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Efeito da Degradação Química sobre a Rugosidade Superficial e Estabilidade de Cor de Resinas Compostas Micro-híbrida e Nanoparticulada

Effect of Chemical Degradation on Surface Roughness and on Color Stability of Micro hybrid and Nanofilled Composites

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Resumo

A resina composta é o material restaurador mais utilizado atualmente. Apesar de seu sucesso clínico, o material está sujeito a alterações em suas propriedades físicas, quando exposto ao meio bucal. O objetivo foi avaliar a influência da degradação química na estabilidade de cor e na rugosidade superficial de resinas micro-híbrida e nanoparticulada. Foram confeccionados 40 discos (6,0mm x 2,0mm) da resina composta micro-híbrida (Z250XT) e 40 da resina nanoparticulada (Z350XT). Após a análise da cor inicial e da rugosidade inicial, 10 discos de cada resina foram imersos em saliva artificial (Controle, pH=6,4), suco de laranja (pH= 3,4), refrigerante de limão (pH=2,9) e vinho tinto (pH=3,1), por 4 horas por dia (37ºC) durante 30 dias. Após, foram realizadas as leituras finais de cor e rugosidade. Os dados obtidos para a estabilidade de cor foram analisados pelo Teste de Kruskal-Wallis, seguido pelo Teste de Dunn (α=5%). Para a análise da rugosidade foi utilizado o Teste t para amostras pareadas (α=5%). O manchamento provocado pela saliva artificial, suco de laranja e refrigerante foi similar, mas foi estatisticamente diferente do manchamento produzido pelo vinho, em ambas as resinas testadas. O vinho produziu o mesmo grau de manchamento nas resinas micro-híbrida e nanoparticulada. Foi observado que o suco de laranja, o refrigerante e o vinho promoveram aumento da rugosidade superficial dos discos de resina. Diante disso, concluiu-se que o vinho alterou a cor de resinas compostas micro-híbrida e nanoparticulada, e que a rugosidade superficial dos discos de resina foi alterada por todas as bebidas utilizadas.
Palavras chave: Resinas Compostas. Corantes. Cor.

Abstract

Composite resin is the most used restorative material. Despite its clinical success, it is susceptible to changes in its physical properties when exposed to the oral environment.
The aim was to evaluate the influence of acidic beverages on the color stability and on the surface roughness of the nanofilled and micro hybrid composite. Forty discs (6.0mm x 2.0mm) of the micro hybrid composite (Z250XT) and 40 of the nanofilled composite (Z350XT) were manufactured. After the measurement of the initial color and the initial roughness, 10 discs of each composite were individually immersed in artificial saliva (control, pH = 6.4), orange juice (pH=3.4), lime soda (pH = 2.9) and red wine (pH = 3.1) during 4 hours per day at 37°C for 30 days. At the end of the experiment, the final analysis of color and surface roughness were performed. The data obtained for the color stability were analyzed by Kruskal-Wallis and Dunn's test (α= 5%). For the analysis of the surface roughness, the t test for related samples was used (α= 5%). The color changes promoted by artificial saliva, orange juice and soda were similar, but they were statistically different from staining produced by wine, in both tested composites. Wine produced the same degree of staining in both composites. Orange juice, soda and red wine increased the surface roughness of the composite discs. It was concluded that red wine significantly changes the color of micro hybrid and nanofilled composites. Furthermore, all beverages increased the surface roughness of the composites tested in the study herein.
Keywords: *Composite Resins. Coloring Agents. Color.*

1 Introduction

The use of composite resins has turned into an important reality in restorative dentistry. Currently, these composites are among the most popular aesthetic restorative materials in dental practice, due to improvements in their physical, mechanical and aesthetic properties[1]. Despite the great advances in respect to its composition and its properties, these materials still suffer chemical and structural changes due to numerous adverse conditions in the oral environment, which can be detrimental to the integrity of the composite over time[2]. Among these conditions, one can mention the chemical challenges that these materials are subjected to in the oral cavity, such as the ingestion of acidic drinks. These substances can cause changes in material properties, such as: the wear of the organic matrix and the displacement of the particles of inorganic load, resulting in the formation of gaps, which make the irregular surface, favoring the accumulation of biofilm and pigmentations leading, consequently, to the impairment of the longevity of the restorations[3].

