

# Surface roughness, gloss and color change of different composites after exposure to ultimate challenges

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**Aim:** The study aimed to investigate the effect of the association of chemical and mechanical degradation on the surface roughness, gloss and color of nano and microfilled composites.

**Method:** Disc-shaped specimens (n=10) were prepared for three nanocomposites (Filtek Z350XT, IPS Empress Direct, Charisma Diamond) and three microfilled composites (Estelite  $\Sigma$  Quick, Durafill VS, Renamel). After polishing, baseline surface roughness, gloss and color measurements were obtained. Specimens were submitted subsequently to the following challenges: chemical for 1 week (Hydrochloric acid, Coca-Cola and Red Wine) and mechanical (Toothbrushing). Surface roughness and gloss data were analyzed by Kruskal-Wallis followed by Dunn's test. Color difference ( $\Delta E$ ) was analyzed by one-way ANOVA and Tukey test.

**Results:** The initial data were compared with those after challenges using the Wilcoxon test ( $p < 0.05$ ). All composites showed a significant increase ( $p < 0.05$ ) of surface roughness, after overstress. Filtek Z350 XT and Renamel had the lowest final surface roughness values and roughness increase ( $p < 0.05$ ). The comparative analysis revealed that Estelite  $\Sigma$  Quick and Renamel did not significantly change the gloss after challenges ( $p = 0.185$ ). Filtek Z350 XT and IPS Empress Direct had higher color variations, after the challenges than the other resins ( $p < 0.05$ ).  
**Conclusion:** Chemical and mechanical challenges influence on characteristics of all composites, except for gloss on Estelite  $\Sigma$  Quick and Renamel composites.

**Keywords:** Dental Resin; Physical properties; Immersion; Toothbrushing.

## INTRODUCTION

Changes in surface roughness, gloss, and color can compromise the longevity and the clinical success of restorations<sup>1</sup>. The composites undergo the action of intrinsic factors due to the physicochemical reactions in the inner portions of the restorative material, and also suffer the action from extrinsic factors, such as acidic substances that degrade organic matrix, exposing the material to coloring agents present in foods and beverages<sup>2,3</sup>, and abrasive substances, for example, from the brushing process<sup>4</sup>, influencing the composite characteristics, such as surface roughness<sup>5</sup> and gloss.

Surface roughness has been recognized as high clinical relevance parameter that contributes to dental plaque accumulation<sup>6</sup>. It is known that composite roughness is influenced by the size, distribution and volume of the filler content present in the restorative material. Furthermore, the increase of roughness is correlated with the characteristics of gloss and color of the composites restoration because of the possibility of a lower light reflectance and higher retention of pigments<sup>1</sup>.

It has been observed that the better the polish obtained at the end of the composite restoration, the greater the scattering of light inside the material and consequently this can result in a gloss increase<sup>7</sup>. In a condition that is necessary to produce highly aesthetic restorations, the gloss has an important role, since it needs to reproduce the optical properties of the enamel surface, making the restorations imperceptible to the human eye<sup>8</sup>.

The visible differences in color between teeth and esthetic materials are the major cause of patient discontentment with restorative treatments<sup>9</sup>. In this context, to obtain a composites restoration that reproduce colors of the tooth structure is considered a challenge, mainly in anterior teeth where slight color changes over time can influence patient compliance<sup>9</sup>.

The microfilled composites have high surface smoothness but present low mechanical strength in comparison with universal composites and therefore can be indicated as surface resins for anterior teeth<sup>4</sup>. The advent of the nanotechnology field has led to the development of nanocomposites with smoothness characteristics similar to those of microfilled resins, and also with high mechanical strength<sup>4</sup>. In vitro studies have analyzed the performance of these types of composites with the influence of toothbrushing and chemical degradation separately<sup>1-3,8,10</sup>. Thus, it is important to note that in the oral cavity, there is the interaction of these mechanical and chemical challenges<sup>4,5</sup>. However, there is a lack of studies evaluating the effects of chemicals immersions in association with toothbrushing.

