

## EFFECTS OF ARTIFICIAL ACCELERATED AGING ON THE OPTICAL PROPERTIES OF RESIN COMPOSITES

### *EFEITOS DO ENVELHECIMENTO ACELERADO ARTIFICIAL NAS PROPRIEDADES ÓPTICAS DE RESINAS COMPOSTAS*

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**ABSTRACT:** The aim of this study was to evaluate the effects of ultraviolet artificial aging on color, fluorescence, gloss, and translucency of different composite resins. Seventy-five cylindrical-shaped specimens (6 mm in diameter and 1 mm thick) of four different composites: Admira Fusion (VOCO), TPH3 (DENTSPLY), GrandioSO (VOCO) and Filtek™ Z350 XT (3M/ESPE), were built. Enamel samples obtained from bovine incisors were prepared with the same dimensions and used as a control group. Assessments of color, translucency, fluorescence, and gloss were performed at baseline and after artificial aging. Specimens were immersed in artificial saliva and submitted to artificial aging (UV light for 300h). Data were analyzed using one-way ANOVA and Tukey's post hoc test ( $\alpha=0.05$ ). After aging, all resin composites presented significant alterations, mainly Filtek™ Z350 XT. Fluorescence decreased in all resin composites and enamel. Gloss was reduced in all groups, too, with major rates for TPH3 and GrandioSO, which was similar to the enamel reduction. Translucency did not present a significant difference for the resin composites, although there was a reduction in the enamel. After artificial aging resin composites became darker, less fluorescent, less glossy, and with no alteration in translucency.

**KEYWORDS:** Composites. Enamel. Aging. Color. Translucency. Fluorescence. Gloss.

### INTRODUCTION

Resin composites are materials that reproduce tooth color and were developed to replace lost structures and promote esthetically pleasing outcomes during dental treatments. They are mainly made up of an organic matrix resin monomer and inorganic filler particles, which are joined together by a silane. Resin composites also include photoinitiators, accelerators, and pigments. However, some of these components may degrade over time, affecting the resin composite's optical characteristics and durability, and could compromise the esthetic outcome obtained initially. Color stability is a crucial factor for an aesthetic restorative material, and visible color change is a major cause of replacing restorations in anterior teeth (CHAN et al., 2010).

Several factors may promote composite resin degradation. Among them can be highlighted interaction with ambient light (WOZNIAK *et al.*, 1985), thermal variations of the oral cavity, exposure to alcohol, acid or oily substances from the diet, or acids from bacterial metabolism. In the case of ambient light, exposure to the ultraviolet wavelengths present in natural and artificial light sources has been reported to be deleterious (DE

OLIVEIRA et al., 2015. RATTACASO et al., 2011).

Fluorescence is the property of a substance that absorbs light and spontaneously emits it at a longer wavelength. In dentistry, fluorescence has been assumed to be the absorption of ultraviolet (UV) light radiation by a substance and emission of visible light in the blue spectral region. According to Spitzer and Ten Bosch (SPITZER; BOSCH, 1976), the natural tooth emits a strong blue fluorescence under UV light. In an attempt to approximate the resin composite characteristics with those of the dental structure, some fluorescent substances were added to the composition of resins. Previous studies showed a reduction in fluorescence when composite resins were exposed to different modalities of artificial aging (JABLONSKI et al., 2014. YU; LEE, 2013)

Another crucial optical feature is surface gloss. Surface gloss is a visual attribute describing the geometric distribution of light reflected at the surface of a material. This characteristic is directly related to the smoothness obtained on the restoration surface after polishing, which is strongly related to the type of inorganic filler. The interaction of ambient light with the material should not make changes in this property. However, this subject needs to be investigated (CAMPBELL et al., 1986).

