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Effect of Different Surface Penetrating Sealants on the Roughness of a Nanofiller Composite Resin

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This study evaluated the effectiveness of different sealants applied to a nanofiller composite resin. Forty specimens of Filtek Z-350 were obtained after inserting the material in a 6x3 mm stainless steel mold followed by light activation for 20 s. The groups were divided (n=10) according to the surface treatment applied: Control group (no surface treatment), Fortify, Fortify Plus and Biscover LV. The specimens were subjected to simulated toothbrushing using a 200 g load and 250 strokes/min to simulate 1 week, 1, 3 and 6 months and 1 and 3 years in the mouth, considering 10,000 cycles equivalent to 1 year of toothbrushing. Oral-B soft-bristle-tip toothbrush heads and Colgate Total dentifrice at a 1:2 water-dilution were used. After each simulated time, surface roughness was assessed in random triplicate readings. The data were submitted to two-way ANOVA and Tukey's test at a 95% confidence level. The specimens were observed under scanning electron microscopy (SEM) after each toothbrushing cycle. The control group was not significantly different ($p>0.05$) from the other groups, except for Fortify Plus ($p<0.05$), which was rougher. No significant differences ($p>0.05$) were observed at the 1-month assessment between the experimental and control groups. Fortify and Fortify Plus presented a rougher surface over time, differing from the baseline ($p<0.05$). Biscover LV did not differ ($p>0.05$) from the baseline at any time. None of the experimental groups showed a significantly better performance ($p>0.05$) than the control group at any time. SEM confirmed the differences found during the roughness testing. Surface penetrating sealants did not improve the roughness of nanofiller composite resin.

Key Words: surface sealant, toothbrushing, roughness, SEM.

INTRODUCTION

Since the introduction of bonding systems and composite resins, wear and microleakage were the primary clinical limitations, mainly in posterior restorations (1-8). The relative porosity of the restorative and absorption/adsorption of oral fluids may also allow staining agents to penetrate the material (9). In order to improve wear resistance, some specific composites were formulated (7,10). A more recent evolution of composite resins is associated with nanotechnology, resulting in materials that have better mechanical strength and high-polishing durability. These new materials are for universal use, according to the manufacturers (11).

Surface-penetrating sealants were developed to avoid or minimize wear rates of composite resins by filling the microdefects on the restoration surface and to reduce microleakage along the restoration/tooth interface (4-6,12). Additionally, some sealants also act as a chemical gloss by reducing the surface roughness (13). Different formulations of these materials are currently available on the market in a range of combined monomers, such as BisGMA (bisphenol-A-glycidyl dimethacrylate), TEGDMA (triethylene

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glycol dimethacrylate), THFMA (tetrahydrofurfuryl methacrylate) and UDMA (urethane dimethacrylate) (14). Fillers were added to some materials to increase their mechanical properties (4,6,12,13).

A low-viscosity sealant was found to be effective in improving posterior composite wear resistance in a 5-year clinical evaluation. After 1 year, the mean wear of the sealed restorations was about half of those not treated (4). However, hybrid composites were used in that study, which differ from nanofiller composites by presenting a greater filler size. Nanofiller composites have good wear resistance and satisfactory polishing (11), and the application of a surface sealant may not be necessary.

The aim of this *in vitro* study was to compare the effect of different surface-penetrating sealants on a nanofiller composite with regards to surface roughness. The null hypotheses of this study were: 1. There is no difference on the composite surface roughness after the application of surface sealants; 2. There is no difference on the composite-sealant surface roughness after simulated toothbrushing over time.

MATERIAL AND METHODS

Forty specimens of a nanofiller composite (Filtek Z350; 3M ESPE, St. Paul, MN, USA) were obtained using a stainless steel mold (6 mm Ø x 3 mm). The external surfaces of the mold were covered with polyester matrix strips (TDV Dental, Pomerode, SC, Brazil) that were lightly pressed with glass slabs. Polymerization was carried out with a light-emitting diode curing unit (Radi II; SDI, Victoria, Australia) at 1,000 mW/cm^2 for 20 s. After 24 h, the specimens were polished for 15 s with 600-grit sandpaper under water-cooling in low-speed polishing machine, and then ultrasonically cleaned (T-14; Tempo Ultrasonic Ind. Com. Ltda, Taboão da Serra, SP, Brazil) in deionized water for 10 min to remove the polishing debris.