Many studies have reported that drinks that compose the diet of individuals, such as: coffee, tea, grape juice, yerba mate and sodas cause harmful effects in dental composites due to the staining of the surface of the same area related mainly with the absorption or adsorption of coloring substances found in these drinks[4]. In addition to changing color, some of these products can also cause degradation in the surface of the composite due to the reduction of its hardness[5]. In addition, the size, the type and quantity of particles of composite load, as well as the composition of the organic matrix of resin play an important role in this context[6].

With the passing of the years, several modifications were being introduced in particulate load of the composites, more precisely in the composition, size, shape and the amount of the inorganic particles. Despite of that, not only the organic phase but also the inorganic phase has an influence on the material behavior, the characteristics inherent to load particles are directly related to the improvement of the mechanical properties of composite resins[6]. Studies have shown that the composites known as nanofilled have advantages when compared to the micro-hybrid resins, such as: lower contraction of polymerization, better mechanical properties, better brightness, prolonged maintenance of the smoothness of the surface and less wear[7]. The nanofilled resins have a combination of particles of silica and zirconia with size that varies between 4-20 nm and Nano agglomerates with 0,6-10 micrometers. This allows these particles to be distributed along the organic matrix, in a uniform manner, allowing to occur an increase in the quantity of particles of load of material, taking the greatest percentage in volume of the inorganic phase (approximately 59.5%), which would be responsible for the improvement of the mechanical properties of these resins[6].

Despite the advantages conferred to the nanofilled composites, the literature brings no conclusive results on their superiority before the micro-hybrid composites, mainly, to the factors that can lead to staining of the restorations. Thus, the objective of this study was to analyze and compare the surface roughness (Ra) and color change of a micro-hybrid resin and a nanofilled resin subjected to chemical challenge.

2 Material and Methods

2.1 Preparation of Specimens

The composite resins (Table 1) were handled according to the manufacturer's instructions and inserted into an array of metallic cylindrical stainless steel (6.0 mm diameter x 2.0 mm thickness/height), performing insertion in a single increment.

Table 1 - Composite resins tested in the present study*

Composite (Color)	Composition of the resin matrix (Load)	Mean size of load particles	Load percentage	Batch
Filtek Z250 XT (A3)	Bis-GMA UDMA Bis-EMA zirconia/sílica	0.6μm	82% weight 60% volume	44465
Filtek Z350 XT (A3)	BisGMA, UDMA TEGDMA BIS-EMA PEGDMA	4-20nm	78.5% weight 63. 3%volume	984521

* In accordance with the manufacturer.
Source: Research data.

Immediately after the insertion of the material, the matrix filled with the resin was covered by a strip of polyester and, on this, a glass slide was placed on which an axial load of 500g was applied, during 1 minute to compress the composite resin, making the surface flat and with standardized thickness. The material photoactivation (Radii-cal, SDI Dental Product SDI, Bayswater, Victoria, Australia) was carried out through the glass slide by means of blue light for 20 seconds. The intensity of visible light (1,200 mW/cm^2) was monitored through a radiometer (RD7, Ecel Indústria e Comércio Ltda, Ribeirão Preto-SP). Prior to polishing, the specimens were kept in distilled water and stored in an oven at 37 ºC.

After 24 hours, the resin discs were subjected to the finishing with Sof-Lex discs Pop On (medium and fine granulation) (3M ESPE, Sumaré-SP, Brazil). This polishing was performed to simulate a clinical situation. At the end of these procedures, the specimens were washed with distilled water for 30 seconds, dried with absorbent paper and immersed in artificial saliva for over 24 hours in an oven at 37 ºC for then perform the initial measurements of surface roughness and color. So that the readings of color and surface roughness were standardized, a marking on the end of the sample was performed with a spherical rotatory sharp instrument rotatory #1012. Thus, the sample was always positioned in the equipment of analysis with this marking pointing upwards. 10 specimens were obtained per group. Each specimen was identified and distributed according to Table 2.

Table 2 - Division of experimental groups

Group	Composite resins.	Cycling solution	pH	Number of samples
1	Filtek Z250 XT	Artificial Saliva	6.4	10
2	Filtek Z250 XT	Orange Juice	3.4	10
3	Filtek Z250 XT	Soft Drink	2.9	10
4	Filtek Z250 XT	Red Wine	3.1	10
5	Filtek Z350 XT	Artificial Saliva	6.4	10
6	Filtek Z350 XT	Orange Juice	3.4	10
7	Filtek Z350 XT	Soft Drink	2.9	10
8	Filtek Z350 XT	Red Wine	3.1	10

Source: Research data.

