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Effects of Acidic Beverages on Color Stability of Bulk-Fill Composites with Different Viscosities

Efectos de las bebidas ácidas sobre la estabilidad del color de resinas tipo Bulk con diferentes viscosidades

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ABSTRACT: The aim of this *in vitro* study is to investigate the effects of acidic beverages on the discoloration of bulk-fill composites with different viscosities. 144 disc-shaped specimens (8mm diameter × 2mm thick) were prepared from four different composite resins including a nanohybrid (Filtek Ultimate), two packable bulk-fill (Filtek Bulk Fill, Tetric N Ceram Bulk Fill), and a flowable bulk-fill composite (Tetric N Flow Bulk Fill). The specimens of each group were divided into 3 subgroups and submerged in distilled water, orange juice and coke (n=12). Color difference (ΔE) were measured using CIELab color space and a compact spectrophotometer (VITA Easyshade) at baseline and after 30 days of immersion. Data were analyzed using two-way ANOVA and Tukey's HSD post-hoc tests ($P < 0.05$). Two-way ANOVA revealed that both material type and staining solution significantly influenced ΔE values ($P < 0.001$). Immersion in orange juice resulted in greater and significant discoloration over time in comparison to distilled water and coke ($P < 0.05$). Tetric N Ceram ($\Delta E = 7.0$) and Tetric N Flow ($\Delta E = 7.7$) bulk-fill restoratives showed highest color change values after 30 days of immersion in orange juice. This study suggests that a discoloration above the clinically acceptable threshold ($\Delta E > 2.7$) was observed for all bulk-fill composites tested. It can be speculated that orange juice has a more negative effect on the color stability compared to coke. Additionally, flowable bulk-fill composite was more prone to discoloration than condensable bulk-fill composites.

KEYWORDS: Composite resins; Color; Dental filling; Dental materials; Dentistry; Food coloring agents.

RESUMEN: El objetivo de este estudio fue investigar los efectos de las bebidas ácidas en la decoloración de resinas compuestas tipo Bulk con diferentes viscosidades. Se prepararon 144 muestras en forma de disco (8mm de diámetro x 2mm de grosor) a partir de cuatro resinas compuestas diferentes que incluyen un nanohíbrido (Filtek Ultimate), dos de pasta condensables tipo Bulk (Filtek Bulk Fill, Tetric N Ceram Bulk Fill) y una fluida (Tetric N Flow Bulk Fill). Las muestras de cada grupo se dividieron en 3 subgrupos y se sumergieron en agua destilada, jugo de naranja y Coca-Cola ($n=12$). La diferencia de color (ΔE) se midió utilizando el CIELab y un espectrofotómetro compacto (VITA Easyshade) al inicio y después de 30 días de inmersión. Los datos se analizaron mediante ANOVA bidireccional y pruebas post-hoc HSD de Tukey ($P<0,05$). El ANOVA bidireccional reveló que tanto el tipo de material como la solución de tinción influyeron significativamente en los valores de ΔE ($P<0,001$). La inmersión en jugo de naranja resultó en una decoloración mayor y significativa con el tiempo en comparación con el agua destilada y la Coca-Cola ($P<0,05$). Las resinas Tetric N Ceram ($\Delta E=7,0$) y Tetric N Flow ($\Delta E=7,7$) mostraron los valores más altos de cambio de color después de 30 días de inmersión en jugo de naranja. En este estudio se observó una decoloración por encima del umbral clínicamente aceptable ($\Delta E>2,7$) para todos los materiales resinosos. Se puede especular que el jugo de naranja tiene un efecto más negativo sobre la estabilidad del color en comparación con la Coca-Cola. Además, la resina fluida fue más propensa a la decoloración que las resinas condensables.

PALABRAS CLAVE: Resinas compuestas; Color; Empaste dental; Materiales dentales; Odontología; Agentes colorantes alimentarios.

INTRODUCTION

Direct composite resin restorations are one of the most preferred treatment options in the restorative dentistry (1). Although there are many different restorative materials in the dental field today, composite resins continue to be the first choice of dentists due to their superior esthetic properties and bond strength to the tooth structures, and enabling conservative tooth preparation (2,3). Conventional composite resins require an incremental placement approach, which is necessary to provide sufficient depth of cure for each placed increment (4,5). Therefore, the placement of composite resin in 2mm increments is recommended to allow adequate light transmission (6). However, it is a time consuming approach, which may also cause void incorporation or moisture contamination between the increments (7,8).