The objective of this study was to evaluate the surface roughness, gloss and color changes of nanocomposites and microfilled resins after the association of chemical and mechanical challenges. The null hypotheses tested were: 1) there is no difference before and after challenges for composites when surface roughness and gloss were evaluated 2) the size and fillers of composites do not influence the surface roughness, gloss and color change after chemical and mechanical challenges.

## MATERIAL AND METHODS

### Specimen Preparation

The composites used in this experiment are shown in Table 1. Sixty disc-shaped specimens (5.0 mm diameter and 1.5 mm height) for each composites (n=10) were built up using a Teflon ring matrix. After the matrix was filled, the material was covered with a polyester strip and a glass slab. The specimens were then light-activated in the upper and lower surfaces for 40 seconds each, using a LED light-curing unit (Ultraled, Dabi Atlante, Ribeirão Preto, SP, Brazil, irradiance of 500 mW/cm<sup>2</sup>, monitored by a radiometer (model 100; Kerr, Danbury, CT, USA). After 24 hours in water storage at 37°C, all specimens were sequentially polished (Aropol E, Arotec Ind. Com. SA, Cotia, SP, Brazil) with the silicon carbide papers: 320-, 600-, 800- and 1200-grit. Uniform pressure and application time of 120 seconds were standardized, under constant water irrigation. The direction of polishing was from left to right and the rotation rate was set as 10.000 rpm. Between each silicon carbide paper and at the end of polishing procedures the specimens were cleaned in an ultrasonic bath (Cristófoli, Campo Mourão, PR, Brasil) with distilled water for 2 minutes. The final polishing was made using a Diamond Flex felt disks (FGM, Joinville, SC, Brazil) associated with polishing paste (Enamelize, Cosmedent Inc., Chicago, IL, USA) manually, to better simulate clinical procedures. After polishing, the specimens were stored in relative humidity at 37°C for 24 hours before the initial readings of surface roughness, gloss and color.

**Table 1.** Characteristics of composites materials analyzed in this study.

Composite resins	Manufacturer	Classification	Mean filler size and composition	Weight (%)	Organic Matrix	Batch #
Estelite $\Sigma$ Quick	Tokuyama Dental Co. Tokyo, Japan	Submicron filled	Spherical silica-zirconia filler 0.1-0.3 $\mu\text{m}$	71	Bis-GMA, TEG-DMA	E536
Durafill VS	Heraus Kulzer, Hanau, Germany	Microfilled	Prepolymerized Silica: 10-20 $\mu\text{m}$ Silica: 0.02-2 $\mu\text{m}$	40	UDMA	010218
Renamel	Cosmedent Inc., Chicago, IL, USA	Microfilled	Pyrogenic silicic acid filler 0.02-0.04 $\mu\text{m}$	59	Bis-GMA Bis-EMA	104819J
Filtek Z350XT	3M/ESPE, St. Paul, MN, USA	Nanofilled	Zircônia and silica clusters:0.6-1.4 $\mu\text{m}$ Silica: 5-20 nm	55	Bis-GMA Bis-EMA TEG-DMA UDMA	1210900268
IPS Empress Direct	Ivoclar Vivadent, Schaan, Liechtenstein	Nanohybrid	Barium alumina fluorosilicate glass, barium glass filler, mixed oxide, copolymer 0.04–3 $\mu\text{m}$	52-59	Bis-GMA TEG-DMA UDMA	N21727
Charisma Diamond	Heraus Kulzer, Hanau, Germany	Nanohybrid	Barium, aluminium, fluoride glass 5-20000 nm	64	TCD-DI-HEA UDMA	010041

### Surface Roughness Measurements

Three equidistant readings of surface roughness (Ra) was registered (Profilometer, SJ-401, Mitutoyo, Kanagawa, Japan) that represents the arithmetic mean of peaks and valleys of surface roughness on the specimens. The measurement was performed in each specimen individually, and the needle was positioned on the specimen surface and moved at a constant speed of 0.05 mm/s, using a cut-off of 0.25 mm. Three readings were performed on each surface specimen in equidistant positions in approximately 120°<sup>11</sup>. The average of these three measurements was calculated as roughness value of the specimen.