2.2. Evaluation of initial surface roughness (Ri)

The specimens were evaluated according to their pattern of surface roughness, by means of the roughness Surfcorder IF1700 (KosakaLaboratoryLtd. Tokyo – 101-0021 – Japan). The sensing tip of 2 µm in diameter, exerting contact pressure of 0,7mN, performed traces at a speed of 0.5 mm/s, totaling 12 readings per specimen. The length of sampling or cut-off used was 0.08mm. Measurements were performed using the parameter that is based on vertical measurement (Ra/µm), of the surface roughness. 3 readings were performed per specimen. The average of 3 readings was used as the measure of each specimen, considered initial roughness (Ri).

2.3 Analysis of the initial color

The initial color of specimens in accordance with the CIELab system, with standard lighting D65 on a white background with the colorimetric spectrophotometer was analyzed (Color guide 45/0, PCB 6807 BYK-Gardner GmbH. Gerestsried – 82538 – Germany). The pattern of each specimen was measured by examining the coordinates (L*, a* and b* of the CIELab system. Before each reading, the specimens were washed with distilled water for one minute and dried with absorbent paper.

2.4 Cycling in the solutions

The specimens were randomly divided into four groups: the control group was maintained in artificial saliva and the three experimental groups were submitted to nutrient cycling in artificial saliva (sodium chloride, potassium chloride, sorbitol, nipagin, carboxymethyl cellulose (CMC) and distilled water; Pharmacy AcquaBella. Uberaba - MG - Brazil), orange juice (water, concentrated orange juice, Xanthan Gum, sugar, natural aroma, acidulant citric acid and antioxidant ascorbic acid, Tropical Indústria de Alimentos SA Visconde do Rio Branco - MG - Brazil), Lemon soda (Sprite- carbonated water, sugar, lemon juice, natural flavoring, citric acidulant, preservatives sodium benzoate and potassium sorbate; Uberlândia Refrescos Ltda. Uberlândia - MG, Brazil), and red wine (fermented grapes, sugar and preservatives potassium sorbate and sulfur dioxide; Antonio Basso e Filhos Ltda. Flores da Cunha - RS - Brazil).

The specimens were immersed daily in their respective solutions (1.5 mL/specimen) for 4 hours, for 30 days. For the control group, the specimens were maintained at 37 ºC by exchanging the artificial saliva every two days. These procedures were repeated for 30 days.

The specimens were kept immersed in artificial saliva at 37° C ± 1° C in the interval among the cycles. The beverages were used at a consumption temperature, i.e., soda and orange juice were kept in a refrigerator to ± 4 ºC, and red wine at room temperature ± 25 ºC.

2.5 Evaluation of the final surface roughness (Rf)

The specimens were evaluated again as to their pattern of surface roughness at the end of 30 days (Rf). The response variable was the difference in the final surface roughness less the initial (Rf - Ri).

2.6 Analysis of the final color

The color was measured again at the end of the cycling, with the same equipment used for the reading of the initial color. The color difference was obtained by calculating $\Delta Eab^* = [(\Delta L^*)^2 + (\Delta^*)^2 + (\Delta b^*)^2]1/2$. The difference in brightness (ΔL), Δa and Δb was calculated by the formula $\Delta L^* = l^*(t) - L^*(0)$ $\Delta^* = ^*(t) - a^*(0)$ and $\Delta b^* = b^*(t) - b^*(0)$, in which (t) corresponds to the time and (0) corresponds to the baseline. The color change was analyzed by the values of Δe, ΔL, Δa and Δb.

2.7 Statistical Analysis

The statistical data were analyzed with the aid of the software Bioestat 5.3 (Mamirauá Sustainable Development Institute, Tefé-AM, Brazil). For the statistical analysis of the change in the color of the samples were used in the Kruskal Wallis test, followed by Dunn's test. For the data analysis of surface roughness T-test was used for the related samples. The significance level adopted was 5%.

3 Results and Discussion

Table 3 shows the variation of color (ΔE) of specimens obtained with the resins Z250 and Z350 XT, after 30 days of immersion in artificial saliva, orange juice, soda or red wine. The discs of resin Z250 XT immersed in orange juice (1.6 ±0.47) and in the soda (1.7 ±0.64) showed the same variation of color than the samples from group 1 (2.3 ±0.67) ($p>0.05$). Since the samples immersed in red wine (12.8 ±4.32) showed greater variation of color than the other groups, for the same resin evaluated ($p<0.05$). The samples of resin Z350 XT showed similar behavior to the samples of the micro-hybrid resin. Thus, the discs of resin Z350 XT immersed in orange juice (1.76 ±0.62) and in the soda (1.54 ±0.32) showed the same variation of color than the samples from group 1 (2.3 ±0.66) ($p>0.05$). Whereas the samples immersed in red wine (13.61 ±4.24) showed greater variation of color than the other groups, for the same resin evaluated ($p<0.05$).

