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Influence of pigment solutions on color stability and surface properties in low-shrinkage and conventional composites

Luciana L Meneghel[1], Ana PP Fugolin[2], Sandrine B Berger[1], Américo B Correr[3], Delise Pellizzaro[4], Karen BP Fernandes[1], Guilherme Genovez-Júnior[1], Allan IF Piauilino[5], Ricardo D Guiraldo[1]

1. Universidade Pitágoras Unopar-UNOPAR, Faculdade de Odontologia, Departamento de Odontologia Restauradora, Londrina, PR, Brasil
2. Biomaterials and Biomechanics, School of Dentistry, Oregon Health & Science University, Portland, OR, USA
3. Universidade de Campinas-UNICAMP, Faculdade de Odontologia de Piracicaba, Piracicaba, SP, Brasil
4. Universidade Estadual de Londrina-UEL, Faculdade de Odontologia, Departamento de Odontologia Restauradora, Londrina, PR, Brasil
5. Universidade de Cuiabá-UNIC, Faculdade de Odontologia, Departamento de Odontologia, Cuiabá, MT, Brasil

ABSTRACT

Color stability is among the most frequent causes of restoration failures, and influences surface properties. ***Aim:*** *The aim of this study was to investigate the influence of pigment solutions on low-shrinkage and conventional composites regarding changes in the physical properties of composite surfaces.* ***Materials and Method:*** *Specimens of four composites (Filtek Z350 XT, Point 4, N'Durance and Venus Diamond) were randomly distributed into three groups to be submitted to each of three pigment solutions (red wine, tomato sauce and coffee) in fifteen-minute daily cycles, for twenty-eight days. There were 12 groups altogether (n = 10). Color, surface roughness and hardness tests were performed. Statistical analysis included Analysis of variance (ANOVA) and Tukey's significance test (α = 0.05).* ***Results:*** *Color changes caused by the solutions did not differ significantly among Filtek Z350 XT, Venus Diamond and N'Durance. Hardness decreased significantly in Filtek Z350 XT and Venus Diamond after chemical challenge with each solution. For the composite independent factor, roughness was highest in Venus Diamond, followed by Filtek Z350 XT, Point 4 and N'Durance.* ***Conclusions:*** *Treatment with different pigment solutions (red wine, tomato sauce or coffee) increased stainability and decreased hardness of both low-shrinkage and conventional composites, while roughness was unaffected.*

Keywords: *composite resins - surface properties - hardness.*

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Corresponding Author:
Ricardo Danil Guiraldo
rdguiraldo@gmail.com

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Influência de soluções pigmentantes na estabilidade de cor e propriedades de superfície em compósitos convencionais e de baixa contração

RESUMO

A estabilidade de cor está entre as causas mais frequentes de falhas de restaurações, que também influenciam suas propriedades superficiais. ***Objetivo:*** *O objetivo do presente estudo foi investigar a influência de soluções de pigmentos em compósitos convencionais e de baixa retração, bem como alterações nas propriedades físicas da superfície dos compósitos.* ***Materiais e Método:*** *Amostras de cada compósito (Filtek Z350 XT, Point 4, N'Durance e Venus Diamond) foram distribuídas aleatoriamente em grupos submetidos a cada solução pigmentante (vinho tinto, molho de tomate e café) em ciclos diários de quinze minutos, durante vinte e oito dias. Assim, totalizando 12 grupos (n = 10). Foram realizados testes de cor, rugosidade superficial e dureza. A Análise Estatística foi realizada usando Análise de variância (ANOVA) e o teste de significância de Tukey (α = 0.05).* ***Resultados:*** *As alterações de cor desencadeadas pelas soluções investigadas não mostraram diferença estatisticamente significativa entre os compósitos Filtek Z350 XT, Venus Diamond e N'Durance. Os valores de dureza registrados para Filtek Z350 XT e Venus Diamond diminuíram significativamente após o desafio químico com cada uma das soluções pigmentantes. Para o fator independente compósito, Venus Diamond registrou a maior rugosidade; foi seguido por Filtek Z350 XT, Point 4 e N'Durance.* ***Conclusões:*** *Os tratamentos das amostras com diferentes soluções pigmentantes (vinho tinto, molho de tomate e café) aumentaram a manchabilidade dos compósitos convencionais e de baixa retração e diminuíram sua dureza, embora não tenham afetado a rugosidade dos compósitos.*