### Surface Gloss Measurements

The surface gloss was measured at a 45° angle of incidence and reflection using a calibrated glossmeter (Microgloss, BYK Gardner, Geretsried, Germany). The instrument measures the intensity of a reflected light beam after striking the surface and compares it to a reference value. The device has a measuring window of 2 mm x 2 mm, over which the specimen is placed and then covered with a film container to avoid external light exposure during the measurement. The average of three measurements was recorded for each specimen.

### Color analysis

Baseline color was measured according to the CIELab (Commission Internationale de l'Eclairage) color system using a reflection spectrophotometer (UV-2450; Shimadzu, Kyoto Japan) over a black background and standard illuminant D65. The CIELab color space graph is a 3-D color measurement: L refers to the lightness coordinate, and its value ranges from 0 for perfect black to 100 for perfect white; "a" and "b" are chromaticity coordinates on the green–red (a = green; +a = red) and blue–yellow (b = blue; +b = yellow) axes<sup>11,12</sup>. Color change was calculated between the color before challenges (baseline) and after challenges, measured by the formula:  $\Delta E = ([\Delta L^*]^2 + [\Delta a^*]^2 + [\Delta b^*]^2)^{1/2}$ <sup>11</sup>.

### Chemical and mechanical challenge

After initial readings, the specimens were submitted to chemical challenge by immersion of the samples individually into 10 ml of hydrochloric acid (pH 1.6, HCl 0.01mol/l, Apothicário, Araçatuba, Brazil) simulating the action of acid from the gastric juice, 10 ml of Coca Cola (pH 2.5, Coca Cola Company, Spaipa S.A., Marília, SP, Brazil) in order to simulate the action of the acid from soft drinks, and 10 ml of red wine (pH 3.4, Concha y Toro Cabernet Sauvignon, Santiago, Chile). The samples were individually stored in sealed tubes for one week in each acid solution at 37°C<sup>13,14</sup>. Between each acidic challenge, the samples were washed and immersed in 10 ml of distilled water for 24 hours at 37°C.

Specimens were individually subjected to 20.000 mechanical brushing cycles (MEV2, Odeme Biotechnology, Joaçaba, SC, Brazil) performed with toothbrushes (one for each specimen, Colgate Classic Clean, Colgate, Palmolive Co. Osasco, SP, Brazil) and toothpaste (Colgate Total 12, Colgate Palmolive, Kolynos Division of Brazil Ltd, Osasco, SP, Brazil) diluted in distilled water (ratio 1:2 by weight). After brushing, the specimens were rinsed with distilled

water for 2 minutes and submitted to ultrasonic bath for 10 minutes with distilled water to remove the abrasive particles from the toothpaste. The specimens were maintained in relative humidity at 37°C for 24 hours before final readings of surface roughness, gloss and color, under the same conditions previously stated. Color changes ( $\Delta E$ ) were calculated by the formula:  $\Delta E = [(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2]^{1/2}$ . All analyses were blinded to the resins.

### Scanning Electronic Microscopy (SEM) Analysis

SEM analysis was not performed initially because same specimens were used in all experimental procedures. Two specimens of each material were mounted on aluminum stubs, sputter-coated with gold (Balzers SCD-050 sputter coater, OC Oerlikon Corporation AG, Pfäffikon, Switzerland) and submitted to SEM analysis (Evo LS15, Carl Zeiss, Oberkochen, Germany) at 5000x magnification.

