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Influence of Mechanical and Chemical Degradation in the Surface Roughness, Gloss, and Color of Microhybrid Composites

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ABSTRACT

Aim: The aim of this study was to investigate the association of different degradations on the roughness, gloss, and color changes of microhybrid composites.

Materials and methods: Ten specimens were prepared for Charisma, Amelogen Plus, Point 4, and Opallis resins. Surfaces were polished and baseline measurements of roughness, gloss, and color were recorded. Specimens were then submitted to chemical and mechanical challenges, and the specimens were reevaluated. Roughness and gloss were analyzed by Kruskal – Wallis and Dunn's test ($p < 0.05$). Color change (ΔE) was analyzed by one-way analysis of variance and Tukey's tests ($p < 0.05$). The initial and final data were compared using the Wilcoxon test ($p < 0.05$). Spearman test checked the correlation between the roughness and gloss ($p < 0.05$).

Results: Regarding surface roughness and gloss, there was no difference between composites before challenges. However, all composites showed a significant increase of roughness after challenges, with highest values for Charisma. The gloss was influenced by challenges, evidencing the best gloss for Point 4. Charisma showed the highest value of color change. There was no correlation between surface roughness and gloss for the initial analysis, and after the challenges.

Conclusion: Composites were influenced by association of challenges, and Charisma showed the highest changes for roughness, gloss, and color.

Clinical significance: The type of composite resin influenced the properties of materials, which are surface roughness, gloss, and color change. The dentist should be aware of the performance of different brands, to choose the correct required composite resin for each type of patient or region to be restored.

Keywords: Chemical degradation, Composite resins, Esthetics, Physical properties, Toothbrushing.

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INTRODUCTION

The characteristics of surface roughness, gloss, and overall color change are considered important for the esthetic success of a restoration with composite resin, due to the optical appearance.[1] Several composite resins are available for direct dental restorations, and it is possible to observe differences between them, such as the monomer system, filler composition, and matrix – filler coupling chemistry, which may contribute with differences in mechanical or chemical degradation.[2]

The microhybrid resins are constituted by macro- and microparticles, presenting higher mechanical resistance and similar opacity as the tooth structure, being indicated mainly not only for the posterior region[3] but also for esthetic areas.[4] However, some studies showed disadvantages, such as reduction of surface smoothness over time.[5]

The surface roughness has been considered a very important characteristic for the longevity of restorations, in which values >0.2 µm present substantial increase in bacteria retention.[6] In addition, this roughness may also be related with other variables, such as gloss and color stability, due to the influence of intrinsic or extrinsic factors, such as exposure to acid agents, abrasives, and pigments, impairing the maintenance of these characteristics.[7]

Satisfactory clinical performance of the material is required to ensure more resistance to chemical and mechanical degradation by the oral environment.[7] Thus, *in vitro* studies have analyzed the performance of composite resins with the influence of chemical and toothbrushing degradation separately.[8,9] However, there is lack of studies evaluating the effects of immersion in chemicals associated with toothbrushing to simulate the interaction that occurs in the oral cavity.

Thus, the aim of this study was to evaluate the effect of chemical and mechanical degradation on the characteristics of surface roughness, gloss, and color of four microhybrid composite resins. The null hypotheses tested were: (1) There is no difference in the association of challenges on the surface roughness of composite resins; (2) there is no difference in the association of challenges on the gloss of composite resins; and (3) there is no difference in the association of challenges on the overall color change of composite resins.

MATERIALS AND METHODS

Specimen Preparation

Four commercial microhybrid composite resins (color A2) are shown in Table 1. Forty specimens of each material were prepared according to the manufacturers' instructions under aseptic conditions and inserted into Teflon ring matrix with internal dimensions of 5 mm diameter and 1.5 mm thick. One increment was inserted and light-activated for 40 seconds (Ultraled – Dabi Atlante, Ribeirão Preto, São Paulo, Brazil, 500 mW/cm^2) from the top surface. The excess material was removed with a scalpel blade 15c.

After 24 hours in water storage at 37°C, all specimens were sequentially polished (Aropol E, Arotec Ind. Com. SA, Cotia, São Paulo, 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, Brazil) 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, Illinois, 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.

