

Effect of Acidic Solutions on the Surface Degradation of a Micro-Hybrid Composite Resin

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Composite resins may undergo wear by the action of chemical substances (e.g., saliva, alcohol, bacterial acids) of the oral environment, which may affect the material's structure and surface properties. This study evaluated the effect of acidic substances on the surface properties of a micro-hybrid composite resin (Filtek Z-250). Eighty specimens were prepared, and baseline hardness and surface roughness (KMN_0 and Ra_0 , respectively) were measured. The specimens were subjected to sorption (SO) and solubility (SL) tests according to ISO 4049:2009, but using different storage solutions: deionized water; 75/25 vol% ethanol/water solution; lactic acid; propionic acid; and acetic acid. The acids were used in two concentrations: PA and 0.02 N. pH was measured for all solutions and final hardness (KMN_f) and surface roughness (Ra_f) were measured. Data were analyzed with paired t-tests and one-way ANOVA and Tukey's test ($\alpha=5\%$). All solutions decreased hardness and increased the Ra values, except for the specimens stored in water and 0.02 N lactic acid, which maintained the hardness. All solutions produced similar SO and SL phenomena, except for the 0.02 N lactic acid, which caused lower solubility than the other solutions. Ethanol showed the highest pH (6.6) and the 0.02 N lactic acid the lowest one (2.5). The solutions affected negatively the surface properties of the composite resin; in addition, an acidic pH did not seem to be a significant factor that intensifies the surface degradation phenomena.

Key Words: composite resin; organic acids; surface roughness; microhardness; sorption and solubility.

Introduction

Dental composite resins had their properties and characteristics substantially improved since they were introduced in dentistry (1), leading to an excellent acceptance by the dentists who can use them for a variety of clinical procedures. They are basically constituted by an organic matrix and inorganic fillers chemically connected by a silane substance. Regarding the organic matrix, which is mostly formulated with dimethacrylate monomers such as 2,2-bis[4-(2-hydroxy-3-methacryloyloxypropyl)phenyl]propane (Bis-GMA), triethyleneglycol dimethacrylate (TEGDMA), and urethane dimethacrylate (UDMA), it constitutes the body of the composite and may influence the handling ability and properties of the material; on the other hand, silica and glass fillers are commonly used to modify esthetic, physical and mechanical characteristics of composites (1).

In contact with the oral environment, composite resins undergo wear and are affected by chemical substances in their surroundings, acting superficially on the material's structure. Even though a great variety of substances may be present at the oral environment, water, saliva, acids, bases, salts, and alcohols have been related to the reduction of hardness, flexural strength and flexural modulus properties (2,3). In addition, the biofilm accumulated

over the restoration can produce acidic substances that may imply surface degradation, leading to the material's softening and surface roughening (4,5). With regard to these acidic substances, the lactic, propionic and acetic acids are commonly found in the oral environment and they are used as storage solutions for screening accelerated hydrolysis phenomena of composite resins (6) and increase of hygroscopic expansion of Bis-GMA-based materials.

Even though chemical substances may affect more actively the organic matrix of composites, the type, size, and concentration of fillers may also influence the material's resistance to degradation. Considering this, micro-hybrid composites present a mixture of silica and glass fillers which vary between 0.4 to 1 μm in size and present a concentration of approximately 66 wt%. They are indicated for the restoration of both anterior and posterior teeth, resulting in excellent mechanical and esthetic properties, in addition to good polishability (1). Though, since dimethacrylate-based composites undergo degradation in contact with different substances found in the oral cavity, it is important to evaluate the influence of these substances on the surface properties of micro-hybrid composites, and also to evaluate whether their pH affects the degradation potential imposed to the material.

The aim of this *in vitro* study was to evaluate the

effect of acidic solutions on the superficial properties of a micro-hybrid composite resin testing the variation of surface roughness, hardness, and sorption and solubility. The first hypothesis tested was that the pH is determinant of the composite's surface aggressiveness; and the second hypothesis tested was that the most acidic solutions affect more negatively the evaluated properties.