Palavras-chave: *resina composta - propriedades de superfície - dureza.*

INTRODUCTION

Polymerization shrinkage in restorative composites, which depends on filler content and degree of conversion[1], poses a major clinical challenge and cause for concern in restorative dentistry. Contraction and increased stiffness of polymer networks generate physical stress at the tooth/restoration interface and decrease restoration longevity[2]. Composites with low polymerization shrinkage were developed to minimize polymerization shrinkage issues.

Color is an important parameter of modern filler-based restorative materials. It can change due to the intake of large amounts of artificially colored drink and food[3]. Color stability, which is directly related to resin matrix structure and filler particle features, is one of the causes of restoration failures, also affecting surface smoothness and susceptibility to staining by extrinsic factors[4].

Diet is one of the main extrinsic factors affecting resin color because the composite surface layer absorbs pigments from exogenous sources[5]. The influence of diet has been extensively researched in composites with traditional polymerization shrinkage rates[6]. The ability of composites to absorb water also enables them to absorb other fluids, resulting in discoloration, organic matrix degradation and reduced mechanical properties[7,8], even in nanoparticulate resins[9]. One method for measuring color change (ΔE^*) is through Hunter's equation. Values above 3.3 for this measurement are not clinically acceptable[10].

Although there are studies on the effects of exposing methacrylate-based resins to pigment solutions, the effects of exposing low-shrinkage composites have not yet been fully explored. Thus, the aim of the current study was to investigate the influence of pigment solutions (red wine, tomato sauce and coffee) on low-shrinkage and conventional composites regarding changes in the physical properties (color change, surface roughness, microhardness) of the composite surface. The null hypotheses tested were that different pigment solutions would not influence (1) color change, (2) surface roughness or (3) microhardness of low shrinkage and conventional composites.

MATERIALS AND METHOD

Specimen preparation

The study was performed on specimens of the low-shrinkage composites Venus Diamond (Heraeus Kulzer, South Bend, IN, USA) and N'Durance (Septodont, Louisville, CO, USA), and the conventional composites Point 4 (Kerr, Orange, CA, USA) and Filtek Z350 XT (3M ESPE, St. Paul, MN, USA). All composites were shade A2. Composite features are specified in Table 1.

Thirty cylindrical specimens were prepared from each composite. The material was inserted (in a bulk increment) in 5.0-mm-diameter and 2.0-mm-tall molds using polyester strips (base and top), and photoactivated with a light-emitting diode curing unit (Radii Cal; SDI, Bayswater, Victoria, VIC, Australia) for 40 seconds (1400 mW/cm^2); the light source was kept perpendicular to the specimen surface. Photoactivation and finishing procedures were only performed on the top of each specimen. The base of each specimen was marked with small grooves to identify the material. The specimens were stored in distilled water at 37ºC for 24 hours, after which the top surface was finished using the following procedures: water sandpaper, grits 600, 1200 and 2000 for 1 minute each; sequential abrasive discs (Sof-Lex Pop-On; 3M ESPE, St. Paul, MN, USA) grits medium, fine and superfine for 20 seconds each; and felt disc (Diamondflex; FGM, Joinville, SC, Brazil) with diamond polishing paste (Diamond Excel; FGM) for 20 seconds. The specimens were subjected to ultrasonic washing to remove any finishing and polishing debris.