Table 3 - Mean (± standard deviation) of the change of color (ΔE) presented in each group

Storage solutions	Values of ΔE	
	Z250 XT	Z350 XT
Artificial Saliva	$2.3(\pm0.67)^{a,A}$	$2.3(\pm0.66)^{c,A}$
Orange Juice	$1.6(\pm0.47)^{a,A}$	$1.7(\pm0.62)^{c,A}$
Soft Drink	$1.7(\pm0.64)^{a,A}$	$1.5(\pm0.32)^{c,A}$
Red Wine	$12.8(\pm4.32)^{b,A}$	$13.6(\pm4.24)^{d,A}$

* Lowercase compare the data within a same column and letters compare the lines. Different letters indicate the presence of statistically significant differences ($p<0.05$)

Source: Research data.

Regarding the comparison of staining of the same solution, for the different composite resins tested, it was observed that the staining produced by each solution was the same for the two tested resins (p>0.05).

Table 4 shows the comparison between the surface roughness of composite resin Z250 XT before and after immersion in the tested solutions. The artificial saliva was the only solution that showed no significant changes in the surface roughness of composite resin. Whereas the orange juice (0.332 ±0.57; p=0.0412), the soda (0.289 ±0.37; p=0.0170) and the red wine (0.447 ±0.30; p=0.0007) increased significantly, the roughness of the composite resin after 30 days of cycling.

Table 4 - Mean Values (± standard deviation) of the initial surface roughness (Ra), final and the roughness variation (ΔRa) of samples of resin Z250 XT in each group

Storage solutions	Ra initial	Ra final	ΔRa	t	p
Artificial Saliva	0.631 (±0.21)	0.706(±0.23)	0.075 (±0.26)	-1.817	0.0692
Orange Juice	0.655(±0.54)	0.988(±0.35)	0.332 (±0.57)	-1.910	0.0412
Soft Drink	0.553(±0.23)	0.842(±0.36)	0.289 (±0.37)	-2.465	0.0170
Red Wine	0.457(±0.16)	0.904(±0.24)	0.447 (±0.30)	-4.589	0.0007

Source: Research data.

Table 5 shows the comparison between the surface roughness of composite resin Z350 XT before and after immersion in the tested solutions. Similarly, to what was observed for micro-hybrid, the artificial saliva was the only solution that showed no significant changes in the surface roughness of composite resin. Whereas the orange juice (0.439 ±0.32; p=0.0011), the soda (0.204 ±0.11; p=0.0066) and the red wine (0.271 ±0.16; p=0.0479) increased significantly, the roughness of the composite resin after 30 days of cycling.

Table 5 - Mean Values (± standard deviation) of the initial surface roughness (Ra), final and the roughness variation (ΔRa) of samples of resin Z350 XT in each group

Storage solutions	Ra initial	Ra final	ΔRa	t	p
Artificial Saliva	0.739 (±0.27)	0.784(±0.34)	0.045 (±0.1)	-1.623	0.0734
Orange Juice	0.519 (±0.12)	0.959(±0.31)	0.439(±0.32)	-4.235	0.0011
Soft Drink	0.630(±0.16)	0.835(±0.3)	0.204(±0.11)	-3.075	0.0066
Red Wine	0.755(±0.39)	1.026(±0.26)	0.271(±0.16)	-1.859	0.0479

Source: Research data.

There are extrinsic and intrinsic factors which influence the stability of the color of composite resins. The extrinsic factors, which may be taken into consideration, in the present study are the pigments present in the solutions that the resin discs were subjected to, and the intrinsic factors are related with the composition of the resins (organic matrix and load particulates)[8]. In the present study, an analysis was made of the influence of different means of immersion on the change of color of two distinct composite resins, being a resin micro-hybrid (Filtek Z250 XT) and another nanofilled resin (Filtek Z350 XT). The discs of composite resin were immersed in orange juice (yellowish pigments), soda (without pigments) and red wine (purple pigmentation). As a control, artificial saliva was used, without any addition of pigments.

