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Discoloration of *Bulk*-Fill Versus Conventional Composites: A Spectrophotometric Evaluation

Decoloración de resinas *Bulk* vs resinas convencionales: Evaluación espectrofotométrica

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ABSTRACT: Purpose: The purpose of this study was to evaluate the discoloration of a dual-cure bulk-fill (DCBF) and light-cure bulk-fill (LCBF) composites relative to conventional composites (CC) after water and coffee immersion. Methods: One-hundred disc-shaped specimens (8mm in diameter × 4mm thickness) were prepared using five commercially available composites (n=10); two LCBF (XB-X-trafil Voco, FB-FiltekTM Bulk Fill), one DCBF (FU-Fill-upTM Coltene), and two CC (CE-Clearfil Majesty ES2, EQ-Estelite ΣQuick). Initial and final color readouts were measured with a spectrophotometer (Easyshade,Vita) according to the CIELAB color system. Statistical analyses were performed using 2-way ANOVA with Bonferroni adjustment to evaluate ΔE, ΔL, Δa, and Δb parameters ($p<0.05$). Results: Significant interactions were noted among the material types and immersion solutions for all evaluated parameters ($p=0.00$). ΔE values were observed in the order of FU>FB≥XB>EQ≥CE in water immersion groups and XB>FB>EQ≥FU≥CE in coffee immersion groups. Conclusion: Within the limitations of this study, DCBF composite is more susceptible to intrinsic discoloration compared to LCBF and CC. However, DCBF exhibited lower extrinsic discoloration than LCBF and comparable with CC after coffee immersion. Depending on their chemical and structural compositions, composites will exhibit color change, which compromises the esthetic performance of composite restorations.

KEYWORDS: Color stability, bulk-fill composites, dual-cure composites, discoloration, staining, intrinsic discoloration.

RESUMEN: Propósito: El propósito de este estudio fue evaluar la decoloración de resinas bulk de curado dual (DCBF) y bulk activadas por fotopolimerización (LCBF) en relación con las resinas convencionales (CC) después de la inmersión en agua y café. Métodos: Se prepararon cien muestras en forma de disco (8mm de diámetro × 4mm de espesor) utilizando cinco resinas compuestas disponibles comercialmente (n=10); dos LCBF (XB-X-trafil Voco, FB-FiltekTM Bulk Fill), un DCBF (FU-Fill-upTM Coltene) y dos CC (CE-Clearfil Majesty ES2, EQ-Estelite ΣQuick). Las lecturas de color inicial y final se midieron con un espectrofotómetro (Easyshade, Vita) de acuerdo con el sistema de color CIELAB. Los análisis estadísticos se realizaron utilizando ANOVA de 2 vías con ajuste de Bonferroni para evaluar los parámetros ΔE, ΔL, Δa y Δb ($p<0,05$). Resultados: Se observaron diferencias significativas entre los diferentes tipos de resina y las soluciones de inmersión para todos los parámetros evaluados ($p=0,00$). Se observaron valores de ΔE en el orden de FU>FB≥XB>EQ≥CE en los grupos de inmersión en agua y XB>FB>EQ≥FU≥CE en los grupos de inmersión en café. Conclusión: Dentro de las limitaciones de este estudio, la resina DCBF es más susceptible a la decoloración intrínseca en comparación con LCBF y CC. Sin embargo, DCBF mostró una decoloración extrínseca más baja que LCBF y comparable con CC después de la inmersión en café. Dependiendo de sus composiciones químicas y estructurales, los composites mostrarán cambios de color, lo que compromete el desempeño estético de las restauraciones de resinas con el tiempo.

PALABRAS CLAVE: Estabilidad del color; Resinas compuestas; Tinción; Decoloración intrínseca.

INTRODUCTION

Composite resins have been successfully used in dentistry for direct restorations for many years. Since their introduction, manufacturers have been continuously motivated to improve their mechanical, physicochemical, and optical properties. Mechanical properties and volumetric shrinkage are limitations of composite resins. Although the contribution of new-filler technologies improved the wear and fracture strength of composites (1,2), evolution in the organic phase has been reported to decrease volumetric shrinkage (3). Internal contraction stress due to volumetric shrinkage developed during polymerization can cause many clinical problems, such as marginal gap formation, debonding, postoperative sensitivity, microleakage, and cusp deflection (2,3).