The specimens of each composite were randomly distributed into three groups, to be subjected to each colored solution: red wine (RW), tomato sauce (TS) or coffee (CO), thus totaling 12 groups (n = 10). Specimens were stored individually in plastic containers filled with 25 ml of distilled water at 37 ºC. Color, roughness and hardness of each sample were evaluated.

Color change measurement

Color was measured in digital spectrophotometer (VITA Easyshade Advance; Zahnfabrik, Bad Sackingen, Germany) before (baseline) and after exposing the specimens to the colored solutions. The device records the values L^*, a^* and b^*, based on parameters set by the International Commission on Illumination (CIE - *Commission Internationale de l'Éclairage*). According to the CIE L*a*b* method, color is analyzed 3-dimensionally on the following coordinates:

Table 1. Features of composites used in the current study.

Composite	Filtek Z350 XT	Point 4	Venus Diamond	N'Durance
Polymerization Shrinkage	Conventional	Conventional	Low	Low
Monomer Composition	Bis-GMA, UDMA Bis-EMA, TEGDMA PEGDMA	Exact composition not informed by manufacturer	TCD-DI-HEA UDMA	Dimer Dicarbamate Dimethacrylate (DADMA) Bis-EMA, UDMA
Particle Composition	Silica, zirconia and zirconia/ silica clusters	Colloidal silica, barium, aluminum-boron silicate	Glass particles, barium-aluminum-fluorine	Glass particles of barium, Ytterbium fluoride and silica
Particle Type	Nanofilled	Microhybrid	Nanohybrid	Nanohybrid
Average Particle Size	20 ηm, 4 -11 ηm 0.6-1.0 μm	0.4 μm	5 ηm - 20 μm	10 ηm - 500 ηm
Particle Volume (%)	63.3%	58%	64%	65%
Manufacturer	3M ESPE, St Paul, MN, USA	Kerr, Orange, CA, USA	Heraeus Kulzer, South Bend, IN, USA	Septodont, Louisville, CO, USA
Lot Number	#1404200572	#4948994	#010046	#092412A

– L^*: Luminosity, which determines black and white variations at different times;
– a^*: which determines red (positive values) and green (negative values) variations at different times;
– b^*: which determines yellow (positive values) and blue (negative values) variations at different times.

After the measurement procedure, the specimens were placed back in their individual containers. They were divided into groups, based on composite and pigment solution, and subjected to daily submersion cycles as follows: specimens were removed from the plastic containers, gently dried with absorbent paper and plunged into new containers filled with 25 ml[11] of colored solution (red wine, tomato sauce or coffee) at room temperature for 15 minutes[12]. Then, they were removed from the solution, washed in distilled water and placed back in their original containers. Cycling procedures were repeated for 28 days[11]; solutions and distilled water were changed every 5 days.

– Color variation (ΔE^*) was determined through Hunter's equation:
* $\Delta E_{ab}^* = [(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2]^{1/2}$
– Color variations (ΔE^*) were classified as follows[10]:
– $\Delta E^* < 1$: color changes undetectable to the human eye;
– $\Delta E^* < 3.3$: clinically acceptable color changes;
– $\Delta E^* > 3.3$: clinically unacceptable color changes resulting in the need of resin replacement due to poor aesthetics.

Roughness Test

A roughness meter (SJ-410; Mitutoyo, Tokyo, Japan) was used to assess surface roughness in each specimen after it was gently dried. The device was adjusted to perform a straight trajectory of 0.25 mm, with five repetitions at a speed of 0.1 mm/s. Mean Roughness (Ra) in µm was calculated from readings carried out in three different directions. Mean roughness was measured before and after cycling in pigment solutions.

Microhardness Test

Hardness was tested by measuring indentations produced on the top surface of each specimen (40x magnification) using a microhardness testing machine (HMV-G; Shimadzu, Kyoto, Japan) under a load of 50 g at loading time of 5 seconds. Each specimen was gently dried, after which three indentations were made. The Knoop hardness was expressed as the mean of three indentations made on the same sample. Microhardness was measured before and after cycling in pigment solutions.