The assessment of the change in the color of the specimens was performed with a spectrophotometer, using the CIELAB system. The CIELAB system is a chromatic measurement method developed by the CIE (Comission Internacionale de L'Eclairage) in 1976, which expresses numerically, the color of an object. It is based on three spatial coordinates perpendicular to each other: the L* axis, which represents the brightness of the object, varying from black (L=0) to white (L=100); the shaft*, which refers to the variation of color of red (a* positive) to green (a* negative); and the axis b*, which is the variation of color from yellow (b * positive) to blue (b * negative).(8) These measures of color were always performed in the same position, under the same conditions of luminosity and on a white background. The change of color (ΔE) among the chromaticity coordinates is calculated by applying the formula cited above. This value is obtained by the analysis of the values of L*a*b* initial and final evaluations obtained in one and the same object, in the case of this research, the same specimen[9].

The results show that the orange juice and soda did not promote significant color changes in the tested composite resins, being this staining similar to that caused by the control group. Whereas the wine changed significantly, the color of the resin discs, promoting the same degree of darkening in composite micro-hybrid and nanofilled resins . The wine shows, in its composition, higher concentration of pigments, mainly, in the colors red and purple, than the other tested solutions[10]. The ΔE promoted by the wine reached values of 12.8 to 13.6 for the composite resins Z250XT and Z350XT, respectively. According to the literature, there are three intervals of change of color (ΔE), which may indicate whether these changes are or are not significant from the clinical point of view. Thus, if the ΔE is lower than 1, it means that this change of color is imperceptible to the human eye. If the value is between 1.0 and 3.3, it means that this change is noticeable only by a qualified person, i.e., that the color of the resin is clinically acceptable. However, the value of ΔE exceeding 3.3 indicates that the change in the color of the material can be easily observed, being, therefore, clinically unacceptable[11].

This change of color was favored by the pH of the solution, which promotes the chemical degradation of the organic matrix and, consequently, favors the staining of the composite and by the presence of alcohol, which promotes the softening of the resin and promotes an irreversible degradation of the composite resin, as the increase in the material porosity, which favors the impregnation of pigments on the resins surface[11]. According to the results obtained here, it was observed that the red wine exerts action of the same intensity in micro-hybrid and nanofilled composite resins.

It was observed that the orange juice and the soda did not significantly change, the color of the composite resins, since the ΔE of micro-hybrid composite resin was 1.6 for the orange juice and 1.7 for soda, and ΔE of nanofilled composite resin was 1.7 for orange juice and 1.5 for soda. As previously described, although the acid pH of these solutions promotes the degradation of organic matrix of the resin, the solutions have few pigments that could cause the staining of the composites.

Despite the literature suggests that the nanofilled composite resins show improvement in the smoothness of the surface and color stability of nanofilled resins due to their structural characteristics of particles of smaller load[6,7], the present study showed that the nanocomposites have superior resistance to staining in relation to the micro-hybrid composite, after challenge with the tested beverages, which corroborates with other studies recently published[12-14].

The increase of surface roughness is a factor that also promotes the accumulation of pigments on an object. In the present study, the analysis of surface roughness revealed that all the tested solutions, except for artificial saliva, promoted the increase of surface roughness of micro-hybrid and nanofilled resins. It also facilitated the staining of resin discs immersed in red wine.

The literature shows that acidic solutions increase the roughness of the composites, probably because soften their surface, which leads to the leaching of the organic matrix and, in consequence, the displacement of the particles of load, contributing to the formation of gaps, turning the surface rough [11,14,15]. This would be a feasible explanation for the results obtained, due to the low pH of solutions: orange juice 3.4; soda 2.9 and red wine 3.1.

Some studies have shown that alcohol can also affect the surface integrity of composite resins. The absorption of alcohol molecules by the resin matrix could also result in a softening of the composite surface. This explains the change in surface roughness of the resins when immersed in the red wine[11.14].

According to the results obtained in the present study, it was observed that the wine was the only solution that has promoted significant staining of micro-hybrid and nanofilled composite resins . It was also possible to observe that the orange juice, wine, and soda changed the roughness of the resins due to the acidic pH of these solutions. However, one should take into consideration that some extreme conditions were used in this study and that, many times, they do not happen daily in the oral environment, in which the saliva has a fundamental role in the medium pH neutralization in which the composite resin is. However, the methodology used here can make a forecast of how the composite resin restoration would behave in the long term, when present in teeth of individuals that make the intake of these beverages routinely.

4 Conclusion

Before the methodology used and experimental conditions used here, it can be concluded that: wine has changed significantly, the color of the micro-hybrid and nanofilled composite resins; and the roughness of resin discs has been changed by all the used drinks, apart from artificial saliva.

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