Material and Methods

Specimen Preparation and Group Allocation

The micro-hybrid composite resin Filtek Z-250™ (3M ESPE, St. Paul, MN, USA) was investigated in this study. Eighty disc-shaped specimens (6 mm in diameter x 1 mm thick) were prepared using a Teflon mold. The composite was placed in a single increment and covered with a glass-slide that was pressured to extrude excess material. Then, each specimen was photo-activated with a light-emitting

diode curing unit (LED Radii; Bayswater, VIC, Australia) for 20 s on the top and bottom surfaces and polished with #600- and #1200-grit silicon carbide abrasive papers.

After their preparation, all specimens were randomly allocated into eight groups according to the storage solution (n=10): deionized water (Water); 75/25 vol% ethanol/water solution (Ethanol); 85 vol% lactic acid (LA PA); 0.02 N lactic acid (LA 0.02 N); 99 vol% propionic acid (PA PA); 0.02 N propionic acid (PA 0.02N); 99 vol% acetic acid (AA PA); or 0.02 N acetic acid (AA 0.02N). The molecular formula and molecular structure of each solution is shown in Table 1.

Baseline Surface Analysis

Before storage in the solutions, hardness and surface roughness measurements were obtained for each specimen. Hardness was evaluated using a microhardness tester (FM 700; Future Tech, Chung Ho, Taipei Hsien, Taiwan), with a 50 g load and a dwell time of 15 s. Three indentations were made across the center of each specimen, and the Knoop Microhardness Number (KMN) was obtained. The baseline mean hardness value was classified as KMN_0 . Surface roughness was measured by a single evaluator who rotated the specimens over the surface profilometer (Surfcorder SE1200; Kosaka Lab., Tokyo, Japan) clockwise at 3, 6 and 9 h angles. The mean roughness parameter (R_a , μm) for each specimen was recorded as the average of three readings. This baseline roughness measurement was classified as R_{a0} .

pH Measurement

The pH of all the solutions was verified by a digital pHmeter (PM 608 Plus; Analion, Ribeirão Preto, SP, Brazil) adding 20 μL of each solution in glass vessels, except of the PA solutions, which could not be measured as they had no water to ionize the acidic molecules. All the pH measurements were performed three times at 23 °C and 50% relative humidity.

Sorption and Solubility Analysis

After baseline measurements, all specimens were prepared for a sorption and solubility test, which was performed following the ISO 4049:2009 standard (7) and placed in a desiccator at 37 °C containing freshly dried silica gel and calcium chloride. After 24 h, the samples were removed, stored in a desiccator at 23 °C for 1 h and weighed on a scale with a 0.01 mg precision (AUW 220D; Shimadzu Corp. Nakagyo-ku, Kyoto, Japan). This cycle was repeated until a constant mass (m_1) was obtained. Thickness and diameter of the specimens were measured at five points using a digital caliper (0.01 mm precision), rounded off to the nearest, and these measurements were used to calculate the volume (V) of each specimen (in mm^3).

Table 1. Storage solutions used in this study and their molecular formula and molecular structure

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Storage solution	Molecular formula	Molecular structure
Deionized water (Water)	H_2O	
75/25 wt% ethanol/water (Ethanol)	$\text{C}_2\text{H}_6\text{O} / \text{H}_2\text{O}$	
85 wt% lactic acid (LA PA)	$\text{C}_3\text{H}_6\text{O}_3$	
0.02N lactic acid (LA 0.02N)	$\text{C}_3\text{H}_6\text{O}_3 / \text{H}_2\text{O}$	
99 wt% propionic acid (PA PA)	$\text{C}_3\text{H}_4\text{O}_2$	
0.02N propionic acid (PA 0.02N)	$\text{C}_3\text{H}_4\text{O}_2 / \text{H}_2\text{O}$	
99 wt% acetic acid (AA PA)	$\text{C}_3\text{H}_6\text{O}_2$	
0.02N acetic acid (AA 0.02N)	$\text{C}_3\text{H}_6\text{O}_2 / \text{H}_2\text{O}$	