Surface Roughness, Gloss, and Color Analysis

The surface roughness was analyzed on a profilometer SJ-401 (Mitutoyo, Kanagawa, Japan), using the Ra values and moved at a constant speed of 0.05 mm/s, using a cut-off of 0.25 mm. Three equidistant readings were performed on each surface in different positions, and each reading was obtained after turning the specimen approximately 120°. The average of these three measurements was calculated as roughness value of the specimen.

Surface gloss was measured at a 45° angle of incidence and reflection using a calibrated glossmeter (Microgloss, BYK, Geretsried, Germany). The gloss value varies according to the light incidence on the surface resin; the lack of reflection shows values of 0 GU, while a glass surface with a high refractive index has about 100 GU. The device has a measuring window of 2 mm × 2 mm. The average of three measurements was recorded for each specimen.

The color of composite discs was analyzed according to the Commission Internationale de l'Eclairege L*a*b* (CIELab) color space using a reflection spectrophotometer UV-2450 (Shimadzu, Kyoto, Japan) over a black background and standard illuminant D65. The CIELab system is a chromatic value color space that measures the value and chroma on three coordinates: L* – lightness of the color measured from black ($L^* = 0$) to white ($L^* = 100$); a* – color in red ($a^* > 0$) and green ($a^* < 0$) dimensions; and b* – color in yellow ($b^* > 0$) and blue ($b^* < 0$) dimensions, and the final values were calculated after final readings by the ΔE formula.

Chemical and Mechanical Challenges

After initial analysis the specimens were randomly immersed in 10 mL of hydrochloric acid (HCl 0.01M, pH 1.6,

Table 1: Characteristics of composite resins used in the study

Composite resins	*Manufacturer*	*Size of particles*	*Weight (%)*	*Batch #*
Charisma	Heraeus Kulzer, Hanau, Germany	Ba and SiO_2 glass particles from 0.04 to 2 μm	64	010510
Amelogen Plus	Ultradent, South Jordan, UT, USA	Barium boron aluminum particles from 0.4 to 0.7 μm	61	9031
Opallis	FGM dental products, Joinville, SC, Brazil	Barium-aluminum glass silanized silica pigments and silica particles from 0.02 to 3 μm	57.8	271110
Point 4	Kerr USA, Orange, USA	Barium aluminoboro silicate glass silicon dioxide from 0.4 μm in the average	57	3601238

Apothicário, Araçatuba, São Paulo, Brazil), simulating the action of acid from the gastric juice; in 10 mL of Coca Cola (pH 2.5, Coca Cola Company, Spaipa SA, Marília, São Paulo, Brazil) to simulate the action of acid from soft drinks; and in 10 mL of red wine (pH 3.4, Conchay Toro Cabernet Sauvignon, Chile), each one for 1 week at 37°C. The specimens were washed and immersed in 10 mL of distilled water for 24 hours at 37°C between immersions in each acidic solution.

After acid challenges, the specimens were subjected to mechanical challenges of 20,000 brushing cycles (ElQuip, São Carlos, São Paulo, Brazil) with toothbrushes (Colgate Classic Clean, Colgate Palmolive Co, Osasco, São Paulo, Brazil) and dentifrice (Colgate Total 12, Colgate Palmolive, Kolynos Division of Brazil Ltd, Osasco, São Paulo, Brazil) diluted in distilled water at a ratio of 1:2 per weight.

The specimens were immersed in an ultrasonic device for 10 minutes to remove the abrasive particles from the dentifrice. The specimens were maintained in relative humidity at 37°C for 24 hours for the final readings of surface roughness, gloss, and color, as previously described. All analyses were blinded to the type of composite resin.

Scanning Electron Microscopy Analysis

Scanning electron microscopy (SEM) analysis were performed (n = 3) to show the surface aspects of composite resins after the challenges. Three 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 examined with a SEM (Evo LS15, Carl Zeiss, Oberkochen, Germany) at 1,000× magnification of the most representative area of the specimen.