Optical properties of restorative materials such as color, gloss, translucency, and fluorescence could be influenced by its chemical composition, polymerization systems, and particle size since they are directly related to the surface smoothness of the resin and, thus, on the susceptibility of extrinsic staining of the restoration. Initiators, UV irradiation, temperature changes, degradation of filler particles, the degree of polymerization, and conversion of monomers are also related to alterations of these materials (JANDA et al., 2004. UCHIDA et al., 1998. PEARSON; LONGMAN, 1989. FERRACANE et al., 1985). The degree of conversion of resin composite monomers can range from 50-70% (HALVORSON et al., 2002). Changes in optical properties could be a result of physical and chemical reactions of these residual monomers. In an attempt to improve the characteristics of these materials and increase their durability, new monomers and changes in the inorganic matrix in the composition of resins have been proposed.

Artificial aging can be employed for analysis in the short and long terms, resulting in a

material degradation that can lead to changes in mechanical and optical properties of the material. The aging is dependent upon many factors and can promote alterations in the surface microstructure, the chemical composition of the matrix, and filler particles of composite resins (DOS REIS et al., 2013).

The aim of this study was to evaluate the effect of accelerated aging on the stability of the optical properties of composites. The null hypothesis tested is that accelerated aging does not affect optical properties, such as translucency, fluorescence, gloss, and color of composite resins.

## MATERIAL AND METHOD

### Specimen Preparation

#### Resin composites

In this study, the following four commercial resin composites in shade A2 were used: Admira Fusion, TPH3, GrandioSO and Filtek Z350XT. Details are listed in Table 1.

**Table 1.** Material tested

Composite	Type	Manufacturer	Monomer and Composition	Lot
Admira Fusion	Nano hybrid	VOCO GmbH, Cuxhaven, Germany	BHT, Ormocer, Barium Glass Aluminium Boro-Silicate, Silicon Dioxide, methacrylates.	CHV56172
TPH3	Nano hybrid	DENTSPLY DeTrey GmbH, Konstanz, Germany.	BisGMA, EDAB, Barium Glass Aluminium Boro-silicate, barium glass Fluorine Aluminium Boro-Silicate, Silica.	860689F
GrandioSO	Nano hybrid	VOCO GmbH, Cuxhaven, Germany	BisGMA, TEGDMA, BHT, methacrylate matrix.	1416274
Filtek™ Z350 XT	Nanoparticulated	3M/ESPE. St. Paul, MN, USA	UDMA, BisGMA, TEGDMA, BisEMA. Ceramics treated silanized (60-80 wt%), silica, zirconia, Bisphenol A polyethylene glycol diether dimethacrylate, polyethylene glycol dimethacrylate, 2,6-di-tert-butyl-p-cresol.	881381

UDMA: diurethane dimethacrylate; BisGMA: bisphenol A diglycidyl ether dimethacrylate; TEGDMA: triethylene glycol dimethacrylate; BisEMA: ethoxylated bisphenol A dimethacrylate; EDAB: Ethyl 4-dimethylaminobenzoate; BHT: butylated hydroxytoluene.

Resin composites were packed into standardized circular molds measuring 6 mm in diameter and 1 mm thick. The molds and materials were covered with Mylar strips on the top and bottom and placed between two cover glasses. Finger pressure was then applied to extrude excess material and eliminate porosities. The materials were light-cured (Radii Cal curing light, SDI, Victoria, Australia; wavelength, 440-480 nm;

intensity, 1200 mW/cm<sup>2</sup>) according to the manufacturer's instructions. Fifteen specimens were fabricated for each material. The specimens were stored in distilled water for 24h at 37°C to ensure complete polymerization.

The specimens were attached to a metal holder and polished with sequential (1200, 2400, and 4000 grit) aluminum oxide abrasive papers (FEPA-P, Struers, Ballerup, Denmark) in a

polishing device (DP- 10, Panambra Industrial e Técnica SA, São Paulo, SP, Brazil) for 30 s each.

### Enamel

Fifteen extracted, non-damaged, and intact bovine incisors were stored in a 0.1% thymol solution at room temperature until required. Cylindrical samples with a 6 mm diameter and 1 mm thickness of enamel were prepared from the labial surface of each tooth using a trephine mill (FNMoraes, São Paulo, Brazil).