Next, the specimens were immersed in the solutions at 37 °C for 7 days, then removed, blotted dry and weighed (m_2). After weighing, they were dried again inside the desiccators and weighed daily to record a third constant mass (m_3) (as previously described). For each sample, the data of sorption (SO) and solubility (SL) were calculated using the following formula:

$$SO = (m_2 - m_3)/V \text{ and } SL = (m_1 - m_3)/V$$

Hardness and Surface Roughness Variation

After the SO and SL evaluation, each group was measured again for the hardness and surface roughness tests, as aforementioned; so, the final mean hardness (KMN_1) and mean roughness (Ra_1) were obtained, and the variation between the baseline and the final measurements were calculated as follows:

$$\Delta KMN = KMN_0 - KMN_1; \text{ and } \Delta Ra = Ra_1 - Ra_0.$$

Statistical Analysis

The hardness and surface roughness data (baseline and final measurements) were analyzed by paired t-tests. Data from hardness and surface roughness variation (ΔKMN and ΔRa , respectively) and the sorption and solubility data were all analyzed by one-way ANOVA and Tukey as a *post hoc* test. A significance level of 5% was set for all analyses.

Results

Hardness and Surface Roughness

The baseline and final hardness and surface roughness mean values are graphically demonstrated in Figures 1 and 2, respectively. With the hardness results,

the storage solutions degraded the material, since the specimens became significantly softer, except for those immersed in deionized water and in lactic acid 0.02 N ($p=0.084$ and $p=0.687$, respectively) (Fig. 1). Regarding the ΔKMN results, propionic acid PA caused the highest variation, but statistically similar to the other solutions, except for the 0.02 N lactic acid, which showed the lowest ΔKMN mean value ($p<0.001$) (Table 2). With regard to the surface roughness evaluation, all storage solutions produced rougher surfaces on the specimens ($p<0.001$) (Fig. 2). However, the ΔRa results did not vary among the groups, ranging from 0.04 to 0.06 μm (AA PA and PA 0.02 N groups, respectively) (Table 2).

Sorption and Solubility

The sorption and solubility results are demonstrated in Table 2. All the solutions provided similar sorption effect to the specimens ($p=0.484$); in contrast, while the 0.02 N

Table 2. Surface roughness (ΔRa - μm) and microhardness (ΔKMN) variation, sorption (SO) and solubility (SL) data in $\mu\text{g}/\text{mm}^3$, standard deviation (SD) and pH of the solutions evaluated.

Group	ΔKMN	ΔRa	SO	SL	pH
Water	10.5 ^{AB}	0.05 ^A	22.5 (4.7) ^A	0.7 (8.9) ^{AB}	4.6 (0.6)
Ethanol	14.4 ^{AB}	0.05 ^A	25.6 (7.8) ^A	-3.4 (5.6) ^{AB}	6.6 (0.1)
LA PA	15.3 ^{AB}	0.05 ^A	26.7 (13.9) ^A	-0.3 (10.1) ^{AB}	-
LA 0.02N	- 1.9 ^B	0.06 ^A	20.5 (6.1) ^A	-13.3 (12.3) ^B	2.5 (0.1)
PA PA	20.7 ^A	0.04 ^A	19.0 (10.1) ^A	4.1 (13.0) ^A	-
PA 0.02N	15.9 ^{AB}	0.05 ^A	23.5 (7.5) ^A	-1.0 (9.5) ^{AB}	3.0 (0.1)
AA PA	16.5 ^{AB}	0.05 ^A	23.2 (6.0) ^A	-6.5 (9.1) ^{AB}	-
AA 0.02N	12.9 ^{AB}	0.05 ^A	26.6 (11.9) ^A	1.7 (10.1) ^A	3.01 (0.1)

Different superscripts capital letters in a same column represent statistically significant differences among the storage solutions ($p<0.001$).