An ideal composite should exhibit a high degree of conversion (DC) and a minimal volumetric shrinkage due to polymerization. The volumetric shrinkage stress depends on multiple factors; the initiator type and concentration, monomer species, filler characteristic, and composite's DC and elasticity modulus (3). Bulk-fill composites have been recently developed as a new category of composite resins. Conventional light-cure resin composites have been recommended to be applied incrementally in 2mm thick layers to the cavity. However, light-cure bulk-fill (LCBF) composites have been reported to be able to overcome incremental placement with a single layer up to 4-5mm thickness without adverse effect on polymerization shrinkage and degree of conversion (4). The improved formulation of LCBF composites is obtained through high photo-initiator content or

an additional initiator type, greater translucency, and the use of different types of fillers (5).

Manufacturers recently introduced another type of bulk-fill composites called dual-cure bulk-fill composites. The dual-cure bulk-fill (DCBF) composites can be inserted as a single layer and eliminate insufficient polymerization at deep layers. These DCBF composites contain both light- and chemical-cure ingredients. The photoactivation provides fast polymerization of the superficial layer of the composite resin and initial stabilization of the restorations; then chemical cure components provide a slower chemical-cure reaction that completes the polymerization in deeper layers of the composite restoration (6,7) .

In the available literature, a new category of composites called bulk-fill composites' exhibited similar or better performance in physicochemical properties compared to conventional composite resins (8). According to systematic reviews and meta-analysis, it was reported that under clinic conditions, the survival of bulk-fill composites for direct posterior restorations were similar to that of conventional composites (8, 9). However, their optical properties and color stability have not yet been thoroughly studied. Unpredictable color stability and stainability of composite restorations are one of the main reasons for replacement.

The discoloration of composite restorations might be caused by intrinsic and extrinsic factors. Intrinsic factors include the choice of photoinitiator system, matrix composition, filler loading, particle size distribution, duration of polymerization, and conversion of the matrix monomers (10,11). Extrinsic factors include staining solutions (e.g., tea, coffee, red wine) smoking and oral hygiene habits. Also color alteration of composites can be attributed to water sorption, dehydration, quality of polymers, chemical degradation, and oxidation of unreacted C=C bonds (12).

As improved formulations of new category resin-based composites are developed, it is essential that studies evaluate their color properties, such as color match and color stability, for the success of esthetic restorations. So far, there is limited data available investigating the color stability of the bulk-fill composites and the lack of information in the literature regarding the optical properties of DCBF composites. The aim of the present study was to evaluate the color stability of a DCBF composite in comparison with LCBF composites and conventional composites after water and coffee immersion. The null hypothesis was: there will be no differences in color stability among all three types of composites after water and coffee immersion.

MATERIAL AND METHODS

SPECIMEN PREPARATION

Five commercially available composite resins were evaluated, including two LCBF composites (X-tra fil Voco, FiltekTMBulk Fill), one DCBF composite (Fill-upTM), and two conventional composites (Clearfil Majesty ES-2, Estelite Σ Quick). All bulk-fill composites are suitable for direct restoration without a superficial capping layer. The composite's chemical compositions are listed in Table 1. One-hundred disc-shaped specimens (8 mm in diameter × 4mm thickness) were prepared for composites listed in Table 1 to form 10 experimental groups (n=10).

All specimens were prepared according to the manufacturer's instructions. The composite was inserted into the mold in 2mm-thick for incremental composites (CE, EQ) and in 4 mm-thick for bulk-fill composites (XB, FB, FU). To achieve a flat surface and eliminate the oxygen inhibition layer, a Mylar strip and microscope glass slide were applied to the upper surface of composite with finger pressure and light-cured 10 s with a light-curing device that provides 1200 mW/cm² intensity

(3M ESPE Elipar S10, Germany). For each group, the specimens were polished by using medium, soft, and fine abrasives in wet conditions (Soft-Lex 3M ESPE, Seefeld, Germany). Specimens were polished under slight pressure and during polishing discs were changed after every five specimens. All procedures were carried out by the author due to the manufacturer's recommendations. After the polishing process, the specimens were rinsed in an ultrasonic cleaner for 10 min.