Statistical Analysis

Statistical analysis was performed in the SAS System for Windows 9.0 (SAS Institute Inc., Cary, NC, USA).

Two-way ANOVA was applied for color change (factors: composite and solution), and three-way ANOVA for surface roughness and hardness (factors: composite, solution and cycling); followed by Tukey's significance test ($\alpha = 0.05$) for all tests.

RESULTS

Color Changes

Color change values are shown in Table 2. For coffee, the composites Point 4 and N'Durance underwent greater color change than Filtek Z350 XT and Venus Diamond. For tomato sauce, the composites Venus Diamond and Filtek Z350 XT underwent weaker color change than Point 4, while color change in N'Durance did not differ from the others. For red wine, color change was similar in the composites Venus Diamond, Filtek Z350 XT and N'Durance, though weaker than in Point 4.

Surface Roughness

Mean roughness and its standard deviation are shown in Table 3. Venus Diamond was the roughest, followed by Filtek Z350 XT, Point 4 and N'Durance. The differences among these composites were statistically significant. The pigmented solutions did not affect composite surface roughness.

Microhardness

Hardness values for Filtek Z350 XT and Venus Diamond decreased significantly after chemical challenge with each colored solution, as shown in Table 4. Hardness decreased in all composites treated with coffee. For tomato sauce, hardness decreased in Point 4, Filtek Z350 XT and Venus Diamond. For red wine, hardness decreased in Filtek Z350 XT and Venus Diamond.

Table 2. Means and standard deviation for composite color changes (ΔE) with each pigment solution.

Composite	Solution		
	Coffee	Tomato Sauce	Red Wine
Point 4	4.20±0.41 $^{a, B}$	4.56±0.25 $^{a, B}$	5.85±0.68 $^{a, A}$
Filtek Z350 XT	2.73±0.37 $^{b, A}$	3.38±0.42 $^{b, A}$	3.84±0.89 $^{b, A}$
N Durance	3.62±0.40 $^{a, A}$	4.00±0.87 $^{ab, A}$	4.31±0.49 $^{b, A}$
Venus	2.51±0.51 $^{b, A}$	3.16±0.44 $^{b, A}$	3.75±0.85 $^{b, A}$

Mean values followed by different uppercase letters in rows and lowercase letters in columns are significantly different at p<0.05 (Tukey test).

Table 3. Mean roughness and standard deviation (Ra, µm) before (initial roughness) and after (final roughness) treatment.

Composite	Solution	Initial Roughness	Final Roughness
Point 4 (γ)	Coffee	0.12±0.02 $^{a, A}$	0.12±0.03 $^{a, A}$
	Tomato Sauce	0.12±0.02 $^{a, A}$	0.12±0.02 $^{a, A}$
	Red Wine	0.12±0.01 $^{a, A}$	0.13±0.01 $^{a, A}$
Filtek Z350 XT (β)	Coffee	0.13±0.01 $^{a, A}$	0.14±0.02 $^{a, A}$
	Tomato Sauce	0.13±0.02 $^{a, A}$	0.14±0.02 $^{a, A}$
	Red Wine	0.13±0.03 $^{a, A}$	0.14±0.02 $^{a, A}$
N Durance (δ)	Coffee	0.10±0.01 $^{a, A}$	0.11±0.01 $^{a, A}$
	Tomato Sauce	0.10±0.01 $^{a, A}$	0.10±0.01 $^{a, A}$
	Red Wine	0.11±0.01 $^{a, A}$	0.12±0.01 $^{a, A}$
Venus (α)	Coffee	0.17±0.02 $^{a, A}$	0.18±0.02 $^{a, A}$
	Tomato Sauce	0.17±0.02 $^{a, A}$	0.18±0.02 $^{a, A}$
	Red Wine	0.17±0.01 $^{a, A}$	0.18±0.02 $^{a, A}$

Mean values followed by different uppercase letters in rows and lowercase letters in columns are significantly different at p<0.05 (Tukey test).
Different Greek letters indicate significant difference between composites for the composite independent factor at p<0.05 (Tukey test).

