity for 24 hours to ensure complete polymerization.

After this period, the top surface of all samples was polished using an automated polishing machine (APL-4; Arotec Ind. Com., Cotia, São Paulo, Brazil), with 360-, 600-, and 1,200-grit sandpaper, under water irrigation; samples were cleaned using an ultrasonic cleaner (model 2210; Branson Ultrasonics Corp., Danbury, CT, USA) with deionized water for 2 minutes between the different sandpapers and at the end of the process.

TABLE 1. MATERIALS USED IN THIS STUDY.

Code	Material	Product/Shade	Batch no.	Composition*	Manufacturer
SUP†	Nanofilled composite resin	Supreme XT/A2E	7EF	Filler: 59.5 vol.% combination of aggregated zirconium/silica cluster with primary particle size of 5–20 nm, and a non-agglomerated 20-nm silica filler Resin: Bis-GMA, Bis-EMA, UDMA, and TEGDMA	3M ESPE, St. Paul, MN, USA
VIT†	Microhybrid composite resin	Vit-l-escence/Pearl Neutral	G0212	Filler: 58 vol%, microhybrid filler of 0.7 µm Resin: Bis-GMA based	Ultradent Products Inc., South Jordan UT, USA
OPL‡	Microhybrid composite resin	Opallis/ EA2	131107	Filler: 57–58 vol% combination of Barium-Aluminum, silicate, and nanoparticles of silicon dioxide. Particle size between 40 nm and 3 µm, with an average particle size of 0.5 µm. Resin: Bis-GMA, Bis-EMA, and TEGDMA	FGM Dental Products, Joinville, Santa Catarina, Brazil
B‡	Liquid polish	Biscover LV	0700008228	Dipentaerythritol pentaacrylate esters and ethanol	Bisco Inc., Schaumburg, IL, USA

*In accordance with †Lee and Powers, and ‡manufacturers.

Fifty specimens of each composite were etched with 32% phosphoric acid (UNI-ETCH; Bisco Inc., Schaumburg, IL, USA) for 15 seconds, rinsed with distilled water, and air-dried. Subsequently, the Biscover LV surface sealant (Bisco, Inc.) was applied using an applicator tip; samples were light-polymerized for 30 seconds, according to the manufacturer's instruction, using a halogen light-curing unit (UltraLux). The other 50 specimens of each composite remained unsealed.

Surface Roughness Evaluation

Surface roughness was determined with a profilometer (Surftest SJ-400; Mitutoyo Corp., Tokyo, Japan). Each specimen was individually fixed in a clamping apparatus and the profilometer meter's needle was positioned on the specimen surface, moved at a constant speed of 0.05 mm/s, using a cut-off of 0.25 mm and characterized by the arithmetical mean of surface roughness (Ra). Three readings were taken on each surface in different positions, and the average was calculated. Each reading was obtained after turning the specimen 120 degrees.

Knoop Microhardness Evaluation

Knoop microhardness number (KHN) was performed using a microhardness tester (HMV-2000;

TABLE 2. KNOOP MICROHARDNESS (KHN) OF THE MATERIALS TESTED BEFORE AND AFTER AGING PROCESSES.*

		Aging				
	Before	Distilled water	Red wine	Orange juice	Cola soft drink	Accelerated aging
VIT	82.5 (1.1) B a	79.2 (1.7) B b	71.5 (1.2) B d	74.8 (1.8) B c	72.4 (2.0) B d	72.3 (2.2) B d
VIT-B	43.8 (4.9) E a	42.9 (5.0) D a	39.8 (5.3) E a	44.6 (6.1) C a	43.5 (4.9) D a	41.1 (2.6) C a
OPL	79.6 (0.9) C a	71.8 (2.0) C c	62.2 (2.5) C e	77.8 (1.3) B b	67.5 (2.0) C d	71.8 (0.8) B c
OPL-B	41.6 (3.2) F a	39.2 (2.7) E b	36.7 (4.0) F bc	39.5 (2.7) D ab	37.6 (5.0) E bc	36.1 (1.7) D c
SUP	96.9 (4.0) A a	92.4 (2.8) A b	89.1 (1.8) A c	90.3 (2.4) A bc	90.2 (1.8) A bc	91.7 (3.4) A bc
SUP-B	45.3 (5.3) D a	43.0 (2.8) D a	43.8 (4.2) D a	45.1 (6.5) C a	46.2 (4.1) D a	42.9 (5.8) C a