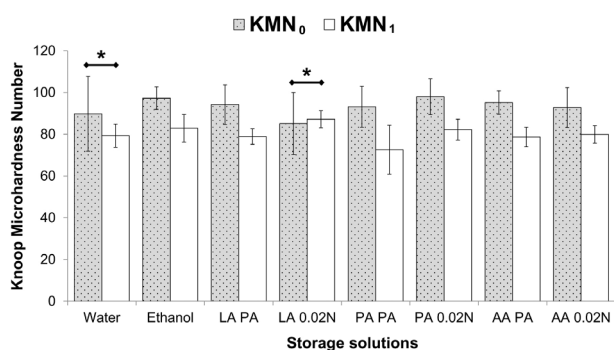


Figure 1. Mean hardness values of the composite resin before (KMN_0) and after (KMN_1) storage in deionized water (Water), 75/25 vol% ethanol/water solution (Ethanol), 85 vol% lactic acid (LA PA), 0.02 N lactic acid (LA 0.02N), 99 vol% propionic acid (PA PA), 0.02 N propionic acid (PA 0.02N), 99 vol% acetic acid (AA PA), and 0.02 N acetic acid (AA 0.02N). The horizontal line with asterisk (*) indicates similarity between the baseline and the final measurements ($p>0.05$).

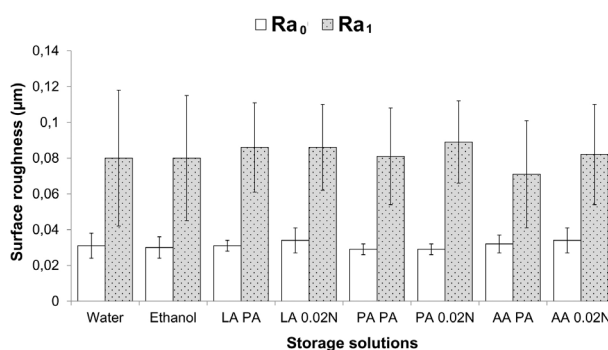


Figure 2. Mean surface roughness values of the composite resin before (Ra_0) and after (Ra_1) storage in deionized water (Water), 75/25 vol% ethanol/water solution (Ethanol), 85 vol% lactic acid (LA PA), 0.02 N lactic acid (LA 0.02N), 99 vol% propionic acid (PA PA), 0.02 N propionic acid (PA 0.02N), 99 vol% acetic acid (AA PA), and 0.02 N acetic acid (AA 0.02N). All solutions increased significantly the surface roughness of the composite resin ($p<0.001$).

lactic acid caused the lowest solubility result in the study, the propionic acid PA and the 0.02 N acetic acid showed the highest mean solubility values.

pH Measurement

Table 2 shows the pH values of the evaluated solutions. The ethanol solution presented the highest pH in the study (6.6); deionized water showed a pH of 4.6. The 0.02 N acidic solutions diluted in water presented the lowest pH values, and the lactic acid was the most acidic solution (pH=2.5).

Discussion

Several studies have demonstrated that acidic substances have a potential of degrading composite resin materials (5,8-10). Thus, one expects that any solution with low pH would increase the surface degradation processes, being more aggressive than the less acidic solutions. Nevertheless, the first study hypothesis that the pH of the storage solutions would determine the aggressiveness of the solutions on the composite's surface was totally rejected, as even the lowest and the highest pH values altered the composite's superficial properties in a similar fashion, suggesting that other factors are influencing more the surface and structure degradation of resin-based materials.