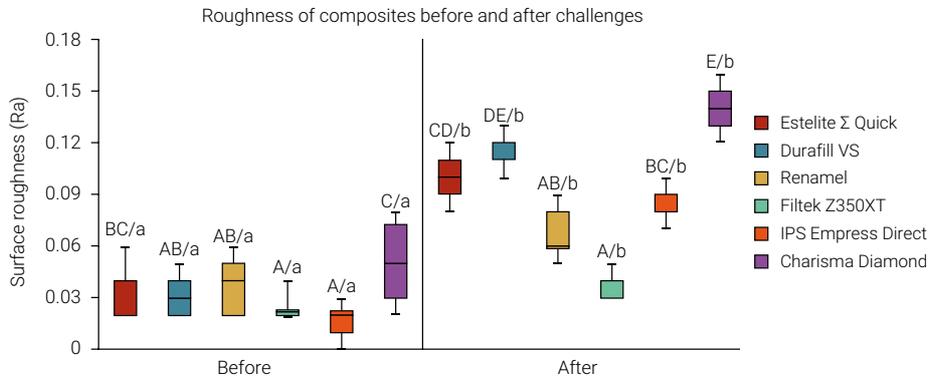
### Statistical Analysis

The assumptions of equality of variances and normal distribution of data were checked using the Shapiro–Wilk test, once the homogeneity was not achieved, data of surface roughness and gloss were analyzed with nonparametric tests, Kruskal-Wallis followed by Dunn's test. However, the normal range was observed for the color change, in which the means were evaluated by an ANOVA and Tukey's multiple comparisons. Wilcoxon test was applied for surface roughness and gloss analysis considering two-time evaluation (before and after challenges). Spearman test was used to check the correlation between the surface roughness and gloss parameters. Statistical testing was performed using SPSS Version 20 (IBM SPSS Statistics for Windows, Armonk, NY, USA). A p-value less than 0.05 was considered statistically significant.

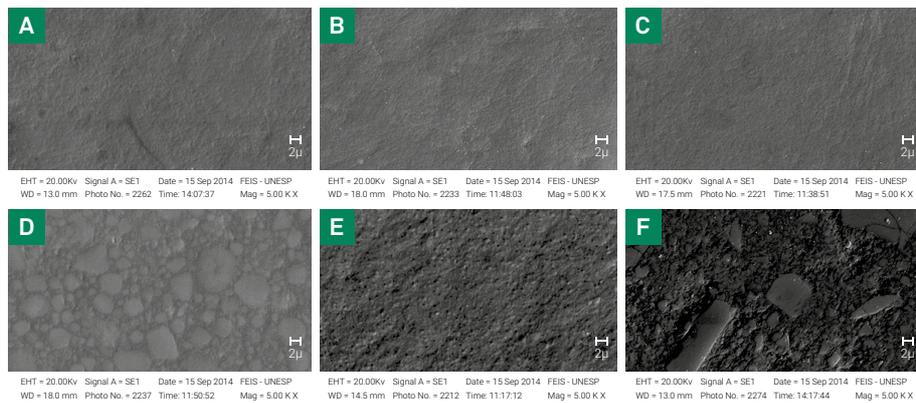
## RESULTS

Data of surface roughness are shown in Figure 1. Charisma Diamond had the highest initial surface roughness value, with a significant difference for all resins, except for Estelite  $\Sigma$  Quick ( $p=0.052$ ). After challenges, Charisma Diamond and Durafill VS showed the highest values of surface roughness (Fig. 2B and F). All composites showed a significant increase in amount of surface roughness after the association of challenges. Filtek Z350 XT and Renamel had the lower Ra values when compared to other composites, as shown in Fig. 2C and D.