Statistical Analysis

The normality test (Shapiro–Wilk) was not applicable for the surface roughness and gloss variables. Thus, the statistical tests used for this analysis were the nonparametric Kruskal–Wallis followed by Dunn's test ($p < 0.05$), and the Wilcoxon test ($p < 0.001$) to compare the initial and final mean values of roughness and gloss. However, normality was observed for the overall color change; therefore, the means were evaluated by one-way analysis of variance and Tukey's multiple comparisons test ($p < 0.05$). Spearman test was used to check the correlation between the surface roughness and gloss parameters ($p < 0.05$). Statistical testing was performed using Statistical Package for the Social Sciences version 20 (IBM SPSS Statistics for Windows, Released in 2011, IBM Corp. Armonk, New York).

RESULTS

No significant difference was observed for initial analysis of surface roughness ($p > 0.05$). However, all resins exhibited significant increase in the Ra values after challenges ($p < 0.001$) (Figs 1A to D). Charisma presenting the highest change of Ra with statistically significant difference compared with other resins ($p < 0.05$). The resins Amelogen Plus and Point 4 had lower Ra values, while Opallis showed intermediate Ra values (Graph 1). Concerning the gloss values, no significant difference was observed in the initial analysis ($p > 0.05$). After challenges, all resins showed decrease of gloss values ($p < 0.001$), and the resin Point 4 showed the highest values in comparison with other resins ($p < 0.05$) (Graph 2). The color analysis revealed that the Charisma showed the highest overall color change, with statistical significant difference compared with the other resins ($p < 0.05$) (Table 2). The Spearman correlation test showed that there was no correlation between surface roughness and gloss for the initial analysis ($p = 0.06$), and after the challenges ($p = 0.58$).

DISCUSSION

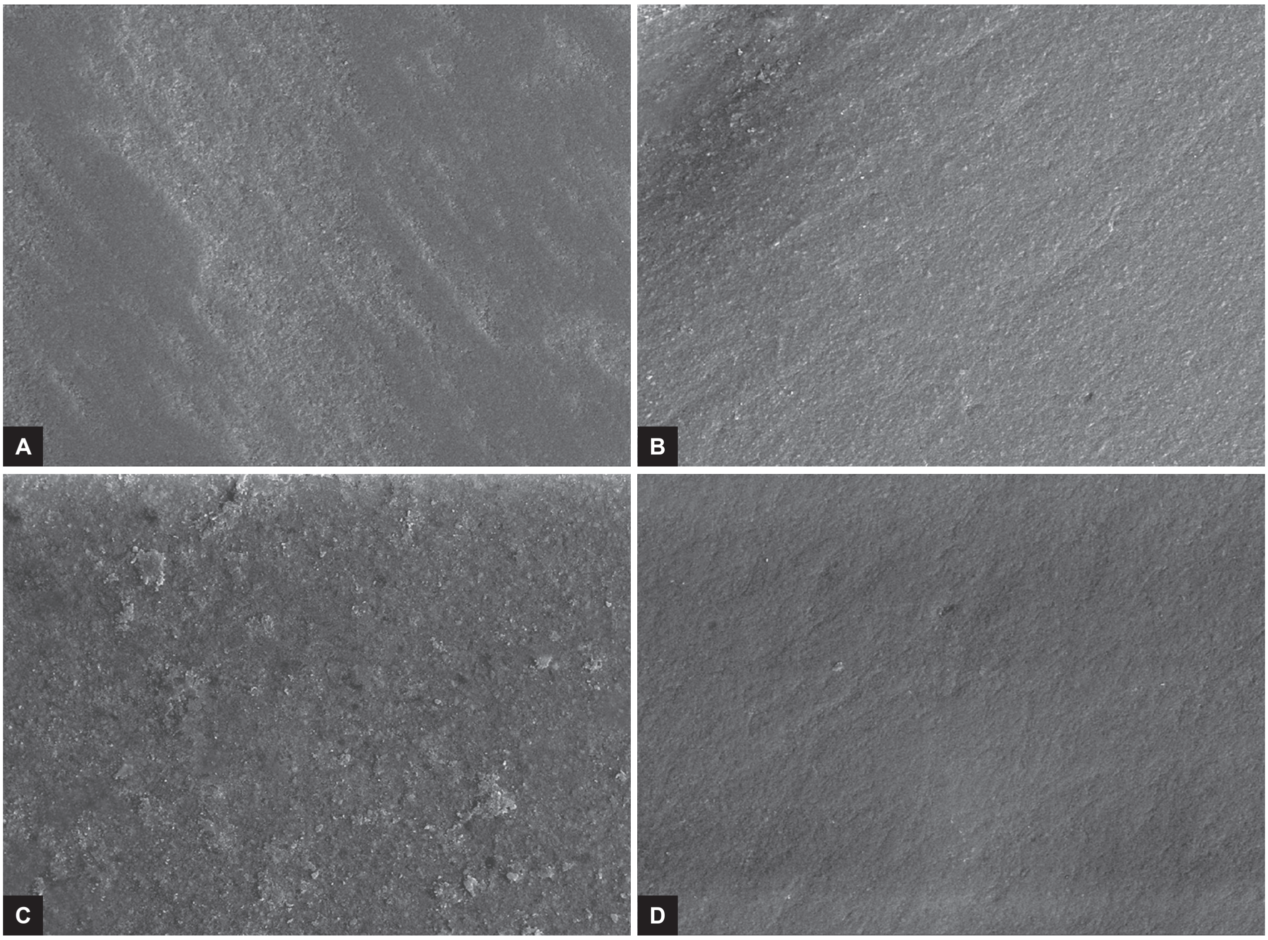
This study was performed to demonstrate the influence of the association of mechanical and chemical challenges in microhybrid composites, which are considered resins of universal use, being indicated for posterior[3] and anterior regions,[4] with excellent mechanical and esthetic properties and good polishability.[10] In this sense, it is indispensable that characteristics of surface roughness, gloss, and color remain stable over the lifetime of the restoration.[1]