According to Bagheri et al. (11), the type of storage solution and the composition of the soaked material are important factors for the degradation of composite materials. On the other hand, factors such as the solubility parameter [from both the storage solution (solvent) and the polymeric material (solute)], the cross-link nature of the resin matrix, and the solvent sorption uptake may influence more directly the polymer's degradation rate (12). Consequently, the combination of these factors may affect the wear undergone by the restorative material and its durability.

Independent from the factors involved in superficial degradation processes, data analysis revealed that the acidic solutions altered the hardness and surface roughness of the investigated composites (Figs. 1 and 2). These solutions were chosen because they can be used as ageing simulators, with water simulating the wet oral environment (9), the ethanol-water solution representing the medium of choice for accelerated ageing (9,13), and the organic acids simulating the substances that may be produced by dental biofilm (4,13-15), which would be in intimate contact with the restorative material's surface. Hardness is defined as the resistance of a material to permanent deformation, tested by means of an indentation, and is related to the microstructure and composition of the composite resin (16,17). Therefore, any factor affecting the resin matrix arrangement, the matrix/filler interaction or the filler particles, leading to leaching of these components, causes

a significant reduction on its surface properties, including hardness.

The results showed that all the solutions, except deionized water and 0.02 N lactic acid, reduced the hardness of specimens (Fig. 1). According to Lee et al. (4), 75% ethanol solution presents a close solubility parameter [$[(3.12 \times 10^{-4} \text{ J/m}^3)^{1/2}]$ (18) to Bis-GMA-based materials (e.g.: Filtek Z-250™), and the closer the solubility parameter between solvent and solute, the greater the solute's degradation (14). In fact, and according to Wu and McKinney (19) the surface of any Bis-GMA-based material would be softened by solvents with solubility parameter ranging from 1.82 to $2.97 \times 10^{-4} (\text{J/m}^3)^{1/2}$. Regarding the organic acids used in the present study, the propionic and the acetic acids have solubility parameter similar to ethanol (4) and, as a consequence, they resulted in reduction of hardness. On the other hand, deionized water and 0.02 N lactic acid maintained stable the hardness of specimens, probably because both of them have a solubility parameter which is not similar to Bis-GMA-based materials. These results corroborate a previous study that found that propionic and acetic acids softened the evaluated composites, while water and lactic acid solutions did not affect hardness (4).

Unlike hardness, all the solutions increased surface roughness of specimens (Fig. 2). Surface roughness measures the material's topography and depends on several factors, namely the type of fillers, their size, shape and concentration (20), the resin matrix conversion percentage (21), the filler-matrix interaction (9,21), and the hardness of the material (22). In this study, these factors were kept the same; thus, the differences obtained are specifically due to the effect of the solutions on the material's surface and structure. Several studies have demonstrated that acidic solutions increase the roughness of composites, probably because they soften their surface, leading to the leachability of resin components and, in consequence, to the displacement of filler particles, contributing to the formation of a rough surface (8,23,24). In fact, all the solutions that softened the composite resin also increased its surface roughness (Fig. 2), although there were no significant differences among the roughness variation data (Table 2). However, even though deionized water and 0.02 N lactic acid did not alter the specimen's hardness significantly (softening effect), the specimen's surface roughness was increased, a fact that may be explained by the sorption and solubility phenomena (14).

Sorption and solubility are related to the solvent entrance into the resin matrix, which may alter its structure by swelling and plasticization processes, and finally to the release of non-reacted components and byproducts of the polymer network (14). In the present study, deionized water and ethanol solution were used as positive and

negative controls, respectively. Even though water was less deleterious to composite as it did not significantly reduce hardness, Filtek Z-250 was sensible to water uptake, probably due to the presence of TEGDMA in its composition, which is hydrophilic and may imply in high sorption. Nevertheless, all solutions showed similar sorption results (Table 2), probably because this characteristic is more material-dependent, that is, more influenced by the hydrophilicity and the cross-linking nature of the resin matrix, than by the type of solvent (25). Considering that the same composite resin was evaluated, all solvents were able to cause similar sorption effect. It is also important to observe that all sorption values of this study were lower than the reference values of ISO 4049 (7).