The specimens were randomly assigned to 4 groups based on the surface treatment: Control group (no treatment), Fortify (Bisco Inc., Iatasca, IL, USA), Fortify Plus (Bisco Inc.) and Biscover LV (Bisco Inc.). The surfaces of the specimens in the experimental groups were conditioned with 37% phosphoric acid (Bisco Inc.) for 20 s, washed with air-water spray for 30 s and dried with an oil-free air stream. A thin coat of sealant was applied, air thinned for even distribution and light-cured according to the manufacturer's instructions.

After storage in deionized water at 37ºC for 24 h, surface roughness was analyzed with a profilometer (Surfcorder SE 1700; Kosakalab, Tokyo, Japan). The roughness values were expressed in micrometers (*Ra*). For each specimen, three measurements were made using a stylus tip with a 2 μm-diameter and 0.0001 μm accuracy. This device was adjusted to record the measurements under predefined parameters of 2.85 mm for the reading extension and a 0.25 mm cut-off.

Simulated toothbrushing was performed using toothbrush heads with soft nylon bristles (Oral B Indicator; Procter & Gamble do Brazil, São Paulo, SP, Brazil) under a 200 g load (6). The toothbrush heads were changed after every 10,000 strokes. For each specimen, 30,000 strokes were performed at a frequency of 250 strokes/min. A double pass of the toothbrush head was considered a stroke. Assuming that 10,000 cycles represented approximately 1 year of toothbrushing (7,15), the cycles were divided into different aging simulations of 1 week (312 strokes); 1 (1,250 strokes), 3 (2,500 strokes) and 6 months (5,000 strokes); and 1 (10,000 strokes) and 3 years (30,000 strokes). A slurry was prepared by mixing a 2:1 ratio of deionized water and a carbonate calcium particle dentifrice (Colgate Total 12; Colgate-Palmolive Ind. e Co. Ltda, São Paulo, SP, Brazil) immediately before testing. After testing, the specimens were cleaned with running water followed by an ultrasonic bath for 10 min.

Surface roughness of each abraded specimen was determined after each simulation period and recorded as described above. Each specimen was dried with absorbent paper and roughness tracings (*Ra* - roughness average) were taken on the test surface using Surfcorder SE 1700 (Kosakalab, Tokyo, Japan) equipment. The operating parameters were established at Lt (assessment length) of 2.85 mm and Lc (cut-off) of 0.25 mm (6). Three random readings were taken on each evaluated surface. Baseline R (mm) was obtained by calculating the arithmetic mean of these three readings. Comparisons before and after testing of the surface roughness of each material were determined by two-way ANOVA and Tukey's tests ($p<0.05$).

To illustrate possible events, scanning electron micrographs were taken of the surface of each resin composite before and after the abrasion procedures. Specimens were mounted on metal stubs, sputter coated with gold (SCD-050; Bal-Tec AG, Balzers, Liechtenstein) and examined under a scanning electron microscope (JSM-5600LV; JEOL Ltd., Akishima, Japan) at different magnifications.

RESULTS

Surface Roughness

Table 1 shows the means and standard deviations of surface roughness for all the tested groups.

Without toothbrushing, Fortify Plus showed greater surface roughness than all the other materials ($p<0.05$). From 1 week to 3 months of toothbrushing, no differences were found ($p>0.05$). From 6 months to 1 year, Fortify Plus showed greater surface roughness when compared with the other groups ($p<0.05$). At the 3-year evaluation, no differences were found ($p>0.05$).

At each tested time, Fortify Plus presented a greater surface roughness that at the initial time ($p<0.05$). Among the other groups, no significant differences were found over time ($p>0.05$).

SEM Analysis

The SEM micrographs confirmed the results. Figures 1A-1C present the sealant-free surface of Z-350 at the initial control time, after 1 week and after 3 months of toothbrushing simulation, respectively.