### Optical properties

#### Color and Translucency Analysis

Before artificial aging, baseline color and translucency of each specimen were assessed under standardized ambient conditions according to the CIE  $L^*a^*b^*$  system, using a spectrophotometer (CM2600d, Konica Minolta) and an integrating sphere. The device was adjusted to use the D65 standard light source with 100% UV included or 100% UV excluded and specular reflection included (SCI). The observer angle was set at 2°, and the device was adjusted to a small reading area (SAV). The color of each sample was measured three times and averaged. The results of the color measurements were quantified regarding three coordinate values ( $L^*$ ,  $a^*$ ,  $b^*$ ) as established by the Commission Internationale de l'Eclairage (CIE), which locates the color of an object in a three-dimensional color space. The  $L^*$  axis represents the degree of lightness within a sample and ranges from 0 (black) to 100 (white). The  $a^*$  axis represents the degree of the green/red color within the sample, while the  $b^*$  axis represents the degree of the blue/yellow color within the sample. The color was measured over white ( $L$ : 84.95;  $a$ : -0.38;  $b$ : 2.93) and black ( $L$ : 2.58;  $a$ : -0.15;  $b$ : -0.24) standard backgrounds. Optical contact between the specimen and the backgrounds was improved by using an interfacing layer of a polyethylene glycol 400 solution (LabSynth, Diadema, São Paulo, Brazil). Computations for the CIEDE2000 ( $\Delta E_{00}$ ) color difference formula were made according to the following equation:

$$\Delta E_{00} = [(\Delta L'/K_L S_L)^2 + (\Delta C'/K_C S_C)^2 + (\Delta H'/K_H S_H)^2 + R_T (\Delta C'/K_C S_C) (\Delta H'/K_H S_H)]^{1/2}$$

Where  $\Delta L'$ ,  $\Delta C'$ , and  $\Delta H'$  are the differences in lightness, chroma, and hue for before and after aging in CIEDE2000, and  $R_T$  is a function (rotation function) that accounts for the interaction between chroma and hue differences in the blue region. Weighting functions,  $S_L$ ,  $S_C$ ,  $S_H$ , adjust the total color difference for variation in the location of

the color difference in  $L'$ ,  $a'$ ,  $b'$  coordinates and the parametric factors  $K_L$ ,  $K_C$ ,  $K_H$ , are correction terms for experimental conditions.

Translucency was expressed using the translucency parameter (TP), which was calculated as the following expression:

$$TP = [(L_B^* - L_W^*)^2 + (a_B^* - a_W^*)^2 + (b_B^* - b_W^*)^2]^{1/2}$$

The subscript B refers to the color coordinates of specimens over the black background and the subscript W refers to those measurements over the white background.

#### Fluorescence analysis

Fluorescence measurements were performed using the spectrofluorimeter RF-5301 PC (Shimadzu Corp., Kyoto, Japan) with the excitation wavelength at 365nm. The emission spectrum (400 to 600nm) was obtained and the value of the emission peak in arbitrary units (a.u.) and wavelength were recorded using the "peak pick" tool of a specific software (RFPC - Shimadzu Corp., Kyoto, Japan).

#### Gloss Analysis

The gloss of the surface was measured with the Novocurve glossmeter (Rhopoint TM, East Sussex, England). The measuring principle of this device is based on a light beam that strikes the surface at an angle of 60°; the glossmeter measures the intensity of the reflected light and compares it with a reference value. Each time before a new test series was measured, the glossmeter was calibrated by comparing the results with the calibration plate. To perform the measurements, the specimen was placed on the top plate and completely covered the aperture at the center. The specimen was covered with a container to exclude external light during the measurement. Three readings were carried out at the center of each specimen. The three readings were averaged to obtain a single value for each specimen.

#### Artificial aging

After examining the samples at baseline, they were artificially aged in a weathering machine (SUNTEST CPS<sup>+</sup> - Atlas Material Testing Technology GmbH, Linsengericht, Hesse, Germany), following the standard of ISO 7491. The temperature was adjusted to 37±5°C and the irradiance to 765 W/m<sup>2</sup> to simulate an exposition of 160klux, corresponding to intense daylight, for 300 h, which means 1 year of clinical service. During the artificial aging, samples were immersed in artificial saliva at 37 ±1°C, which was prepared

according to the formulation of Gohring et al.[16] After aging, optical properties were evaluated again.