With regard to solubility, it is possible to observe that all the storage solutions caused a low solubility effect (Table 2) also lower than the reference value of ISO 4049, demonstrating that the composite resin was structurally stable after the immersion procedures. Interestingly, the LA 0.02 N group showed a high negative solubility value (additional to the highest standard deviation value obtained) when compared with the other groups. This indicates that some lactic acid molecules, which are constituted by a pendant hydroxyl polar group (Table 1), have probably interacted by hydrogen bonds with the composite structure, resulting in reduced release of by-products (products of solvent degradation or released by the polymer's structure) (13). This may also explain why the 0.02 N lactic acid did not reduce the microhardness of the composite resin (Fig. 1), showing the lowest Δ KMN (Table 2).

Another purpose of the present study was to investigate whether the most acidic solutions would affect more negatively the evaluated properties (second tested hypothesis). Comparing the findings of the study with each other it is possible to detect that the 0.02 N lactic acid showed the lowest pH value and equally the lowest aggressiveness to the material's structure: same hardness before and after the immersion procedure, same sorption and lower solubility effects when compared with the other solutions, and similar surface roughness variation (Figs. 1 and 2, and Table 2). Thus, the second tested hypothesis was also rejected. According to a recent study (6), which investigated the effects of neutral and extremely acidic or alkaline pH storage media on dental resin surface properties, the alkaline pH was more aggressive to hardness and surface roughness properties, while the evaluated composites seemed to withstand the neutral and acidic media environments.

The evaluated micro-hybrid composite resin had its surface properties influenced by the storage solutions where they were immersed. In addition, the 0.02 N lactic acid presented the most acidic nature, but reduced surface

aggressiveness. Therefore, a low pH value does not seem to be a significant factor affecting the surface degradation of the evaluated composite resin. However, further investigations evaluating the effect of these solutions on mechanical and surface properties of composite resin materials containing different types, sizes and content of fillers are required.

Resumo

Resinas compostas podem sofrer desgaste pela ação de substâncias químicas (saliva, álcool, ácidos bacterianos) presentes no ambiente oral, que podem afetar a estrutura e as propriedades superficiais do material. Assim, este estudo avaliou o efeito de substâncias ácidas nas propriedades superficiais de uma resina composta micro-híbrida (Filtek Z-250). Oitenta espécimes foram preparados e mensurados quanto à dureza e rugosidade superficial iniciais (KMN_0 e Ra_0 , respectivamente). Os espécimes foram submetidos a testes de sorção (SO) e solubilidade (SL) de acordo com a ISO 4049:2009, porém usando diferentes soluções de armazenamento: água deionizada, solução etanol/água 75/25% em volume, ácido láctico, ácido propiônico e ácido acético. Os ácidos foram utilizados em duas concentrações: PA e 0,02 N. O pH foi mensurado para todas as soluções e a dureza e rugosidade superficial finais (KMN_1 e Ra_1 , respectivamente) foram mensuradas. Os dados foram analisados com testes t-pareado e ANOVA a um fator, e teste de Tukey ($p < 0,05$). Todas as soluções reduziram a dureza e aumentaram os valores de Ra, exceto para os espécimes armazenados em água e ácido láctico 0,02 N, que mantiveram a dureza. Todas as soluções produziram fenômenos de SO e SL similares, exceto o ácido láctico 0,02 N, que causou menor solubilidade do que as outras soluções. Etanol mostrou o maior pH (6,6) e o ácido láctico 0,02 N o menor deles (2,5). As soluções afetaram negativamente as propriedades superficiais da resina composta; além disso, um pH ácido não parece ser um fator significativo para intensificar os fenômenos de degradação superficial.

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Received April 16, 2014

Accepted July 28, 2014