*Means followed by distinct letters. Capital letters in a column and lower case letters in rows are statistically different (5%) (standard deviation).

Shimadzu Corp., Kyoto, Japan), with a 50-g load applied for 15 seconds. The specimens were individually fixed in a clamping apparatus and positioned perpendicular to the tester tip, and KHN values were evaluated by the C.A.M.S program (New Age Industries, Southampton, PA, USA). In each sample, five indentations at different points were recorded, and the microhardness average value was calculated.

Artificial Aging Procedures

After the roughness and microhardness baseline evaluation, 10 specimens from each experimental group were aged with a UV-accelerated aging chamber EQUV (Equilam Ind. Com. Ltda, Diadema, São Paulo, Brazil), according to the ASTM G154.[11] The accelerated aging process consisted of alternating periods of ultraviolet light (8 hours) and condensation (4 hours), under heat (65 ± 3°C or 45 ± 3°C) and 100% humidity. Samples were subjected to a total of 252 hours of aging and 168 hours of UVB irradiation with a 313-nm emission peak.

The other samples of each resin-based composite were individually immersed ($N = 10$) in vials containing 5 mL of cola soft drink (Coca-Cola®, pH 2.36; Coca-Cola Co, Ribeirão Preto, São Paulo, Brazil), orange juice (Del Valle orange, ph 3.39; Del Valle juices of Brazil Ltda, Americana, São Paulo, Brazil), red wine (Concha y Toro Cabernet Sauvignon 2004, pH 3.41, Santiago, Chile), or distilled water (pH 6.37) for a 28-day test period, kept in incubator (ECB-2, Adamo Products for Laboratory Ltda., Piracicaba, São Paulo, Brazil) at 37°C temperature. The vials were sealed to prevent evaporation of the solutions and changed regularly every week.[12]

After aging procedures, roughness and microhardness were reevaluated. The data were analyzed using two-way analysis of variance (ANOVA) and a Fischer's PLSD test ($\alpha = 0.05$).

RESULTS

Knoop Microhardness

The two-way ANOVA showed significant differences among materials ($p < 0.0001$), aging processes ($p < 0.0001$), as well as interactions among these factors ($p < 0.0001$).

Table 2 shows the KHN of the materials before and after the aging processes. The artificial aging decreased the microhardness values for all materials, except for the Vit-l-escence (Ultradent Products Inc., South Jordan UT, USA) and Supreme XT (3M ESPE, St. Paul, MN, USA) sealed composites. Water storage had only a small effect on the KHN, when compared with other aging methods. The application of surface sealant decreased the KHN of sealed composite resins.

TABLE 3. SURFACE ROUGHNESS (μM) OF MATERIALS TESTED INDEPENDENTLY AFTER THE AGING PROCESSES.*

Groups	Surface roughness (μm)
VIT	0.052 (0.007) b
VIT-B	0.037 (0.009) d
OPL	0.059 (0.011) a
OPL-B	0.040 (0.009) c
SUP	0.050 (0.009) b
SUP-B	0.032 (0.008) e

*Means followed by distinct letters are significantly different at $p < 0.05$ (standard deviation).

Surface Roughness

Two-way ANOVA showed significant difference only among materials ($p < 0.0001$). The sealed materials presented the lowest surface roughness values compared with the unsealed composites (Table 3). All composites presented statistically different roughness values, except for Vit-l-escence and Supreme XT (Table 3).

