Original Article

EFFECT OF DIFFERENT CLINICAL APPLICATIONS ON PHYSICO-MECHANICAL PROPERTIES OF COMPOSITE RESINS

EFEITO DE DIFERENTES APLICAÇÕES CLÍNICAS NAS PROPRIEDADES FÍSICO-MECÂNICAS DE RESINAS COMPOSTAS

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ABSTRACT: Although clinicians use fluoride agents to reduce the occurrence of caries, and surface sealing agents to protect composite restorations, the effects of these agents on composite resins have not yet been investigated. The goal of this study was to determine the effect of different surface applications (fluoride or surface sealant) on resin composites with different organic structures (Siloranes, Sonicfill, 3M Z550, Kalore). In this study, 120 discs and 120 bars made of composite resin were stored in water for three months before being thermally aged by cycling between 5°C and 55°C for 10,000 cycles. The discs were 15 mm in diameter and 1 mm thick; the bars were 25 × 2 × 2 mm. The surface sealant and fluoride were applied to the specimens, and evaluations were performed after 24h. Initial and final calculations were performed for flexural strength, microhardness, roughness, gloss, water sorption, and solubility. Silorane composite showed the lowest water sorption levels ($p < 0.05$) for both initial and aged groups. Silorane and SonicFill composite groups showed the lowest solubility ($p < 0.05$). Both before and after aging, the SonicFill group showed the highest values of flexural strength and microhardness. Silorane showed the highest roughness and lowest gloss values. Lower water sorption and solubility rates were seen on materials fabricated from hydrophobic monomers. High water sorption and solubility degrades the mechanical and surface properties. Fluoridation and surface sealant application can alter the surface properties but do not have any effect on the mechanical properties.

KEYWORDS: Acidulated phosphate fluoride. Adhesives. Mechanical properties. Silorane resins. Surface properties.

INTRODUCTION

Aesthetic properties are not the only reason why dental composites are widely used today; their ability to adhere to the tooth surface is also very important. Dental composites absorb fluids because of the nature of the organic matrix; sorption is affected by inadequate polymerization or the effects of oral fluids (WEI et al., 2011). Water sorption (WS), which occurs mainly by diffusion into the organic matrix, can result in degradation. Several factors, such as the types of organic fillers, thermal changes, or elapsed time will change the WS amounts (TOLEDANO et al., 2003). When water molecules diffuse into the composite, it expands the material by a process called "hydroscopic expansion," which triggers the chemical degradation that will result in the release of degradation products or filler particles from the material. The degradation of a composite can affect both the mechanical and surface properties of the material (PETROPOULOU et al., 2015, YAP et al., 2002).

Surface micro-irregularities can occur not only by degradation of the composite after aging, but also by the application of acidulated phosphate fluoride (APF). APF, which is used frequently for caries inhibition, contains acids which etch the enamel and consequently enhance the fluoride uptake (YAP et al., 2002). Hydrogen ions from the phosphoric acid and fluoride ions from sodium fluoride are present in the APF solution, and hydrofluoric acid is therefore generated. However, APF solution is also reported to dissolve dental materials that contain inorganic components; this affects the surface properties of dental materials such as composite resins, glass ionomers, and porcelain (MAIA et al., 2003. KIM et al., 2005).

Surface properties are among the main factors affecting the aesthetic appearance of composite materials. Even after employing appropriate finishing and polishing techniques, the surface can exhibit micro-irregularities that initiate material wear. To overcome this problem, a thin low-viscosity resin, called a "surface sealant," can be applied over polymerized composite restorations. Surface micro-irregularities or structural defects that have been filled with resin by capillary action enhance the surface smoothness, which provides a more uniform, regular surface (TAKEUCHI et al., 2003, CATELAN et al., 2010).

To date, no studies have assessed the effects of thermal aging on the mechanical properties or the surface properties (such as roughness and gloss) of composite resins with surface sealing or APF

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treatments. Consequently, our objectives were: (1) to determine whether the aging procedures would alter the mechanical properties of the composites, and (2) to ensure that there would be no alteration in the mechanical and physical properties of the composites as a result of the different surface treatments or aging in water and thermocycling that were finally selected.