Bulk-fill composite resins that can be cured up to 4 to 5mm increments without compromising the polymerization are introduced to overcome these difficulties (9).

The formulations of bulk-fill composites differ from conventional composites in that they contain stress-relieving monomers, more reactive photoinitiators, prepolymerized filler particles and fiberglass rods (7). In addition, the translucency of the bulk-fill composites has been increased to allow more light transmission. It is claimed that such differences allow bulk-fill composites to have lower polymerization shrinkage compared to conventional composites (10). As a result of their modified activator systems, reduced filler load and increased filler size, activation time is also considerably becomes shorter (11). Although there is no agreed classification on bulk-fill composite resins,

bulk-fill composites with mechanical properties similar to flowable composites are defined as low viscosity and those similar to packable composites are defined as high viscosity bulk-fill composites (12). Nowadays, high-viscosity bulk-fill composites have become highly preferred resins due to their resistance to intraoral conditions (13). In addition to the beneficial single increment placement of bulk-fill composites, one of the major disadvantages is that they have insufficient aesthetic properties because of their limited color options (14). For this reason, manufacturers have introduced bulk-fill composites with a blending effect which refers to its ability to acquire a color resembling similar to the adjacent tooth structure (15).

Composite resin materials used in direct restorations are expected to have satisfactory aesthetic properties as well as strength and durability. Although improvements have been made in the organic matrix and filler particles of composite resins, color change is still a current problem for both patients and dentists. Exposure of composite resins to various beverages in the oral environment may result in color change due to the interactions occurring on the material surface (16). Color matching and anatomical form are essential for predicting the clinical life of composite resin restorations and are also included in the parameters used by the United States Public Health System (USPHS) to evaluate the quality of existing restorations (17).

Superficial discolorations may occur due to the accumulation of colorants in the food and beverages on the surface of the restorative material, and these superficial discolorations can be removed by polishing (18). However, it is inevitable to replace the restoration in cases with severe discoloration. The color stability of composite resins depend on physical properties such as organic matrix, filler particles and conversion rate, and restorative technique such as finishing and polishing procedures (18,19).

Color selection of tooth-colored restorative materials is an essential stage of restorative treatment protocol. Color determination in dentistry is carried out in two methods, visually or instrumentally. Visual color determination is the most frequently applied method, however it is a subjective process which holds several disadvantages such as inconsistency and lack of standardization (20). To overcome these limitations, instrumental color determination is used in dental research studies with color matching devices such as spectrophotometers and colorimeters (21,22). These devices are objective and the results can be quantified (23,24). Commission Internationale de L'Eclairage (CIE) introduced CIELAB color system to provide better interpretation of color perception. This system is the most frequently used for color measurements in dentistry and describes color in three dimensions: lightness, hue, and chroma (25).

It is a known fact that acidic beverages with low pH cause degradation in the matrix structure of resins, and there are studies reporting that color change is observed accordingly (26). However, no study has been found to examine the color stability of bulk-fill composites with different viscosities against acidic beverages with increasing consumption. Considering the widespread use of bulk-fill composites and the fact that color change is an important concern that results in replacement of the restorations, further studies are needed on this topic. Therefore, this in vitro study aimed to investigate the effects of the acidic beverages on the color change of bulk-fill composites with different viscosities. The tested null hypotheses were that 1) acidic beverage type would not make any difference in color stability of bulk-fill composites, and 2) the viscosity of the bulk-fill composites would not influence the color stability.

MATERIALS AND METHODS

A total of 144 disc-shaped specimens were prepared from four different bulk-fill composi-

tes, including a nanohybrid conventional composite resin (Filtek Ultimate Universal Restorative; 3M ESPE, St. Paul, MN, USA), a packable bulk-fill composite resin (Filtek Bulk Fill; 3M ESPE, St. Paul, MN, USA), a packable bulk-fill composite resin (Tetric N Ceram Bulk Fill; Ivoclar, Vivadent, Schaan, Liechtenstein), and a flowable bulk-fill composite resin (Tetric N Flow Bulk Fill; Ivoclar, Vivadent, Schaan, Liechtenstein) of shade A2 (Table 1). The specimens were 8mm in diameter and 2mm in thickness and fabricated using a stainless steel mold. Composites were loaded into the mold and pressed between mylar strips and glass slabs during polymerization to obtain

a smooth surface. The top surfaces of the specimens were polymerized for 20 seconds using a light emitting diode (LED) curing device (Elipar Deep Cure; 3M ESPE, St. Paul, MN, USA) with a light intensity of 1250 mW/cm². The polymerized specimens were stored in distilled water at 37°C for 24 hours to allow complete curing. Afterwards, the surface of all specimens was wet grounded by sanding using silicon carbide papers with descending grits (320,600,1200,2400,4000) under water cooling for 60 seconds. All samples were ultrasonically cleaned for 10 minutes, and were then placed in distilled water for 24 hours at 37°C before baseline color evaluation.