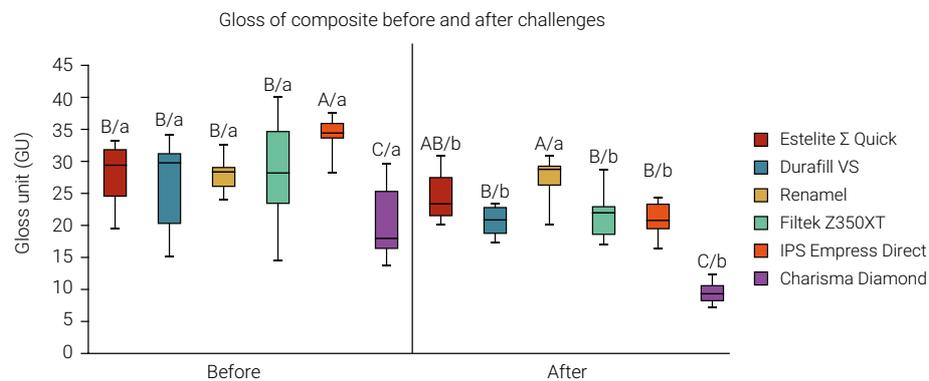
The initial analysis of the gloss revealed that the IPS Empress Direct and Charisma Diamond had the highest and lowest gloss values, respectively, with statistical difference for the other composites ( $p<0.05$ ). After association of the challenges, it was found that the microfilled resins had the lowest gloss reduction values (Fig. 2A, B and C). The comparative analysis revealed that Estelite  $\Sigma$  Quick and Renamel did not show statistically significant change of gloss after challenges ( $p=0.185$ ). These values can be seen in Figure 3. The Spearman correlation test showed positive correlation between surface roughness and gloss for the initial analysis ( $p=0.0198$ ; Coef. 0.8827), however, there was no correlation after the challenges ( $p=0.1107$ ; Coef. 0.7143).



**Figure 1.** Box plots of surface roughness values (Ra:  $\mu\text{m}$ ) of resin composites before and after the challenges. Different uppercase letters represent statistical significance among composites ( $p < 0.05$ ). Different lowercase letters indicate statistical significance before and after challenges ( $p < 0.05$ ).



**Figure 2.** Representative scanning electron micrographs of composites after submission challenges. (A) Estelite  $\Sigma$  Quick, (B) Durafill VS, (C) Renamel, (D) Filtek Z350 XT, (E) IPS Empress Direct, and (F) Charisma Diamond. Note different patterns of corroded resin matrix provided by the association of chemical and mechanical challenges. Original magnification 5000X.



**Figure 3.** Box plots of gloss (GU unit) of resin composites before and after challenges. Different uppercase letters represent statistical significance among composites ( $p < 0.05$ ). Different lowercase letters indicate statistical significance before and after challenges ( $p < 0.05$ ).

Regarding color changes, after challenges it was found that Filtek Z350 XT and IPS Empress Direct had higher color variations than the other resins ( $p < 0.05$ ) (Table 2).

**Table 2.** Comparisons of color change among different materials,  $\Delta L^*$ ,  $\Delta a^*$ ,  $\Delta b^*$  e  $\Delta E$ .

Composite resins	$\Delta L^*$	$\Delta a^*$	$\Delta b^*$	$\Delta E$
Estelite $\Sigma$ Quick	0.69	0.72	-1.17	1.80 (0.42) <sup>B</sup>
Durafill VS	-1.08	0.26	-0.85	1.50 (0.66) <sup>B</sup>
Renamel	-0.24	-0.16	1.59	1.70 (0.46) <sup>B</sup>
Filtek Z350 XT	-2.08	0.94	-0.80	2.80 (1.08) <sup>A</sup>
IPS Empress Direct	1.63	2.40	1.70	3.54 (0.80) <sup>A</sup>
Charisma Diamond	1.31	0.55	0.17	1.65 (0.74) <sup>B</sup>

Means followed by the same letters in columns are not statistically different for the ANOVA and Tukey test at  $p > 0.05$ .

## DISCUSSION

*In vitro* studies have analyzed separately the influence of chemical challenges<sup>2,14,15</sup> and toothbrushing<sup>7,8,10</sup> on surface changes of resin composites. However, it is well known that the degradation of materials in the oral environment is a complex process, which involves mechanical and chemical mechanisms<sup>5,15,16</sup>. Thus, challenges were not performed separately in this present study to predict the performance of composites after ultimate challenge. The characteristics of surface roughness, gloss and color are considered important properties for the clinical success of restorative materials<sup>1</sup>.