MEASUREMENT OF COLOR PARAMETERS OF SPECIMENS

The CIELAB color system (CIE: The Commission Internationale de l'Eclairage) evaluates color differences based on the coordinates L*, a*, and b*. The color difference values (ΔE*) expressed between initial and different intervals from the mean ΔL*, Δa*, and Δb* values for each specimen and calculated using the following formula:

$$\Delta E = [(\Delta L^*)^2+(\Delta a^*)^2+(\Delta b^*)^2]^{1/2}$$

L* value measures the lightness coordinate, whereas a* and b* are chromaticity coordinates. a* is a measure of green-red coordinate; and b* is a measure of blue-yellow coordinate (13). After obtaining the test specimens, baseline color measurements were measured (Spectrophotometer Easy Shade, VITA Zahnfabrik, Germany) according to the CIELAB system as initial color measurement. In water immersion (W) groups (FUW, FBW, XBW, CEW, and EQW), color measurements were made after storage in water at 37°C for 1wk in an incubator. The spectrophotometer was calibrated according to the manufacturer's recommendations then, the probe tip was placed in the center of the specimens, and color parameters were measured. Measurement was done inside a booth using standard illuminant D65 on a neutral gray background based on International Organization for Standardization standard 7491 (14). Three measurements were done consecutively for each specimen, then the mean values were determined as the initial color of the specimen.

In the coffee immersion (C) groups (FUC, FBC, XBC, CEC, and EQC), ten specimens from each material were stored at 37°C and 100% relative humidity for 24 h before initial color measurements, then immersed for 7 days in the prepared coffee solution. The staining solution prepared with 15g coffee (Nescafe Classic, Nestle, Switzerland) was dissolved in 500ml of boiling distilled water, and each group of specimens was stored at 37°C for 1-wk in an incubator (15). The solution was freshly made and replaced daily. Color measurements of specimens were made after 1-wk coffee immersion. Before the measurement, the specimens were rinsed in distilled water for 1 min and dried with absorbent paper. Three measurements were done consecutively for each specimen, then the mean of values was determined.

STATISTICAL ANALYSIS

Statistical analyses were performed with SPSS for Windows 17.0 (SPSS Inc., Chicago, IL, USA). As data were normally distributed (Kolmogorov-Smirnov, Shapiro-Wilk), and statistical analysis was performed using two-way analysis of variance (ANOVA) with Bonferroni adjustment in order to evaluate differences in ΔE, ΔL, Δa, and Δb between groups. P-values <0.05 were consider statistically significant.

Table 1. Composites used in the study and composition of composites obtained from manufacturers.

Materials	Code	Type	Composition		Filler content	Shade	Manufacturer	Lot No.
			Resin	Filler	wt% / vol%			
x-tra fil	XB	Light-cure bulk-fill composite	Bis-GMA, UDMA, TEGDMA	Ba–B–Al–Si glass	86/70.1	A2	Voco Cuxhaven Germany	1437495
Filtek™ Bulk Fill	FB	Light-cure bulk-fill composite	Bis-GMA, UDMA, Bis-EMA, procrylat resins	Zirconia/ silica, ytterbium trifluoride	64.5/42	A2	3M/ESPE St.Paul, MN USA	N636501
Fill-up™	FU	Dual-cure bulk-fill composite	TMPTMA, UDMA, , Bis-GMA, TEGDMA, dibenzoyl peroxide; benzoyl peroxide	Zinc oxide coated.	65/49	U	Coltene/ Whaledent AG, Altstätten Switzerland	G85305
Clearfil Majesty ES-2 Classic	CE	Light-cure conventional composite	Bis-GMA, dimethacrylate,	Silanated barium glass	78/66	A2	Kuraray Notritake Dental Inc. Okayama Japan	420013
Estelite I Quick	EQ	Light-cure conventional composite	Bis-GMA, UDMA, TEGDMA	Silica zirconia, PFSC	82/71	A2	Tokuyama Dental Corp. Japan, Italy S.r.l. Vicenza, America Inc.	E057

Abbreviations: BisGMA, bisphenylglycidyl dimethacrylate; BisEMA, ethoxylated bisphenol-A dimethacrylate; UDMA, urethane dimethacrylate; TMPTMA, trimethylolpropane trimethacrylate; TEGDMA, triethylene glycol dimethacrylate: PFSC, prepolymerized filler of silica composite.

RESULTS

Each composite and immersion solution combination showed a particular color distribution with proper CIE system coordinates. Significant interactions were noted among the material types and immersion solutions for all evaluated parameters ($P<0.001$). There was a significant difference in ΔE^* between water and coffee immersion ($P<0.001$). The effect of composite type on color change (ΔE^*) after immersion in coffee and water was also found to be highly significant ($P<0.001$).