Table 4. Means and standard deviation for Knoop hardness (KHN).

Hardness	Composite	Coffee	Tomato Sauce	Red Wine
Initial Hardness	Point 4	74.3* (7.3) Ab	71.0* (5.8) Abc	68.7 (7.5) Ac
	Filtek Z350 XT	92.0* (4.3) Aa	92.8* (3.9) Aa	93.7* (3.8) Aa
	N Durance	73.1* (4.0) Ab	70.2 (3.2) Ac	71.9 (4.1) Ac
	Venus	89.0* (4.8) Aa	81.0* (11.0) Ab	82.2* (9.0) Ab
Final Hardness	Point 4	60.3 (5.5) Ab	58.1 (7.4) Ab	63.0 (7.0) Aa
	Filtek Z350 XT	73.1 (7.2) Aa	69.6 (7.8) Aa	69.8 (4.8) Aa
	N Durance	57.1 (4.4) Ab	66.8 (4.2) Aab	63.7 (5.5) Aa
	Venus	63.8 (6.1) Aab	64.0 (6.5) Aab	61.8 (3.8) Aa

Different lowercase letters in the same column (comparison between composites within initial hardness or final hardness) and uppercase letters on the same rows indicate significant difference at $p<0.05$ (Tukey test). * Indicates significant difference between initial and final hardness in each resin and solution at $p<0.05$ (Tukey test).

DISCUSSION

In vitro studies generally involve limitations. There is divergence in the literature regarding the selection of ideal pigment solutions for replicating composite behavior as realistically as possible. Coffee, tomato sauce and red wine solutions were selected for the current study. The results show (Table 2) that the tested solutions produced clinically unacceptable color changes ($\Delta E^*> 3.3$)[10] in all selected composites. Schmitt et al.[13] found similar results in a study evaluating the stain resistance of micro-hybrid and nanoparticulate resins subjected to multi-step polishing. Arreghi et al.[14] found similar results in nanohybrid and bulk-fill flowable composites exposed to tea, coffee, Coke, red wine and orange juice for six months.

Although many foods contain dyes, different dye types available on the market can lead to different consequences. In line with results found in the current study, Ardu et al.[15] showed that red wine has greater staining potential than coffee, probably because red wine contains tannin and anthocyanin, which may have a significant effect on the color change of the composite during aging, as well as high discoloration power.

Although the results for coffee were not significant, Asmussen et al.[16] reported that coffee may have increased staining potential if the solution is kept at 60ºC. All solutions used in the current study were kept at room temperature (20ºC); so further research may be required to clarify this point. In addition, although filler particles do not absorb water, they can play an important role in composite susceptibility to staining due to weak matrix-filler bond[15].

Color in composites treated with tomato sauce (Table 2) did not differ significantly. The absolute values for color change were clinically unacceptable. Tomato sauce was used because it is often included in diets around the world, and its effect on color change in resinous composites has not yet been studied. Its acidic pH may influence composite color change, since it is associated with the discoloration mechanism[15]. Another study used tomato-based sauce (ketchup) on micro-hybrid resin and recorded significant color change[11]. Similarly, other acid-pH condiments found in diets worldwide, particularly Asian diets, were also found to cause color changes in nano and microparticulate composites[17].