MATERIAL AND METHODS

Four composite resins (Filtek Silorane, GC Kalore, Kerr SonicFill, and Filtek Z550) were studied (Table 1). The samples were fabricated in a Teflon mold covered with a transparent Mylar strip, and gently pressed with a glass slide to expel excess material; the disc-shaped samples were 15 mm in diameter and 1 mm thick, while the bars were 25 × 2 × 2 mm. The composite resins were light-polymerized (Optilux; Kerr, USA) through the Mylar strip following the manufacturers' instructions. The distance of the tip from the specimen was maintained to within 1 mm. The output of the polymerizing light was verified with a radiometer (Kerr/Demetron, Orange, USA). After polymerization, each of the four groups with the different resins was further subdivided into three subgroups with ten disks and ten bars in each subgroup. Group 1 was the Control group, which was not subjected to any surface treatment. Group 2 consisted of composites that received surface treatment with APF solution for 1 min. Group 3 consisted of composites which were then covered with dental bonding agent as a surface sealing material (Adper Single Bond Plus, 3M ESPE, St Paul, MN,USA) and light-polymerized. The specimens in the same group were stored together in a cylindrical vial in 20 mL of distilled water at 37°C for 24 h. For all three groups, WS and solubility (SL), flexural strength (FS), roughness, and gloss values were measured after 24 h and again after water immersion for 90 days followed by 10,000 cycles of thermal cycling between 5°C and 55°C.

Table 1: Resin composites used in this study

	Filler Loading	Fillers	Matrix
Z550	Nanohybrid %82 wt %68 vol	Zirconia silica,	Bis-GMA, UDMA, Bis-EMA, TEGMA PEGDMA
Siloran	Microhybrid %76 wt %55 vol	Quarz (silane layer), radiopaque yttrium fluoride	Silorane (3,4-epoxycyclohexylethylcyclopolymethylsiloxane, bis-3,4-epoxycyclohexylethylphenylmethylsilane)
Sonicfill	Nanohybrid % 83,5 wt % 83 vol	Glass, oxide, and Silicon dioxide.	TMSPMA EBPADMA, bisphenol-A-bis-(2-hydroxy-3-mehacryloxypropyl) ether, TEGDMA
GC Kalore	Nanohybrid %82 wt %69 vol	Prepolymerized filler (with lanthanoid fluoride), fluoro-alumino-silicate glass, strontium/barium glass, silicon dioxide, lanthanoid fluoride	DX-511, UDMA (urethane dimethacrylate) and dimethacrylate co-monomers

Water sorption and solubility

WS and SL tests were performed according to ISO 4049, and the calculations were performed using the equations described below:

$W_{sp} = (m_2 - m_3)/V$

$W_{sl} = (m_1 - m_3)/V$,

where W_{sp} is water sorption, and W_{sl} is the solubility, which is calculated by weighing the specimens (m_1) after having placed them in a desiccator containing dehydrated silica gel at 37°C for 24 h; m_2 is the mass of the specimen in micrograms, after immersion in water followed by removal of the excess water using absorbent paper; m_3 is the dehydrated mass in micrograms of the specimen after water storage followed by storage in a desiccator containing dehydrated silica gel at 37°C until constant mass was achieved (AX200, Shimatzu Corp., Japan); and V is the specimen volume in mm^3. An electronic micrometer was used to measure the diameter and thickness of each specimen.

Flexural strength

A universal testing machine (AGX, Shimatzu, Japan) was used for calculating the FS of the composites. A gap of 20 mm was maintained between the supports. A load was applied at a constant crosshead speed of 0.5 mm/min until fracture occurred.

$\sigma=3Fl/2bh^2$

F maximum load (Newton). l distance between supports, millimeters. b width at the centre of the specimen, millimeters. h height at the centre of the specimen, millimeters. d deflection due to load, millimeters.

Microhardness

Microhardnesses were calculated using an automatic microhardness indenter (Innovatest, England). The Vickers hardness is defined as the test force divided by the apparent area of the indentation under the applied test force. The test load was increased from 0.4 to 500 mN at a constant rate. The load and the penetration depth of the indenter were continuously measured during the load–unload hysteresis.