### Statistical Analysis

To compare the effect of UV aging over the different composites, the percentage of change of fluorescence emission (%FL), gloss (%GL), and translucency (%TP) after aging was calculated in relation to the values at the baseline evaluation, using the following formula:

$$\% \text{ of change} = ((\text{aged} - \text{baseline}) / \text{baseline}) \times 100$$

The normal distribution of the data was confirmed using Kolmogorov-Smirnov test. The

data of  $\Delta E$ , %FL, %GL, and %TP were submitted to one-way ANOVA and Tukey's test ( $\alpha=0.05$ ). The absolute data of FL, GL, and TP were submitted to repeated measurement ANOVA.

### RESULTS

The results of one-way ANOVA and Tukey's test for  $\Delta E_{00}$ , %FL, %GL, and %TP are presented in Table 2. In relation to the color change after aging, Z350 showed a higher color change than the other materials tested.

**Table 2.** Mean ( $\pm$ SD) of  $\Delta E_{00}$  and percentage of fluorescence, gloss, and translucency change in relation to baseline evaluation, and results of ANOVA and Tukey's test.

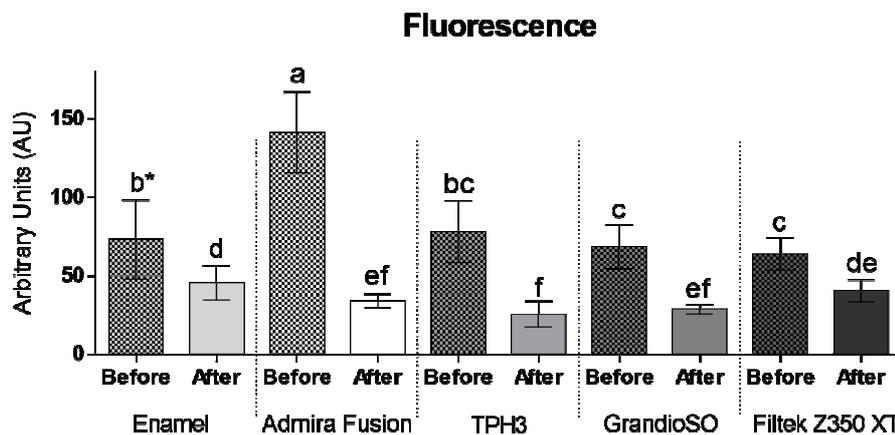
Groups	$\Delta E_{00}$	% fluorescence	% gloss	%translucency
ENAMEL	2.77 ( $\pm$ 0.81)a*	-55.77 ( $\pm$ 14.72)b	-64.13 ( $\pm$ 11.36) ab	-15.64 ( $\pm$ 1.11)a
Admira Fusion	2.95 ( $\pm$ 0.34)a	-75.30 ( $\pm$ 5.68)a	-50.64 ( $\pm$ 15.48)c	7.07 ( $\pm$ 1.48)b
TPH3	3.27 ( $\pm$ 0.71)a	-64.47 ( $\pm$ 14.89)ab	-76.43 ( $\pm$ 11.47)a	4.71 ( $\pm$ 1.08)b
GrandioSO	3.23 ( $\pm$ 0.38)a	-38.72 ( $\pm$ 10.89)c	-74.72 ( $\pm$ 16.24)a	0.60 ( $\pm$ 0.60)b
Z350	7.85 ( $\pm$ 0.59)b	-41.43 ( $\pm$ 14.65)c	-50.66 ( $\pm$ 17.72)bc	1.14 ( $\pm$ 0.58)b
ANOVA	p=0.000**	p=0.000**	p=0.000**	p=0.000**

\*Groups followed by the same letter on each column do not present significant differences.

\*\*Significant differences.

For fluorescence, all tested materials, including natural enamel, showed a significant reduction after aging ( $p=0.000$ ). The highest percentage of reduction was observed for Admira

Fusion (Table 2), which was the composite with highest initial fluorescence (Figure 1). This material presented baseline means of fluorescence emission significantly higher than the natural enamel.