The means and standard deviation of color coordinates (ΔL^*, Δa^*, Δb^*) and color difference (ΔE^*) values according to the results of the 2-way ANOVA of all groups are presented in Table 2. The lowest color difference was observed in the material CE, while the highest color difference was observed in the material FU at the water immersion group. In the coffee immersion group, the lowest color difference was observed in CE, while the highest color difference was observed in XB.

For all water immersion groups, ΔE^* values were observed in the order of FUW>FBW>XBW>

EQW>CEW. FUW had significantly higher ΔL*, Δa*, Δb*, and ΔE* values (5.55±1.21; 2.46±0.63; 4.65±1.59 and 7.75±1.69 respectively) than all water immersion groups. XBW had the second-highest and FBW the third-highest ΔL* and ΔE* values but without being significantly different among them (P=0.782 and P=0.367 respectively) and significantly higher ΔL* and ΔE* values than CEW and EQW groups. CEW had the lowest ΔE* value (1.43±0.2) and EQW had the second-lowest ΔE* value (1.63±0.7) among all groups and without statistically significant difference among them (p=0.76). Also, no statistically significant differences were found between CEW and EQW groups among the ΔL*, Δa*, and Δb* parameters (p=0.752, 0.306, and 0.446 respectively).

XBC had significantly higher ΔL*, Δb*, and ΔE* values (6.075±1.23, 5.88±1.48, and 8.59±1.74 respectively) than all of the coffee immersion groups. For all coffee immersion groups, the ΔE* values were observed in the order of XBC>FBC>EQC>FUC>CEC. In the coffee immersion groups, no significant differences were observed among the EQ, FU, and CE groups. Total color change (ΔE*) was observed to be increased in the coffee immersion groups except for the FUW group. In the water immersion group, all groups except CE and EQ presented color alteration above the clinically accepted level. In the coffee immersion group, all the groups presented color alteration above the clinically accepted level and no statistically difference between FUC, CEC, and EQC.

Table 2. Mean ± standard deviations of color coordinates (ΔL*, Δa*, Δb*) and color differences (ΔE*) for each composite and immersion solution. Different letters in the same line indicate significant differences between the mean values of groups (p<0.05).

	FU	CE	EQ	FB	XB
Water (W)					
ΔL*	5,55±1,21[a]	0,46±0,95[b]	0,26±1,22[b]	3,81±1,003[c]	3,63±0,73[c]
Δa*	-2,46±0,63[a]	-0,42±0,21[b,c]	-0,18±0,23[b]	-1,26±0,23[c]	-0,82±0,40[c]
Δb*	-4,67±1,59[a]	-0,78±0,54[b,c]	-0,31±1,34[b]	-1,58±0,76[c]	-0,31±0,34[b]
ΔE*	7,751±1,69[a]	1,43±0,26[b]	1,63±0,71[b]	4,37±1,02[c]	3,77±0,76[c]
Coffee (C)					
ΔL*	4,46±1,81[a]	2,95±0,60[b]	4,01±0,90[a]	5,50±2,03[a]	6,07±1,23[c]
Δa*	-1,35±0,86[a]	-0,76±0,43[b]	-0,12±0,15[c]	-1,46±0,33[a]	-1,30±0,23[a]
Δb*	-1,37±1,28[a]	-3,15±1,14[b]	-2,9±1,21[b]	-3,67±1,48[b]	-5,88±1,48[c]
ΔE*	5,70±2,00[a]	4,52±0,62[a]	5,06±1,18[a]	6,88±2,17[b]	8,59±1,74[c]

DISCUSSION

The null hypothesis of this study was rejected since: significant differences in a color change of composite resins were found after coffee and water immersion for one week. The effect of composite type and immersion solution on discoloration was found to be significant (P<0.00).

Under the testing conditions of this in vitro study, color changes after 1-wk water immersion could be evaluated as intrinsic discoloration. Chemical composition, characteristic of monomers, the network configuration of polymers, and conversion degree are important factors that determine the behavior of composite, such as water absorption (12,16). In the present study, conventional composite CE and EQ showed the least color change of all materials after water immersion. High filler content, less resin volume fraction, and amplified photo-polymerization system and less camphorquinone (CQ)/amine use explain the

improved color stability relative to other bulk-fill composites (17).

The degree of water sorption is highly dependent on the type and volume of filler and the chemistry of the monomers, as well as the type of the resin matrix and the bond strength between resin-matrix and filler particles (18). Silica filler content of EQ and pre-silanated glass filler content of CE and also high filler volume (wt82% and wt78% respectively) provides more inert structure in water and improved surface protection from the environment and more hydrolysis-resistant filler-matrix coupling (19).