This study employed chemical challenges with acidic solutions, such as hydrochloric acid to simulate the gastric reflux, which may be present in the oral fluids.[11] Coca-Cola, which presents phosphoric acid and other pigments and substances in its composition; and red wine, due to the low pH and to promote high color change in the restorative materials.[12] The mechanical challenge was performed by toothbrushing, which could influence the surface degradation of restorative materials. The 20,000 cycles used in this study correspond to a period of up to 3 years *in vivo*.[13]

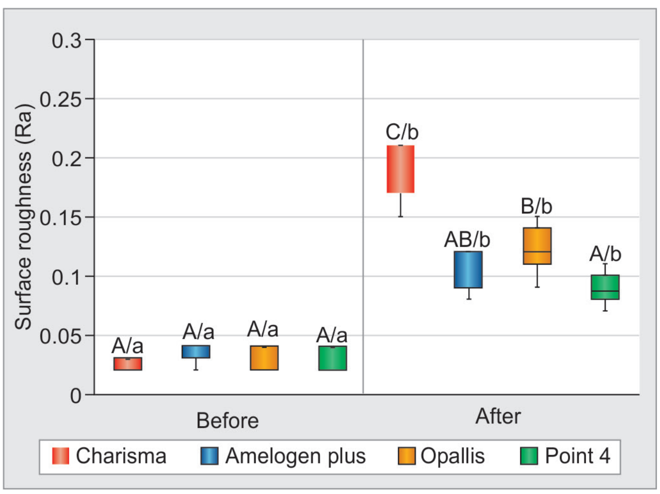
Table 2: Mean (standard deviations) for overall color change

Composite resins	*ΔL**	*Δa**	*Δb**	*ΔE*
Charisma	0.69	0.38	−0.21	2.96 (±1.37)^B
Amelogen plus	0.17	0.77	−1.16	1.77 (±0.38)^A
Opallis	0.75	0.61	−0.15	1.50 (±0.29)^A
Point 4	2.01	1.79	0.82	1.00 (±0.34)^A

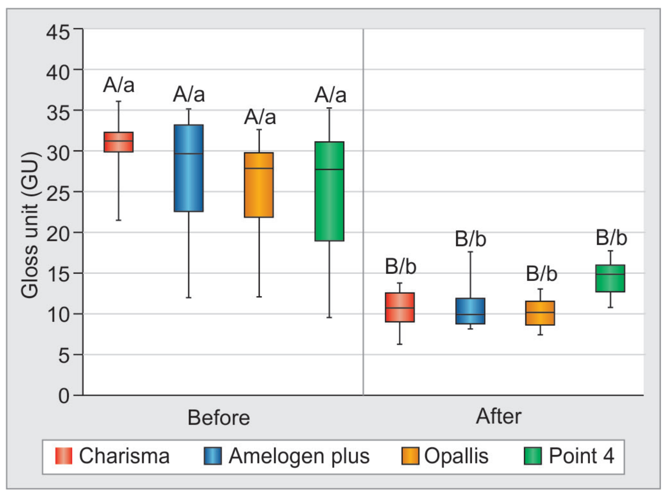
Different uppercase letters in columns indicate statistically significant difference ($p < 0.05$)