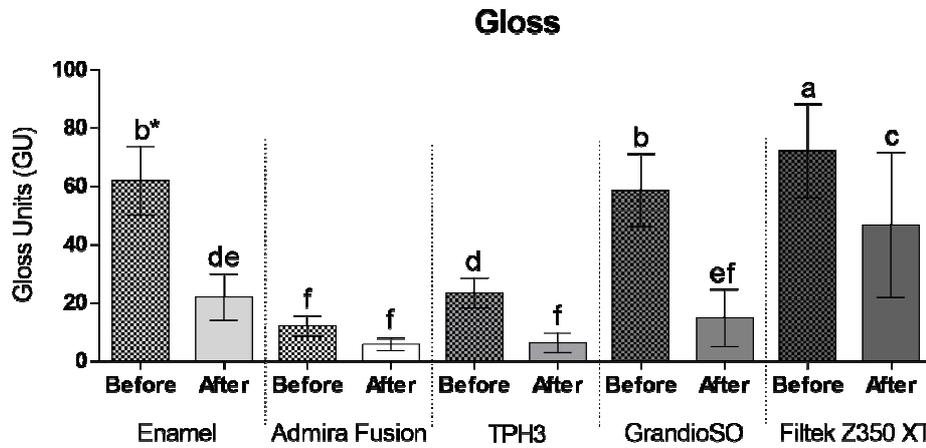
\*Groups followed by the same letter do not present significant differences

**Figure. 1** Means of fluorescence emission (FL) for all groups, before and after aging. Groups followed by the same letter do not present significant differences

The artificial aging also significantly reduced the gloss of the samples ( $p=0.000$ ). The highest percentage of reduction was observed for TPH3 and GrandioSO, which was similar to the natural enamel (Table 2). About the absolute gloss

values, the baseline measurement showed that the Z350 presented a higher gloss than all the other groups, including natural enamel (Figure 2). The composite GrandioSO was similar to the enamel

while the composites Admira Fusion and TPH3 showed smaller values.

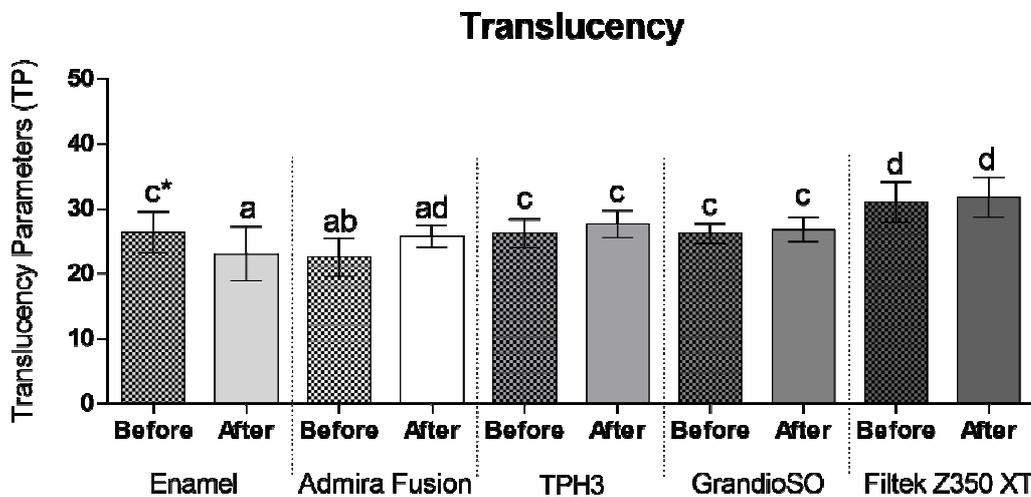


\*Groups followed by the same letter do not present significant differences

**Figure. 2** Means of gloss for all groups, before and after aging. Groups followed by the same letter do not present significant differences

Regarding the translucency analysis, artificial aging did not play a significant effect for the composites tested (Figure 3). The only statistical

significant change observed was a 15.64% translucency reduction for the enamel group (Table 2 and Figure 3).