Roughness and Gloss Measurement

Gloss was measured at the baseline and after aging with a glossmeter in gloss units (GU) (Elcometer 407, Leicester England) with a 60° geometry. The average surface roughness (Ra) was measured three times at the baseline and again after aging; a surface roughness tester (Surtronic 25; Taylor-Hobson, Leicester, England) was employed, with a cutoff value of 0.8 mm, a transverse length of 4.5 mm, and a measuring speed of 0.25 mm/s.

Statistical analysis

Data distribution was first analyzed for the normal distribution using the Kolmogorov–Smirnov test with a statistical software program (SPSS version 22.0, SPSS Inc., Chicago, IL, USA). A p-value of <0.05 was considered statistically significant. Two-way ANOVA was subsequently employed to examine the composites used. Data from the restorative material and surface treatment data were analyzed using one-way ANOVA and Tukey's test. The data that were not normally distributed were analyzed using the nonparametric Kruskal–Wallis test and the Mann–Whitney U test.

RESULTS

The mean WS and SL values for the four restorative materials at the baseline and after each surface treatment are displayed in Figures 1 to 5. The lowest values for sorption were observed for Filtek Silorane (10.1 µg/mm^3), whereas Filtek Z550 showed the highest sorption value (29.5 µg/mm^3). SonicFill had the lowest value of SL (2.7 µg/mm^3), but there was no statistical difference between it and Filtek Silorane (3.1 µg/mm^3).

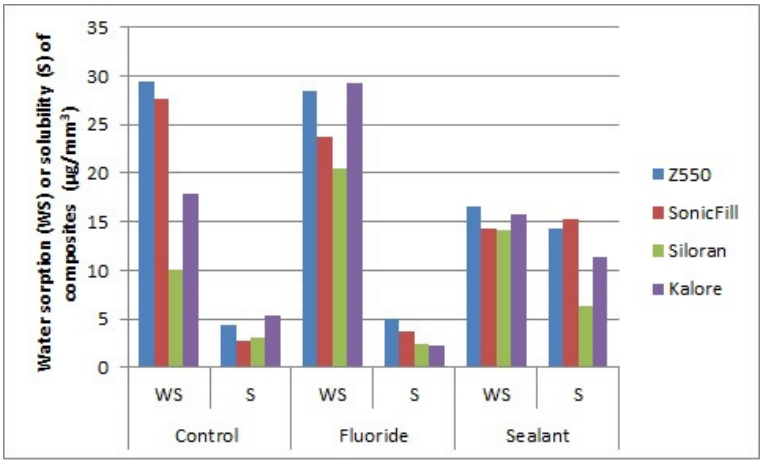

Figure 1. Water sorption (WS) and solubility (S) of composites

FS data are summarized in Figure 2. The statistical analyses indicated that the groups differed significantly ($p < 0.001$). Specifically, the multiple-comparison test demonstrated that SonicFill possessed the highest FS values; significantly lower values were obtained for Filtek Silorane and Kalore.

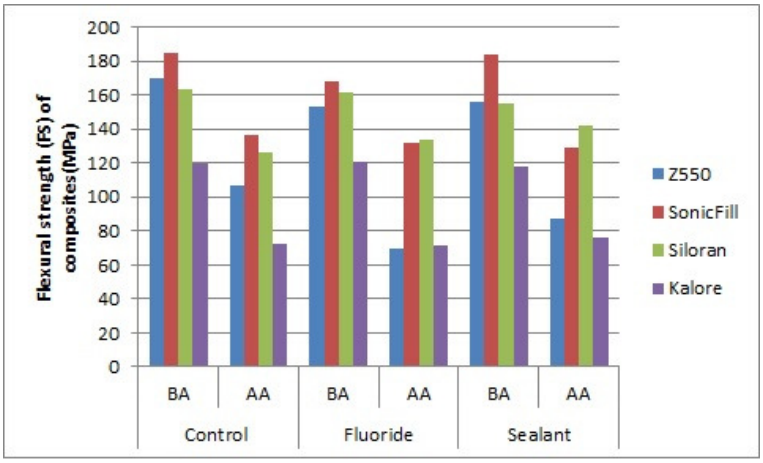

Figure 2. Flexural strength (FS) of composites before aging (BA) and after aging (AA)

Z550 and Sonicfill showed statistically significant reductions in their microhardness values after treatment with fluoride or surface sealant (Figure 3) ($p < 0.05$). There were statistically significant differences found between the surface roughness (Figure 4) and gloss (Figure 5) of the control, APF-treated, and surface-sealant-treated groups ($p < 0.001$).