The chemical challenges tested in this study used acidic solutions and dyes, simulating the influence of intrinsic and extrinsic factors in restorative materials<sup>17,18</sup>. The mechanical challenge was performed by brushing process that, over time, has caused wear on the resin surface, especially in the organic matrix promoting the inorganic particles display, and increasing the surface roughness of the composites<sup>4,7,10</sup>. Twenty thousand brush cycles were used since it is considered superior to 3 years *in vivo*<sup>19</sup>.

The first null hypothesis evaluated was rejected. All of the tested composites showed statistically Ra surfaces after challenges. These results corroborate with previous studies that found the increase in surface roughness after chemical challenge<sup>2</sup> and mechanical challenges<sup>2,4,7,10</sup>.

Regarding the gloss, only two microfilled composites studied did not show statistical difference after the proposed challenges. Lee et al.<sup>20</sup> observed that Renamel showed lowest change in gloss when compared with other composites studied, when a progressive number of toothbrushing cycles were applied, as it was observed in the present study. Renamel also presented the best values of gloss when different finishing and polishing procedures were studied<sup>21</sup>. It is noteworthy that this resin showed the lowest value of gloss loss among all analyzed resins, changing only 1% of the initial gloss after exposure to ultimate challenge proposed in this present study (Fig. 1C).

The lack of difference in gloss after the challenges for both Estelite  $\Sigma$  Quick and Renamel can be justified by the shape and size of fillers<sup>20</sup>. Since Estelite  $\Sigma$  Quick uses spherical fillers, these particles are able to reflect more light than irregular ones<sup>20</sup>. Although Renamel have irregular shapes, the predominance of particles were of one micron or less<sup>22</sup>. However, there is no consensus regarding the change of gloss, since large loss of gloss was observed after performing challenges when various types of composites were evaluated<sup>7</sup>. Therefore, it seemed that change in gloss was primarily influenced by the characteristics of the composites, not by amount of wear<sup>20</sup>.

When comparing the composites in relation to the characteristics evaluated, the second null hypothesis was also rejected. The highest Ra values for the Charisma Diamond resin were found before and after challenges. This composite resin, despite its nanometric particles, present large particle (Fig. 1F) size in the range 0.005-20 $\mu\text{m}$ , which may influence for larger surface roughness values after the degradation of the organic matrix, as also observed in a previous study<sup>23</sup>. The lowest value was found for final surface roughness and increased surface roughness of the nanofilled Filtek Z350 XT. This can be explained by the greater resistance to chemical and mechanical wear found by this resin, due to less exposure of the organic matrix, favored by the presence of nanoclusters in its composition, which can be observed in Fig. D<sup>2</sup>. Renamel exhibited similar surface roughness to the Filtek Z350 XT, probably due to its small average particle size (Fig. 1C)<sup>22</sup>. Higher values of roughness are unacceptable, since it favors the accumulation of bacteria and consequently greater risk of carious lesion and gingival inflammation<sup>7</sup>. In this study, it was observed that after the challenge proposed, all composites presented surface roughness lower than the 0.2 $\mu\text{m}$  (Ra), which is considered clinically acceptable<sup>6</sup>.

Gloss values, they vary according to the incidence of light on surface resin; the lack of reflection shows values 0 GU, while a glass surface with a high refractive index has about 100 GU<sup>21</sup>. To avoid changes in the refractive index of the samples, they were analyzed dry, preventing the formation of water film, which would alter the brightness of the reflection value<sup>24</sup>.

In the present study, the nanohybrid IPS Empress Direct showed the highest initial values of gloss, as seen in a previous study, after performing the polishing<sup>25</sup>. Takahashi, et al.<sup>10</sup> reported that the resin IPS Empress Direct classified for enamel, contain fine barium glass fillers, which may favorable high surface luster. Charisma Diamond resin statistically had the lowest brightness values, probably due to its particular feature regarding insertion of larger particles and a great weight of filler (64%). Furthermore, Charisma Diamond present a distinct monomer called TCD-DI-HEA instead of Bis-GMA and this could also have contributed for this result. It is known that Bis-GMA presents a high resistance to degradation from effects of immersion media by the fact that bis-acryl resin composite materials contain bifunctional acrylato provide increase d mechanical strength and resistance to weakening in the presence of solvents<sup>26,27</sup>.