Table 1. Composite restorative materials used.

Material (Lot Number)	Type	Resin (Photoinitiator)	Filler Type	Filler
Filtek Ultimate (3M ESPE, St Paul, MN, USA) (N817010)	Conventional composite resin	Bis-GMA, UDMA, TEGDMA, PEGDMA, Bis-EMA. (CQ)	20 nm silica particles, 4 - 11 nm zirconium particles.	72.5% by weight
Filtek Bulk Fill (3M ESPE, St. Paul, MN, USA) (N681830)	Bulk fill restorative composite	AUDMA, AFM, DDDMAA, UDMA. (CQ)	Zirconia/silica cluster, ytterbium trifluoride	76.5 by weight
Tetric N Ceram Bulk Fill (Ivoclar Vivadent Inc, Amherst, NY, USA) (W70628)	Bulk fill restorative composite	Bis-GMA, Bis-EMA, UDMA. (CQ, TPO, Ivocerin)	Barium glass filler, ytterbium fluoride, spherical mixed oxide	79% by weight
Tetric N Flow Bulk Fill (Ivoclar Vivadent Inc, Amherst, NY, USA) (X43433)	Flowable bulk fill composite	Bis-GMA, UDMA, TEGDMA. (Ivocerin)	Barium glass, ytterbium trifluoride, mixed oxide, silicon dioxide	64.9% by weight

Abbreviations: AFM, addition-fragmentation monomers; AUDMA, aromatic urethane dimethacrylate; Bis-EMA, ethoxylated bisphenol-A-glycidyl methacrylate; Bis-GMA, bisphenol-A glycidyl methacrylate; CQ, camphorquinone; DDDMA, 1,12- dodecanediol dimethacrylate; PEGDMA, poly(ethylene glycol) dimethacrylate; TEGDMA, triethylene glycol dimethacrylate; TPO, 2,4,6-trimethylbenzoyl diphenylphosphine oxide; UDMA, urethane dimethacrylate.

Baseline color of the specimens were measured with a clinical compact spectrophotometer (VITA Easyshade Compact; VITA Zahnfabrik, Bad Sackingen, Germany), which has more than 90% reliability and validity (27). The spectrophotometer was calibrated according to the manufacturer's instructions before each measurement. Specimens were placed on a white background and the probe tip was placed perpendicular in the middle

of each specimen. The measurement procedures were repeated three times for each disc and L*a*b* values were noted to calculate the average. All measurements were performed under D65 standard lighting conditions in accordance to the CIE Lab system.

Following the initial color measurements, the specimens of each group were divided into 3

subgroups and submerged in two acidic beverages, namely an orange juice (Cappy 100% Orange Juice, Bursa, Turkey) and a coke (Coca Cola Zero Sugar; The Coca-Cola Company, Istanbul, Turkey) Distilled water served as control (n=12). The pH levels of the acidic beverages were measured using a benchtop pH meter (Hanna Instrument, USA) and presented in Table 2. Specimens were placed in light proof containers for 30 days, and immersion media was replaced with fresh ones in every other day.

Table 2. pH levels of the immersion media

Immersion medium	
Distilled Water	6.9
Coke	3.25
Orange Juice	3.6

The samples were rinsed for 10s with distilled water and gently wiped dry prior to the measurement. Color coordinates were measured before immersion as baseline and after 30 days of storage in different immersion media. CIELab system enables the evaluation of perceptible color change based on three coordinates: L* representing lightness or value, a* and b* representing chromacity with axes from red to green and blue to yellow, respectively. The means of the coordinates were calculated, and the L*, a* and b* parameters were determined. A high ΔE score represents an excessive color change. The clinically acceptable color change threshold level was defined as $\Delta E = 2.7$ with a 50%:50% confidence level (28).