Before toothbrushing simulation, Fortify (Fig. 1D), Fortify Plus (Fig. 1G) and Biscover LV (Fig. 1J) were observed on the composite surface. Fortify Plus showed a thicker layer than Fortify and Biscover LV, which were similar to each other.

After 1 week of toothbrushing simulation, the wear of Fortify (Fig. 1E) and Biscover LV (Fig. 1K) were not uniform, as highlighted by arrows. The black arrows indicate the sealant while the white arrows indicate the exposed resin matrix (Fig. 1E and 1K).

After 3 months of toothbrushing, Fortify (Fig. 1F) and Fortify Plus (Fig. 1I) still remained on the composite resin surface. Biscover LV (Fig. 1L) was almost totally removed and the composite filler particles exposed, similar to the control (Fig. 1C).

Surface sealant removal did not occur uniformly when considering the total observed area, as shown in Figure 2.

Table 1. Means (μm) and standard deviations of surface roughness.

Time	Control	Fortify	Fortify Plus	Biscover LV
Initial	0.11 (0.02) Aa	0.08 (0.01) Aa	0.21 (0.10) Ab	0.08 (0.05) Aa
1 week	0.11 (0.01) Aa	0.09 (0.01) ABa	0.14 (0.04) Aa	0.14 (0.07) Aa
1 month	0.13 (0.02) Aa	0.11 (0.01) BCa	0.21 (0.15) Aa	0.14 (0.07) Aa
3 months	0.13 (0.02) Aa	0.12 (0.02) Ca	0.20 (0.10) Aa	0.19 (0.10) Aa
6 months	0.14 (0.06) Aab	0.11 (0.02) BCa	0.23 (0.11) Bb	0.17 (0.10) Aab
1 year	0.12 (0.03) Aa	0.12 (0.02) Ca	0.22 (0.12) Bb	0.19 (0.07) Aab
3 years	0.18 (0.07) Aa	0.15 (0.04) Ca	0.23 (0.10) Ba	0.24 (0.12) Aa

Different uppercase letters in the same column and lowercase letters in the same row indicate statistically significant difference ($p<0.05$).

DISCUSSION

When surface-penetrating sealants were launched on the market, a variety of laboratory studies and clinical trials evaluated these materials under different protocols (4-6,12,13). These studies seem to reveal a consensus about the effectiveness of sealants on reducing microleakage, despite the inability to totally avoid this occurrence (12).

Despite the progress of bonding systems, adhesion is still based on mechanical interlocking of dentin bonding systems to demineralized dentin. Dentin bonding systems, combined with resin-based restorative materials, undergo polymerization shrinkage (15,16), which may create gaps at the interface. This is the rationale of surface-penetrating sealants, which can penetrate these gaps and minimize the occurrence of microleakage. Previous studies have shown that this effect is relevant mainly along the dentin margins (17) by improving the marginal adaptation of composite restorations (18).

Surface-penetrating sealants also reduced the wear of composite resins and minimized the surface roughness under *in vitro* conditions and in clinical evaluations (4-6). A low-viscosity resin applied on the surface of a polymerized composite resin restoration could penetrate into the structural microgaps and microfractures, promoting marginal sealing (19). This material is a nanocomposite that contains only nanomeric particles and nanoclusters as inorganic fillers. Recently, a new composite resin based on nanotechnology was developed (20) promoting an improved chemical integration of the filler particles, which contains only nanomeric particles and nanoclusters (21) within the composite matrix (10), while reducing the wear rates of these composites (22). However, few studies have evaluated the relationship between this nanofiller composite resin and the surface sealant (14,23). Due to this lack of information in the literature, this study evaluated the behavior of this resin with surface sealants with and without filler.

According to the obtained results, the application of different surface-penetrating sealants apparently did not improve the surface roughness of nanofiller materials after 30,000 strokes of toothbrushing simulation. Thus, the null hypotheses of the present study were rejected.

When the control group was compared with specimens protected with Fortify, no differences were detected at any evaluated time. It is possible to speculate that, despite the wettability and low viscosity of this sealant, it did not promote better protection to the organic matrix of the nanofiller composite resin (20).