In this context, the cross-link nature of the resin matrix, and the solvent sorption uptake may influence more directly the polymer's degradation rate<sup>3</sup>. Toothbrushing after the immersion on food-simulating media resulted in the removal of part of the organic matrix around the loosen fillers<sup>5,26</sup>. However, the effect of toothbrushing in the composites resins depends on different factors, such as the type of toothpaste, type

and shape of the brush bristles, the proportion of deionized-water solution as well as speed and weight applied during simulation process<sup>26</sup>.

After association of challenges, the highest gloss values were observed for the micro-filled resin Renamel followed by Estelite  $\Sigma$  Quick. Although, the resin Estelite  $\Sigma$  Quick present high roughness values (Fig. 1A). This finding may indicate that spherical fillers facilitate the abrasive media sliding from the surface of the specimen, instead of the fillers being removed by the abrasive media, explaining why this material retains its gloss after mechanical abrasion<sup>20</sup>. Then, gloss is not influenced only by the surface roughness, but also by other factors such as difference in the refractive indices of the resin matrix and the fillers<sup>20</sup>.

These factors may explain the existing initial correlation between the parameters surface roughness and gloss, since there is greater homogeneity of values in polished surfaces<sup>7,20</sup>. Immediately after finishing and polishing procedures is possible to level the organic and inorganic phase by the regularly wear of rotary cutting instruments. However, when the samples are subjected to experimental challenges, such action is selective and may have influence in the organic phase and in the different types of fillers<sup>15</sup>. This explains the lack of correlation between surface roughness and gloss after the challenges, which may cause a degree of random reflection to the extent that there is loss of surface polishing<sup>26</sup>.

Regarding color results, despite all the composites are marketed as A2, differences were observed between the composites tested after the challenges. The nanofilled Filtek Z350 XT and IPS Empress Direct showed greater variation in color when compared to the others. Filtek Z350 XT resin, despite having nanoparticles in its composition, showed higher sorption capacity than the microhybrid and microfilled resins<sup>17</sup>. This factor may have influenced to reduce the color stability, favoring the absorption of the dyes that are in Coke and wine. The nanohybrid IPS Empress Direct presents small particles of barium, compromising the strength of the material<sup>4,10</sup> and increasing the possibility of incorporation of color pigments<sup>27</sup>. Other factors that may influence for the color change are the amount of photoinitiators agents, fillers and pigments agents incorporated by manufacturers<sup>28</sup>.

However, it is important to remember that these differences in color changes only become clinically noticeable when  $\Delta E$  value is higher than 3.3<sup>12</sup>. According to the present study, IPS Empress Direct was the only resin in that category, since other composites showed good color stability after chemical degradation and brushing. Ren et al.<sup>18</sup> also reported that a nanocomposite (Filtek Supreme Ultra) showed more color change than TPH and Renamel after the stain challenges and after brushings.

An *in vitro* study cannot represent all the conditions and interactions acting on the restorative material in the oral cavity, since cycles of acid challenge and toothbrushing occurs in patients. In this sense, randomized clinical trials are required in order to assess these parameters, and especially the influence of these on the quality of life of the patient, as it interferes directly with the patient's self-esteem. Therefore, information obtained with this present study should prove valuable for clinicians to make decisions in selecting the best materials for aesthetic restorations for their patients. However, none of studied composites had the best performance for all analysis performed in this study.

In conclusion, within the limitations of this study can be drawn that chemical and mechanical challenges influence the surface roughness of all composites, and they have just no influence in gloss of Estelite  $\Sigma$  Quick and Renamel composites. The commercial classification of composite resin (nanocomposite or microfilled) was not a determining factor in relation to the analyzed characteristics.

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