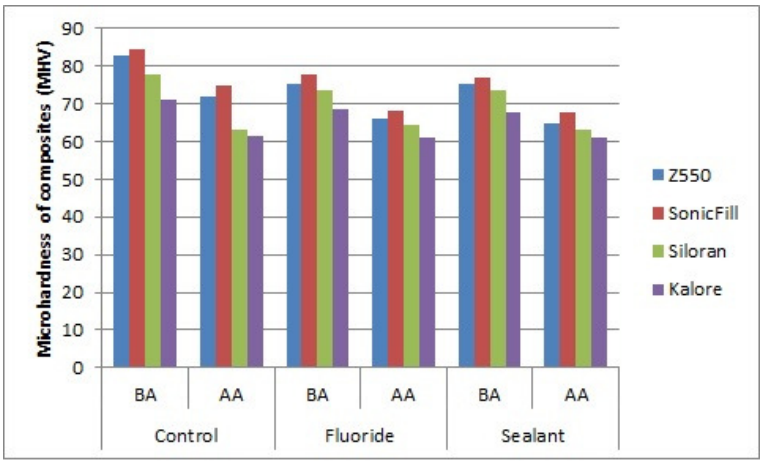

Figure 3. Microhardness of composites before aging (BA) and after aging (AA)

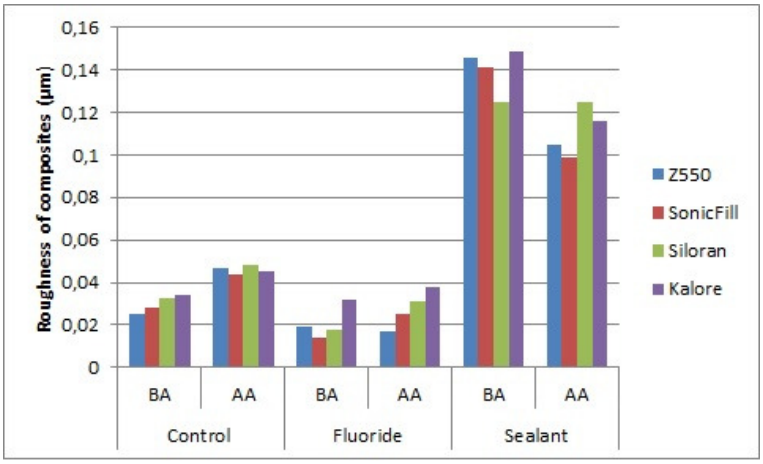

Figure 4. Roughness of composites before aging (BA) and after aging (AA)

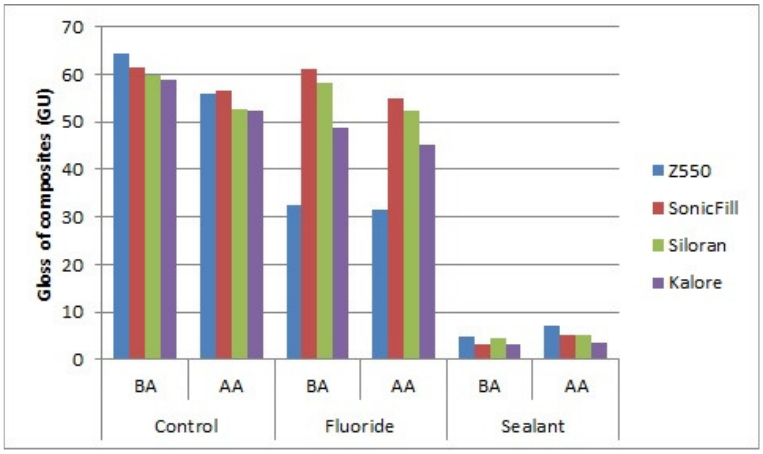

Figure 5. Gloss of composites before aging (BA) and after aging (AA)

DISCUSSION

These experiments show that aging affected the microhardness, FS, roughness, and gloss of the materials. Application of APF solution or surface sealants on the surface of the composite caused alterations in the microhardness, roughness, and gloss.