The color difference formula is:

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

STATISTICAL ANALYSIS

The statistical analysis was conducted using a statistical software (SPSS for Windows; IBM SPSS Inc., Chicago, IL, USA). The normality of the data distribution was analyzed using the Shapiro-

Wilk test. As a result of the normality test, the p values were measured as 0.482, 0.128, 0.492 for distilled water, coke and orange juice, respectively. The ΔE data were analyzed by two-way ANOVA with material type and immersion media as main factors, since the distribution of the data was normal. Tukey's test was performed for pairwise comparisons. A p-value of <0.05 level was considered statistically significant.

RESULTS

The mean (\pm standard deviation) color differences (ΔE) after exposure to two acidic beverages for 30 days were summarized in Table 3. Two-way ANOVA revealed that both material type and staining solution factors significantly influenced ΔE values ($P < 0.001$). Additionally, significant interaction was observed between these two factors ($P < 0.001$). Immersion in orange juice resulted in greater and significant color change over time, when compared to coke and distilled water storage ($P < 0.05$).

Table 3. Mean and standard deviation of color change (ΔE) for composite resins.

	Distilled Water	Coke	Orange Juice
Filtek Ultimate	1.6 (0.2) ^{abA}	3.3 (1.0) ^{bB}	4.9 (1.0) ^{b c}
Filtek Bulk Fill	1.0 (0.3) ^{bA}	3.4 (0.5) ^{bB}	4.3 (0.8) ^{bB}
Tetric N Flow Bulk Fill	2.1 (0.3) ^{aA}	5.9 (1.3) ^{ab}	7.7 (1.1) ^{a c}
Tetric N Ceram Bulk Fill	2.0 (0.2) ^{aA}	5.7 (0.6) ^{ab}	7.0 (1.0) ^{a c}

Means followed by different lower-case letters in the same column, and upper-case letters in the same row, are significantly different ($P < 0.05$).

Color changes of the composite resins were in the range of 1-7.7 ΔE unit. After 30 days, the highest mean ΔE values were observed for Tetric BF Flow (7.7 ± 1.1) and Tetric BF (7 ± 1) immersed in orange juice, respectively. However, there was no statistically significant difference between

them. Additionally, ΔE values of both Tetric BF Flow and Tetric BF were significantly higher than the corresponding values for Filtek Ultimate Universal and Filtek Bulk-fill which were immersed in orange juice ($P < 0.05$). The bulk-fill composites demonstrated significant color alteration compared to the conventional composite, and ΔE values considered clinically critical.

DISCUSSION

The color change of composite resins occurs over time depending on internal and external factors. Surface roughness and irregularities cause bacterial plaque retention and superficial staining, whereas subsurface and internal discolorations occur because of resin matrix composition including its conversion rate and water absorption after polymerization (29,30). Restorative materials are exposed to changing temperatures, humidity and mechanical forces constantly in the oral environment. Therefore, evaluation of restorative materials with *in vitro* studies in which intraoral conditions are imitated is important to predict the clinical success of these materials (13). Therefore, this *in vitro* study aimed to investigate the effects of the acidic beverages on the color change of bulk-fill composites with different viscosities. Based on the results of the study, orange juice produces significantly more color change compared to coke and distilled water. Therefore, the first null hypothesis that “acidic beverage type would not make any difference in color stability of bulk-fill composites” was rejected. Additionally, no significant difference was found between low and high viscosity bulk-fill composites in terms of color stability. Accordingly, the second null hypothesis that “the viscosity of the bulk-fill composites would not influence the color stability” was accepted.

It is known that beverages consumed daily negatively affect the color stability and aesthetics of tooth-colored restorative materials (21), and these color changes are especially related to the

dietetic habits of individuals. In previous studies, the color stability of restorative materials was mostly tested with beverages such as coffee and red wine (21,31). However, the consumption of orange juice as a part of healthy lifestyle and coke, which has an important place in popular culture, is quite high these days (32). The common point of these two different beverages is that they both have acidic pH. It is reported that acidic environmental conditions not only affect composite resins superficially, but also internally due to dislocation of filler particles as a result of chemical interaction with organic matrix (33). Therefore, it is important to investigate the effects of acidic beverages such as orange juice and coke on the color stability of composite resins.

According to the results of the present study, the highest color change was observed for the samples immersed in orange juice. In a previous research, the effects of acidic beverages on the color stability and gloss of composite resins were investigated, and it was reported that coke and cranberry juice caused more color change and gloss loss compared to artificial saliva used as controls. Arregui *et al.* (34) investigated the effects of 6 different beverages on the color stability of composite resins including high- and low-viscosity bulk-fill composites, over a 6-month period and suggested that orange juice produces more color change than coke. Furthermore, it was reported that the ΔE value caused by both beverages was significantly higher than distilled water.