\*Groups followed by the same letter do not present significant differences

**Figure. 3** Means of translucency parameter units for all groups, before and after aging. Groups followed by the same letter do not present significant differences

**DISCUSSION**

The null hypothesis was rejected for the color, fluorescence, and gloss analysis, since the artificial aging significantly affected these optical properties of the studied resins. Therefore, for

translucency analysis, the null hypothesis was accepted.

One of the main disadvantages of the resin composite is its changes in color over the years, due to the challenges faced in the oral environment, derived from plaque accumulation, surface degradation, penetration of agents with chromogens

in the superficial layer of the composite resin, and intrinsic discoloration due to physical and chemical reactions (KORKMAZ-CEYHAN et al., 2014).

Color changes in resin composites are related to changes in the resin matrix and size of the filler particles (FERRACANE et al., 1985). During light curing, initiators and tertiary aromatic amines form products that under temperature or UV light cause resin discoloration toward red or yellow.

The exposure to ultraviolet light causes significant changes in the coordinates in CIE color space in composite resins, and the most significant change occurs in  $b^*$  to a more positive value, or toward a yellowish hue (UCHIDA et al., 1998). The color change of materials is due to the combination of the moving of axes for green and yellow. Yellow, above the green, is induced by UV light and is due to a derivative of camphorquinone. However, there are some speculations in literature for the color change of resins, such as camphorquinone residues, changes in vinyl groups, late polymerization of composite resins, and breaking of chemical linkages induced by UV light (KOLBECK et al., 2006).

Ultraviolet light, temperature, and humidity are known to cause oxidation of amine (GHAVAM et al., 2010). The double bonds of aromatic amines may be a possible explanation for the yellowing of the resins, because these conjugated systems are capable of absorbing UV light, thereby creating a higher energy state. These molecules may then react with oxygen in other aromatic groups, and thus higher conjugated systems are created. Absorption of visible light is increased, so the material becomes yellow (SCHNEIDER et al., 2009). Even if the concentrations of unreacted amine for each composite was not measured, it is predicted that the increased yellowing with nanofilled composites occurred because of the lower conversion degree (OLIVEIRA et al., 2014).

In the present study, the Z350 resin showed the most considerable changes after aging. This material is composed of UDMA, differentiating it from the composition of other materials. This monomer is prone to yellowing after aging in UV light, as reported in previous studies (FALTERMEIER et al., 2007; JOHNSTON; REISBICK, 1997). This trend toward yellowing was also observed when the monomer Bis-GMA was exposed to UV light, and heat (FERRACANE et al., 1985) Z350, TPH3, and GrandioSO are Bis-GMA based. The polymerization of Bis-GMA occurs through carbon double bonds and methacrylate groups, and the existence of residual monomers which do not react during polymerization makes it

more susceptible to degradation, resulting in decreased color stability (PEUTZFELDT, 1997).

Another explanation for the color change is directly related to the particles' size and their hydrolytic degradation, modifying the light scattering and altering the visual color perception (KORKMAZ-CEYHAN et al., 2014). The hydrophilicity of the organic matrix provides an increase in water absorption, resulting in more white and opaque colors. Hydrophobicity is the opposite effect, the matrix absorbs less water and promotes better color stability (SIRIN-KARAARSLAN et al., 2013). The presence of TEGDMA, which is more hydrophilic and therefore predisposed to water absorption (PEUTZFELDT, 1997), may have contributed to the lower color stability of Z350 and GrandioSO.

The translucency parameter of a material refers to the color difference of a material with uniform thickness between a white and a black background. The translucency of a material depends on the rate of absorption and light scattering (MIKHAIL et al., 2013).

The organic matrix, the filler particles, and the dyes present in the formulations have a direct relation to the translucency of a material (MIKHAIL et al., 2013), and the presence of barium glass may decrease the translucency resin (BOUSCHLICHER et al., 1999). It can be assumed that translucency is changed by modifying the refractive index, which is because artificial aging affects the filling particles responsible for reflection and transmittance of light by changing the perception of translucency.