When filler was added to the sealant, as with Fortify Plus, its performance worsened when compared with the control group after 6 months of toothbrushing. The wear of the organic matrix of this sealant potentially allowed the filler to be protruded or lost, resulting in a rougher surface. The surface sealants have limited capacity to penetrate into structural defects as small as 1 or 2 µm (24); therefore, the sealant did not perform as expected on nanofiller surfaces.

Finally, the use of Biscover LV was also ineffective, corroborating with Perez et al. (23), who

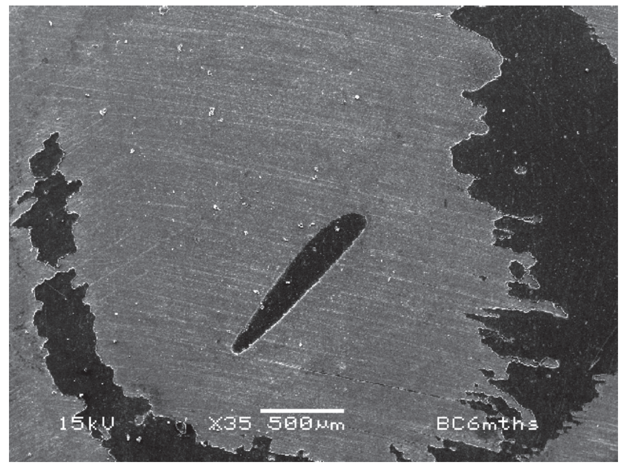

Figure 2. SEM micrograph of Biscover LV after 6 months of toothbrushing.