According to this study, bulk-fill materials showed more susceptibility to discoloration than conventional composites. Different optical properties can be attributed to improved formulations of this new category composites called 'bulk-fill category', such as increased photo-initiator content or an additional photo-initiator type, greater translucency, and the use of different types of fillers (2,4,5,16). It was previously reported that large amounts of CQ as a blue-light-absorbing photosensitizer and an amine initiator in the photoinitiator system of resin composite formulations cause undesirable oxidative color changes (20,21). Modifications of filler type to improve light transmission depth via refractive index match between filler and organic matrix (2,22) to achieve high translucency in bulk-fill composites can cause different optical properties. Modifications to the monomer system in bulk-fill composites that provide to form a crosslinked network to allow for stress relief during polymerization (2) is another factor that is likely to influence the optical characteristics of the composite (12).

In the present study, the color change in conventional incremental composites was lower and considered clinically acceptable; however, bulk-fill composites had a considerable color change in water consistent with the previous report (23). DCBF composite (FU) showed significantly higher initial ΔL^*, Δa^*, Δb^*, and ΔE^* (5.55±1.21, -2.46±0.63, -4.67±1.59, 7.751± 1.69, respectively) values than other all groups (Table 2). FU showed about twice as high ΔE^* value than FB and XB that of other LCBF composites (4.37±1.02 and 3.77±0.76, respectively). In this study, the FU DCBF composite showed higher initial discoloration than LCBF composites XB and FB. There is a lack of information in the literature regarding the color stability of resins with different formulations, especially in the bulk-fill category for comparisons with this study results.

Different from LCBF composites, chemical-curing processes of DCBF composites consist of two parts: faster light-activated polymerization at the top layer and slower chemical-activated polymerization at the deep layers (7). Discoloration of resin over time can be attributed to the addition of self-cure activator and initiators, such as benzoyl peroxide, to ensure slower chemical polymerization of dual-cure resin composites that produce colored oxidation products (21). A major factor of this much darker initial yellow color and a larger color-change of FU can be attributed to the differences in the initiator system.

Coffee and distilled water were chosen as an immersion solution in this study because of the frequent consumption of them in daily life. It was reported that staining of the composite was closely related to water sorption and most of the water sorption was observed during the first week. In the present study, coffee and water were used as an immersion solution for 1-wk. It was reported that 1-wk coffee immersion might simulate more than 7 months of coffee drinking (24).

The two-way ANOVA revealed that the two main factors, immersion solution and material, and their interaction, were significant ($p = 0.00$). The color differences after coffee immersion were different among the products, and all the groups

presented color alteration above the clinically acceptable level. XBF and FB showed greater staining susceptibility than FU, and FU showed comparable staining susceptibility with CE and EQ after coffee immersion. LCBF composites showed greater staining susceptibility after coffee immersion than conventional composites consistent with previous reports (23,25).

Staining susceptibility of composite resins by colorant agents, such as coffee, can be attributed to water sorption and hydrophilicity of the resin matrix and absorption of pigments into the organic phase of composite materials (24,26). In addition, filler type, refractive index of filler, and matrix might be increased after water sorption and responsible for staining susceptibility (27). XB and FB showed higher discoloration than other groups, and this result can be attributed to type of filler particles; it was previously reported that the presence of fillers containing metallic ions would enhance the hydrolytic degradation mechanism (28).

The limitations of this study were that it was performed under *in vitro* conditions, and it is possible that optical properties and behaviors of resin composites in the oral conditions might be different than the results obtained in this in vitro study. This difference can be attributed to insufficient simulation of the oral environment, such as exposure of specimens throughout immersion solution, the cleaning effect of saliva or oral hygiene procedures. Furthermore, this *in vitro* study lacked aging procedures that simulate chewing or thermal changes in oral conditions. Further *in vitro* studies should be performed that incorporate aging procedures and evaluate long-term color stability of light-and dual-cured bulk-fill composites.

Within the limitations of this study, dual-cure bulk-fill composite is more susceptible to intrinsic discoloration compared to light-cure bulk-fill composites and conventional composites. However, dual-cure bulk-fill composites exhibited lower extrinsic discoloration than light-cure bulk-fill composites and were comparable with conventional composites after 1-wk of coffee immersion. Depending on the different chemical and structural compositions, composites will exhibit color change, which compromises the esthetic performance and color match of composite restorations in clinic conditions.

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CONFLICTS OF INTEREST STATEMENT

The author declares that there is no conflicts of interest.

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