The color change potential of both coke and orange juice can be explained by the pH of the citric acid in their content (Table 2), which is low enough to cause softening of the composite resin matrix. In addition, the common opinion of the studies in the literature is that as the pH of beverages decreases, aesthetic and optical properties such as color stability will be affected dramatically (35). Although the pH value of coke is more acidic than orange juice, in the current study,

it was determined that orange juice caused more color change, similar to other studies (34,36). It has been concluded that one of the main reasons why coke causes less color change may be the lack of yellow colorant in its composition (22). The findings of the present study also support this conclusion.

In the current study, bulk-fill composites showed evident color change compared with conventional composites when immersed in coke and orange juice. Some researchers noted that as the thickness of the composite resin samples increases, higher color changes were observed (16,22). According to the study design of the present investigation, the preparation of both conventional and bulk-fill composites with a thickness of 2mm can be speculated as the reason for the less color change of the bulk-fill composites.

In a previous study, it was concluded that flowable composites exhibit less color change compared to packable composites (22). In the present study, it was observed that the flowable composite samples kept in both coke and orange juice undergo a slightly more color change than the packable composite samples. However, this difference was not found to be statistically significant. Flowable composites are simply obtained by increasing the organic matrix ratio in the formulation of conventional composites. It is known that with the increase of the organic matrix ratio, the amount of filler decreases and results in increased water absorption, which makes composite resin more prone to color change (37). The difference between the results of the studies may be due to the difference in the organic matrices of bulk-fill composites compared to conventional composites in order to increase the depth of cure. However, more in-depth studies are needed on the color stability of bulk-fill composites in order to make more precise inferences.

Instrumental color measurement is considered to be as the most reliable method in dentistry (38). For this reason, color measurements were carried out using the Vita EasyShade spectrophotometer device. In the evaluation of colorimetric properties of tooth-colored restoratives, the CIELab system is the most preferred system in terms of standardization and reproduction of studies (39,40). This system characterizes the color based on human perception, and designates to 3 coordinates including, the brightness (value) of a shade (L^*), the amount of red-green color (a^*), and the amount of yellow-blue color (b^*). Absolute measurements are made in $L^*a^*b^*$ color parameters and overall shade changes (ΔE) in the composite resin can be calculated using the abovementioned equation (41). Theoretically, if no color change occurs when a material is exposed to environmental conditions ($\Delta E=0$), that material is considered color stable (42). However, the color perception of the human eye is not as sharp as spectrophotometric analysis. For this reason, studies suggest that in cases where the ΔE value of the restoration reaches or is above the threshold value ($\Delta E=2.7$), the color change of the restoration can be distinguished by the observer and therefore the restoration should be replaced (28). According to the results of the spectrophotometric analysis, it is noteworthy that all samples immersed in coke and orange juice exhibited a color change above the clinically acceptable threshold. This finding can be translated to the clinic as acidic beverages have destructive effects on the color stability of bulk-fill composites.

One of the most important limitations of the present study is that the penetration depth of the coloring agents into the material was not investigated. Detection of external or internal discoloration is decisive in terms of removing the discoloration. Surface roughness measurement, along with 3D profile or SEM images of material surfaces, before

and after immersion, to correlate surface damage to color change, as both properties act together during composite staining would be beneficial for better understanding. Another limitation is that it is not investigated whether the discoloration is removed by a process such as repolishing or brushing after discoloration. Further studies are needed to establish a correlation between depth of penetration of staining agents and the ability to remove the resulting discoloration.

CONCLUSION

Considering the limitations imposed by this *in vitro* study, a color change above the clinically acceptable threshold was observed in all bulk-fill composites and it can be speculated that orange juice has a more negative effect on the color stability of bulk-fill composites compared to coke. In addition, it was concluded that the color change characteristics of Tetric BF flow and Tetric BF groups were quite similar regardless of the used staining solution whereas Filtek Ultimate and Filtek Bulk-fill samples demonstrated significantly less color change.

AUTHOR CONTRIBUTION STATEMENT

Conceptualization and Design: S.S.
Literature Review: S.S. and G.S.
Methodology and Validation: S.S.
Investigation and Data Collection: S.S. AND G.S.
Data Analysis and Interpretation: S.S.
Writing-Original Draft Preparation: S.S. and G.S.
Writing-Review & Editing: S.S. and G.S.
Supervision: S.S.

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CONFLICT OF INTEREST

There are no conflicts of interest.

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