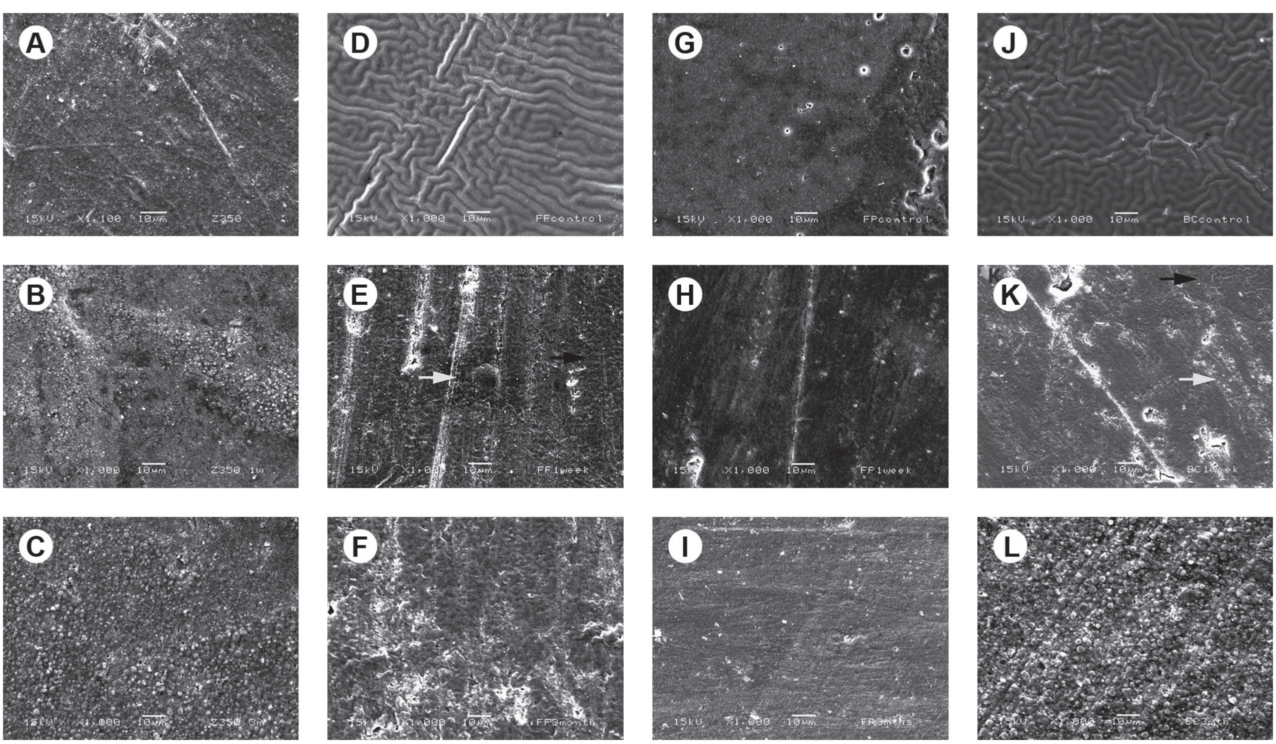

Figure 1. Composite figure of SEM micrographs of the control group at the initial control time (A), after 1 week (B) and 3 months (C) of toothbrushing simulation, and Fortify, Fortify Plus and Biscover LV at the initial time (D, E, F), after 1 week (G, H, I) and 3 months of toothbrushing simulation (J, K, L). Black arrows indicate the sealant and white arrows indicate the exposed resin matrix.

proposed its use alone to provide an initial and temporary gloss and polish (13).

Microscopically, a wave texture on Figures 1D (Fortify) and 1J (Biscover LV) can be observed, probably due to evaporation of the solvent from the material prior to polymerization. Cilli et al. (14) also reported these findings on Biscover surface. However, this is not observed in Figure 1G (Fortify Plus) due to the presence of the sealant filler.

Although surface-penetrating sealants have been claimed to minimize some limitations of resin-based materials, they seem to be ineffective in protecting a nanofiller resin surface against toothbrushing procedures. The surface-penetrating sealant was useless for the nanofiller composite resins as the filler removal from the surface during toothbrushing resulted in very small voids, which did not interfere with the surface roughness (25).

One limitation of this study was the quantification of sealant remaining on the composite surface and evaluation of the bond strength between the sealant and resin. As seen in Figure 2, the sealant appeared to be removed in blocks, not gradually as expected. Other studies, such as microleakage evaluations, should be conducted associated with nanofiller restorations, since microleakage still represents the greatest challenge to resin-based materials, even in the nanotechnology era. Few studies in the literature deal with the surface quality of nanoparticle composites using sealants with and without filler.

In conclusion, the tested surface-penetrating sealants do not seem able to improve the surface roughness performance of a nanofiller composite resin, highlighting the potential of the nanofil technology in obtaining an adequate surface roughness without the use of any sealant.

RESUMO

Este estudo avaliou a efetividade de diferentes selantes aplicados a uma resina nanoparticulada. Quarenta espécimes de Filtek Z-350 foram obtidos depois da inserção do material em um molde de aço inoxidável seguido por fotoativação por 20 s. Os grupos foram divididos (n=10) de acordo com o tratamento superficial aplicado: Grupo controle (sem tratamento superficial), Fortify, Fortify Plus ou Biscover LV. Os espécimes foram submetidos a escovação simulada usando uma carga de 200 g e 250 ciclos/min para simular 1 semana, 1, 3 e 6 meses e 1 e 3 anos, considerando que 10.000 ciclos equivalem a um ano de escovação. Escovas Oral-B de cabeça macia e dentifrício Colgate Total diluído a 1:2 em água foram utilizados. Depois de cada período de simulação, a rugosidade superficial foi medida em triplicata. Os dados foram submetidos à ANOVA de dois fatores e ao teste de Tukey com nível de 95% de confiança. Os espécimes foram observados em microscopia eletrônica de varredura (MEV) depois de cada ciclo de escovação. O grupo controle não foi diferente (p>0,05) que os outros grupos, exceto pelo Fortify Plus (p<0,05), que foi mais rugoso. Nenhuma diferença (p>0,05) foi observada em 1 mês de simulação entre os grupos experimentais e o controle. Fortify e Fortify Plus apresentaram rugosidade regular com o tempo, diferindo da inicial em todos os tempos. Nenhum dos grupos selados mostrou melhor desempenho (p>0,05) que o grupo controle em qualquer um dos tempos. MEV ressaltou as diferenças encontradas durante o teste de rugosidade. Selantes de penetração de superfície não melhoram a rugosidade da resina nanoparticulada.

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