Dental composites contain different inorganic fillers (zirconia, quartz, glass) and different organic methacrylate monomers like bisphenol A-glycidyl methacrylate (Bis-GMA), urethane dimethacrylate (UDMA), ethoxylated bisphenol A glycol dimethacrylate (Bis-EMA), triethylene glycol monomethacrylate (TEGMA), or polyethylene glycol dimethacrylate (PEGDMA) together with additives. Manufacturers try to overcome the problem of polymerization shrinkage mainly by increasing the inorganic filler ratio (FONSECA et al., 2017). Another approach that manufacturers employ to overcome polymerization shrinkage is to use "low contraction" monomers like siloranes or DX-511 in the organic matrix of the composites (WEINMANN et al. 2005; SIDERIDOU et al, 2015).

WS of the polymer matrix might influence the hydrolytic stability of dental composites (ÖRTENGREN et al., 2001). Two of the composites (Filtek Z550 and SonicFill) contain mainly Bis-GMA and Bis-EMA, TEGDMA, and UDMA; organic matrices of this type did not seem to differ from each other in terms of WS levels. The lower WS values of Sonicfill can be explained by the high filler ratio (less organic matrix) in Sonicfill compared with Z550. The hydrophilic nature of Bis-GMA is the key factor controlling the WS of this material (ITO et al., 2005). The TEGDMA monomer was added to the composites to promote dilatation of the high-viscosity polymer. This monomer used for dilatation, which combines with the hydroxyl groups in the Bis-GMA monomer, can cause WS. WS causes expansion of the restoration, which is destructive to the structure (MARTIN et al., 2003). Two of the composites (Filtek Silorane and GC Kalore) showed less WS and SL than the composites containing Bis-GMA; this can be attributed to the fact that the silorane monomer is

hydrophobic, whereas the DX-511 monomer is longer and has a higher molecular mass.

FS has been assessed in several previous studies as a clinically relevant property for restorative materials meant to be used in areas that are exposed to force (SHIBASAKI et al., 2017, FINAN et al., 2013, YAMASAKI et al., 2013, GORACCI et al., 2014). The highest FS values measured in our study were for the resin composite SonicFill. The high filler load of SonicFill reportedly renders the composite capable of sustaining the functional stress; moreover, the application of sonic energy lowers its viscosity for improved interfacial adaptation (KAPOOR et al., 2016). Garoushi *et al*. (2013) noted the absence of a direct relationship between the volumetric content of filler and fracture parameters such as fracture toughness and FS of several commercial composites; they also claimed that other factors besides filler content (such as adhesion between matrix and filler particles, as well as stress transfer between these components) may play a relevant role (GAROUSHI et al., 2013). Kalore exhibited significantly lower FS than the other organic matrix content composites; however, this can be explained by its long monomer structure. The clinical use of SonicFill in high stress-bearing areas is feasible because of its superior mechanical characteristics. Even though SonicFill suffered a significant reduction in FS after aging, it still exhibited excellent performance that can be associated with the bulk-fill composite properties. Moreover, the polymerization shrinkage stress relaxation mechanism could also play a part in reducing the stress levels incurred by hydrolytic expansion.

The filler amount and types of organic matrix of resin composite materials correlate with the hardness of the material and alter the clinical properties, such as resistance to abrasion and polishability. Our study shows that SonicFill has the best values for Vickers hardness, which can be explained by its high inorganic filler content. Filtek Silorane displayed greater hardness than Kalore or Z550, because of its rigid hydrophobic monomer properties.