DISCUSSION

The first null hypothesis tested was partially rejected because the aging had caused alteration in the microhardness; however, it had no effect in the roughness of materials. Furthermore, the second null hypothesis was also rejected because it had difference in the microhardness and roughness between the different tested materials.

In accordance with the results of Table 2, the immersion in distilled water decreased the hardness of the composites studied, except for VIT-B and SUP-B. Literature reports suggest that water could cause a softening of the polymer resin component by swelling the network and reducing the frictional forces between polymeric chains.[13] In low-pH drinks, resins present a high solubility and this solubility causes surface erosion and dissolution, negatively affecting the wear, hardness, and surface integrity by softening the matrix and causing a loss of structural ions.[14,15] Red wine, orange juice, and cola are low-pH beverages, and this could explain the results obtained in the study. Moreover, wine is an alcoholic beverage and it has been reported that the absorption of alcohol molecules contained in beverages and rinses into the resin matrix could result in the softening of the composite surface.[15–17] Bis-GMA and UDMA resin matrix monomers are susceptible to chemical softening by alcohol.[14] In the present study, all the solutions caused a reduction of the KHN composites, except for some sealed materials (Table 2). The surface sealant seems to have blocked the effect of artificial aging for SUP-B and VIT-B (Table 3). However, mechanical profilometry showed no significant difference among the surface roughness values of the materials tested after aging methods.

The accelerated aging process involves UV exposure and temperature and humidity changes, simulating the effects of long-term exposure to environmental conditions.[18] Scanning electron microscopy has shown an increased surface roughness of the various composite resins after UV irradiation exposure,[19] attributed to wear, exposure of interior porosities,[20] and microcracks with the exposure of the filler particles.[19] In the present study, although the microhardness decreased after 168 hours of UV irradiation, there was no significant difference in the roughness of the materials.

Surface sealant is composed mainly of monomer, which in the presence of alcohol as a solvent, may result in lower microhardness values for composite resins that are covered with this material (Table 2). Attar[5] assessed the effect of finishing and polishing procedures on the surface roughness of composite resins and observed that the use of surface sealant significantly improved the surface smoothness of all tested composites, as found in the current study. However, the type of composite resin influenced the values of microhardness (Table 2) and surface roughness (Table 3) of sealed materials; probably the different type and size of load particles of the studied composites could explain such results.

The surface roughness values were maintained for both sealed and unsealed composites after aging. The maintenance of lower microhardness values of the sealed composites after artificial aging could be indicative of the permanence of the surface sealant on the composite restoration, after the aging procedures. Moreover, the maintenance of the KHN for the majority of sealed composite resins after aging processes could be related to a more thoroughly cured surface without the presence of defects, resulting from the finishing and polishing procedures.[21] Although the sealed materials presented a lower surface roughness than unsealed composites, the surface roughness values of all the materials were below the threshold considered critical for the accumulation of bacterial plaque.[10]

In this study, a mechanical profilometer was used to measure the surface roughness of the materials. Joniot and colleagues[22] compared the surface roughness using two surface analyzers and concluded that mechanical profilometry, regarding the texture of surface, tends to show roughness caused by polishing, whereas optical profilometry detects microroughness, which generally reflects the structure of the material itself. According to Tholt and colleagues,[23] the combination of the profilometer and the atomic force microscope could characterize the surface topography over a length scale variable and, consequently, the results are more reliable and precise. Moreover, the constant exposure of the materials to the solutions during testing does not reflect the intermittent exposure that occurs in in vivo conditions. Thus, the study of other physical and mechanical properties will be essential for the development of new materials and restoring techniques in the search for increased clinical longevity of the composite restorations.

CONCLUSIONS

On the basis of the results and within the limitations of this study, it can be concluded that artificial aging decreased the KHN of the composite resins, except for some sealed materials. The sealed composites showed the lowest surface roughness and microhardness values of the composites compared with the unsealed materials; however, the surface roughness was not altered by any of the aging methods.

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