Coffee and red wine stains in the current study (Table 2) match data published in the literature, according to which red wine causes greater color change than coffee[9,18,19]. However, Aguiar et al.[9] found significantly higher pigment concentration in specimens stained with coffee and cola-based soda than in those subjected to red wine. It is worth mentioning that the presence of alcohol in red wine can lead to monomer removal from the surface of the composite resin, enable the absorption of pigmentation agents, and consequently, increase resin wear[20-22]. According to Benetti et al.[23] and Da Silva et al.[24], contact with alcohol influences the color stability and susceptibility to staining of methacrylate-based composites. Asmussen et al.[20] and Guiraldo et al.[1] described the action of alcohol on the polymeric network of the organic matrix of different resins, suggesting that their effects can be indirectly assessed by measuring polymer softening during exposure to alcohol. The degree of conversion from double to single bonds between carbons in the organic matrix affects polymerization shrinkage[25]. Increased degree of conversion is directly associated with increased contraction stress in composites[26]. The degree of conversion depends on factors such as material composition, monomer type, filler particle type and quantity, filler particles/organic matrix interactions, degree of conversion and polymerization techniques[2,27]. Although degree of conversion is an important factor, it does not enable full polymeric network structure characterization.

There was no significant difference in mean roughness values before and after the treatment with coloring substances (Table 3), possibly as a result of the finishing and polishing procedure applied. Finishing and polishing procedures influence materials' properties. When the specimens were prepared, compression against a polyester strip provided a smooth surface. Polishing alone would be enough to explain the low roughness values recorded[28]. Composite resin polishability is influenced by the type, shape and content of filler particles, and, due to the spherical shape of their particles, microparticulate composites are more efficiently polished than hybrids[3,29,30]. Results in the present study show that particle size does not appear to have affected polishing degree, since one nanohybrid composite (Venus Diamond) presented higher mean roughness than another microhybrid, as well as statistical differences in mean roughness in comparison to other nanohybrid composites. The values recorded herein agree with Berger et al.[31], who found no association between filler particle size and composite surface roughness. However, the association between polishing and particle size was observed when roughness was significantly lower in the nanofilled composite 'Filtek Supreme XT' than in the microhybrid after they had both undergone multi-stage polishing systems[13]. Similarly, Alkhadim et al.[32] did not observe major differences in the surface roughness of samples polished in a rotary system.

The composition of composite resins has some advantages related to nanometric particles: low polymerization shrinkage, surface smoothness, less wear, better color stability and improved mechanical properties[33]. In addition, particle geometry has direct impact on surface smoothness and stain resistance[5]. The use of nanoparticles in clusters (nanoclusters) reduces the space between particles, increases particle filler rates and improves the physical properties of the tested material[13]. These features may explain the roughness values recorded for Filtek Z350 XT in the present study, which were lower than the ones recorded for composite 'Venus Diamond'.

The Knoop hardness test was used in the present study because it provides a good estimate of a monomer's degree of conversion after polymerization, which directly affects mechanical properties of composites[34]. Insufficient monomer polymerization can lead to poor color stability, risk of pulp aggression by unpolymerized monomers, susceptibility to staining and regions presenting different Young's modulus values, among other issues[35]. Decreased microhardness values recorded before and after tests conducted with different solutions (Table 4) can be attributed to the chemical composition of the materials and its effects on different resin components[36]. In addition, the polymeric matrix was highly susceptible to softening due to chemical action. Polymeric matrix damage depends on diffusion rates based on the molecular weight of the tested material[21], which can compromise the other properties evaluated in the current study. Composites based on dimethacrylates presented a fast-formed cross-linked network during polymerization, which restricted reaction mobility[37]. These networks swell when they are exposed to solvents, because the attraction between them and solvent molecules is stronger than the attraction between them and polymers[34]. It may therefore be concluded that the solvent permeates the organic matrix. Thus, null hypotheses (1) and (3) were rejected because different pigment solutions led to changes in color and microhardness, whereas hypothesis (2) was accepted because the pigment solutions did not influence surface roughness.

CONCLUSION

The current study found that treating low-shrinkage and conventional composite specimens with different pigment solutions (red wine, tomato sauce or coffee) increased stainability, decreased hardness, and did not affect roughness.

DECLARATION OF CONFLICTING INTERESTS

The authors declare no potential conflicts of interest regarding the research, authorship, and/or publication of this article.

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