In the present study, resin composites did not show significant changes in translucency, contrasting with results of previous studies (LEE et al., 2007), GrandioSO, TPH3, and Z350 showed stability in translucency after artificial aging. Admira Fusion showed differences, but without significance. Nonetheless, the enamel samples showed the highest difference of translucency after aging.

For a material to be considered ideal for aesthetic use it should have fluorescence properties similar to the natural tooth. In previous studies human enamel fluorescence was considered low even before the aging processes. The fluorescence of the enamel is mainly attributed to its organic components, which represents less than 2% of its total composition (TAKAHASHI et al., 1998).

In this study, enamel and resins showed a decrease of fluorescence after aging, consistent with a previous study (LEE et al., 2006). Admira Fusion, which in baseline showed a higher fluorescence than enamel, showed the greatest decrease after aging.

This resin is composed of a matrix characterized by ORMOCER, an organic-inorganic copolymer.

The radiation from the UV light used to accelerate aging has the potential to be oxidative and induces cleavage of single and double bonds of carbon. These links are related to structures found in the organic polymeric matrix of resin-based materials. Therefore, the reduction in fluorescence could be explained by the degradation of these organic complexes (TAKAHASHI et al., 1998).

Gloss defines the appearance and the ability of a surface to reflect direct light and is associated with the smoothness of a surface, being important in evaluations of aesthetic materials. Changes in gloss caused by aging by UV light are related to the degradation of the tooth surface causing an increase in roughness and, therefore, light dispersion and then loss of gloss (SCHULZE et al., 2003), TPH3 and GrandioSO showed significant reduction in gloss after aging.

This study has some limitations in to reproduce of oral environment, which undergoes constant changes in its salivary pH and temperature, contributing to a greater deleterious effect of composite resins. However, it can be seen that there is a considerable degradation of the material, affecting the aesthetics. Thus, long-term clinical studies are suggested to assess with greater fidelity the aging effects over optical properties of composites.

## CONCLUSION

The artificial aging altered all optical properties evaluated, especially color stability of the composites. Z350 showed the worst performance regarding the maintenance of the baseline condition, being the material with the greatest color change. Although the resin GrandioSO presented lower gloss values, it was the material that remained more stable compared to the evaluated parameters. Enamel had the highest changes in translucency.

## CONFLICT OF INTEREST

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**RESUMO:** O objetivo deste estudo foi avaliar os efeitos do envelhecimento artificial ultravioleta na cor, fluorescência, brilho e translucidez de diferentes resinas compostas. Foram confeccionados setenta e cinco espécimes de forma cilíndrica (6 mm de diâmetro e 1 mm de espessura) de quatro compósitos diferentes: Admira Fusion (VOCO), TPH3 (DENTSPLY), GrandioSO (VOCO) e Filtek™ Z350 XT (3M / ESPE). As amostras de esmalte obtidas a partir de incisivos bovinos foram preparadas com as mesmas dimensões e utilizadas como grupo controle. As avaliações de cor, translucidez, fluorescência e brilho foram realizadas na primeira leitura e após o envelhecimento artificial. Os espécimes foram imersos em saliva artificial e submetidos ao envelhecimento artificial (luz UV por 300h). Os dados foram analisados usando ANOVA e teste post hoc de Tukey ( $\alpha = 0,05$ ). Após o envelhecimento, todos os espécimes apresentaram alterações significativas, principalmente para a resina Filtek™ Z350 XT. A fluorescência diminuiu em todos os materiais e no esmalte. O brilho foi reduzido em todos os grupos, também, com taxas maiores para TPH3 e GrandioSO, que foi semelhante à redução do esmalte. A translucidez não apresentou diferença significativa em todos os materiais testados, embora tenha havido uma redução no esmalte. Após o envelhecimento artificial, os compósitos de resina ficaram mais escuros, menos fluorescentes, menos brilhosos e sem alteração na translucidez.

**PALAVRAS-CHAVE:** Resina Composta. Esmalte. Envelhecimento. Cor. Translucidez.

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