Figs 1A to D: Representative SEMs of composites after challenges. (A) Charisma; (B) Amelogen Plus; (C) Opallis; and (D) Point 4. Note different patterns of corroded resin matrix provided by the association of chemical and mechanical challenges. Original magnification, 1,000×



Graph 1: Box plots of surface roughness values (Ra: μm) of 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 ($p < 0.05$)



Graph 2: Box plots of gloss (GU unit) of 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 ($p < 0.05$)

The comparison of different types of microhybrid resins can be criticized in the literature, due to technological progress and appearance of new materials with nanoparticles, since the incorporation of nanoparticles reduces the polymerization shrinkage.[14] However, some studies have found that microhybrid resins present similar results compared with resins that have nanoparticles or microfilled,[4,14,15] although microhybrid resins have presented greater color stability when compared with nanocomposites and microfilled composites.[16]

The first null hypothesis tested was rejected, since all microhybrid resins showed increase in surface roughness values after challenges, corroborating with other studies that analyzed the chemical challenge,[17] or mechanical degradation, separately.[9,18] Before challenges, no difference was observed among composite resins evaluated; however, after challenges, greater changes were observed for Charisma (Fig. 1A), which could be explained by the large percentage of TEGDMA in its composition, causing greater absorption of liquids due to the higher hydrophilic capacity.[19]

Chemical challenges were performed using low pH solutions, which may have favored the degradation of the polymer chain, contributing to the abrasive action of toothbrushing.[7] Opallis resin has greater particles size reaching 3 µm, presenting less surface wear within the matrix, different from Charisma composite as observed in Figures 1A and D. Amelogen Plus and Point 4 showed lower Ra values, presenting similar surface topography as can be seen in Figures 1B and C. Although there was increase in surface roughness after challenges, it was observed that all microhybrid resins evaluated showed values lower than 0.2 µm with no influence to increase the plaque buildup.[6] In this sense, these microhybrid composites are clinically indicated, even after the ultimate challenges.

Regarding gloss, similar results were observed between microhybrid composites before challenges, but all composites also presented decreased gloss values after challenges, rejecting the second hypothesis. These results are in agreement with some studies that observed the influence of acid solutions,[6,15] and toothbrushing[15,20] in gloss change. Point 4 resin showed the lowest final surface roughness (Fig. 1C), which may have further contributed to its highest gloss, since an inverse linear relationship between gloss and roughness has been cited in the literature, due to the increase in surface irregularity affecting the reflection of light.[12,21] However, the gloss is influenced not only by surface roughness, but also by differences in the refractive indices of the matrix and particles present in each resin,[22] which could account for the higher loss of gloss for the resin Amelogen Plus, even with low surface roughness after challenges (Fig. 1B). The present study is also in agreement with other studies[23] that reported higher gloss values regardless of the surface roughness.

The third null hypothesis was rejected, since color changes were observed after association of challenges. The Charisma resin showed greater overall color change, with statistically significant difference compared with other resins. These results can be explained due to the higher surface roughness values presented by this resin, increasing the possibility of impregnation of pigments,[12] and the abovementioned largest hydrophilic capacity.[19] However, all resins evaluated in this study presented ΔE values below 3.7, being considered clinically acceptable.[24]

An *in vitro* study might not represent all the conditions and interactions acting on the restorative material in a complex environment, such as the oral cavity, with the influence of other fluids, enzymes, and proteins present in the saliva.[25] In this sense, randomized clinical trials are required in order to assess these studied parameters. Therefore, results obtained with this present study should provide valuable information for clinicians to make decisions in selecting the best materials for esthetic restorations for their patients.

CONCLUSION

The composite resins were influenced by association of chemical and mechanical challenges for surface roughness, gloss, and overall color change. The Charisma composite showed the highest changes for surface roughness, gloss, and color when compared with the other microhybrid composites.

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