For all the samples in this study, the measured values of surface roughness (Ra) were less than 0.15 µm; this is less than the level that would be perceptible by the patient (0.3 µm) or even the minimum level needed for biofilm retention (0.2 µm) (BOLLEN et al., 1997). Therefore, with the use of 1.23% acidulated fluoride gel and surface sealant, the surface roughness alteration does not exceed the critical level, but remains within clinically acceptable limits.

Surface roughness is not the only parameter that affects aesthetic properties; gloss must also be considered. Gloss can be defined as reflectance of light from the surface. Smooth surfaces reflect more light, and thus, are associated with high gloss (HEINTZE et al., 2006). Inorganic fillers in composite materials play a significant role in determining the optical properties (LIM et al., 2008). Low-roughness composites such as microfilled resin-composites have been shown to possess higher gloss (O'BRIEN et al.,1984). As the surface roughness of a composite resin decreases, its glossiness properties will be increased (ATTAR et al., 2007). Increasing the gloss of a resin composite causes the material to look more aesthetic (LEE et al., 2005).

Composite resins can interact with the fluoride in APF agents through three pathways: the organic matrix, inorganic fillers, or the filler–matrix coupling agents. The hydrogen and fluoride ions in APF gel form hydrofluoric acid which attacks the inorganic filler particles, thereby decreasing the surface hardness of the composite material (YAP et al., 2002). Composites containing barium boroaluminosilicate glass are the most susceptible to attack by APF agents, while microfilled materials are the least sensitive to APF gel (SOENO et al., 2002). The thixotropic and viscosity characteristics of fluoride gels may also affect the surface properties of composites (SOENO et al., 2001).

Although the findings of this laboratory study showed that silorane resins presented good values of sorption and solubility, also a clinical study should be planned to decide effectiveness of silorane and dimethacrylate resins in a complete way.

CONCLUSIONS

The organic matrix can influence the WS and SL behavior of composite resins. Lower WS and SL rates are seen on materials fabricated from hydrophobic monomers.

Furthermore, high WS and SL degrade the mechanical and surface properties. Fluoridation and surface sealant application can alter the surface properties (hardness, roughness, and glossiness) but do not have any effect on the mechanical properties, such as flexural strength.

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RESUMO: Embora os clínicos utilizem agentes de flúor para reduzir a ocorrência de cáries e agentes de vedação de superfície para proteger restaurações compostas, os efeitos desses agentes sobre as resinas compostas ainda não foram investigados. O objetivo deste estudo foi determinar o efeito de diferentes aplicações de superfície (fluoreto ou selante de superfície) em resinas compostas com diferentes estruturas orgânicas (Siloranes, Sonicfill, 3M Z550, Kalore). Neste estudo, 120 discos e 120 barras de resina composta foram armazenados em água por três meses antes de serem envelhecidos termicamente por ciclos alternados entre 5 °C e 55 °C por 10.000 ciclos. Os discos tinham 15 mm de diâmetro e 1 mm de espessura; as barras eram 25 × 2 × 2 mm. O selante de superfície e o flúor foram aplicados nos espécimes e as avaliações foram realizadas após 24 horas. Cálculos iniciais e finais foram realizados para resistência à flexão, microdureza, rugosidade, brilho, sorção de água e solubilidade. O compósito de silorano apresentou os menores níveis de sorção de água ($p < 0,05$) para os grupos inicial e envelhecido. Os grupos compostos Silorane e SonicFill apresentaram a menor solubilidade ($p < 0,05$). Tanto antes quanto depois do envelhecimento, o grupo SonicFill apresentou os maiores valores de resistência à flexão e microdureza. Silorane apresentou a maior rugosidade e menores valores de brilho. Baixas taxas de sorção e solubilidade da água foram observadas em materiais fabricados a partir de monômeros hidrofóbicos. A alta sorção e solubilidade da água degrada as propriedades mecânicas e de superfície. A fluoretação e a aplicação de selante de superfície podem alterar as propriedades da superfície, mas não afetam as propriedades mecânicas.

PALAVRAS-CHAVE: Fluoreto de fosfato acidulado. Adesivos. Propriedades mecânicas. Resinas de silorano. Propriedades de superfície.

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