

# **Time-Dependent Change of Color and Translucency of Recent Restorative Materials in Various Beverages**

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# ABSTRACT

**Objective:** The aim of this study was to evaluate the time-dependent changes in optical properties of recent tooth-colored restorative materials in commonly used coloring beverages.

**Method:** A total of 200 specimens of four different materials (Alkasite, ACTIVA, Equia Forte, Zirconomer) and a composite material as the control group were prepared in Teflon molds and the materials were immersed in four different solutions (coffee, cola, ice tea and saliva) (n=10). Color measurements were performed with a spectrophotometer at baseline, 7, 14 and 28 days. Color changes, translucency parameters (TP) and contrast ratios of the materials were calculated. The data were analyzed by repeated measurements and three-way ANOVA and post hoc Tukey tests.

**Results:** Alkasite showed the greatest color change for all solutions in all immersion periods compared to other materials (p<0.001). The experimented solutions caused a perceptible color change in all materials after 28 days (p<0.05). Coffee and ice tea caused more staining of resin-based materials for all immersion periods (p<0.05). Initial TP and  $\Delta$ TP of glass ionomer-based materials were found to be lower than the resin-based materials.

**Conclusion:** Exposure of the recent materials to different solutions for a certain period of time caused significant changes in optical properties. Resin-based alkasite, ACTIVA and composite showed the highest change the optical properties in coffee and ice tea. **Keywords:** Alkasite, color change, composite resin, contrast ratio, translucency.

# **1. INTRODUCTION**

The resin-based materials are popular materials in modern dentistry due to their esthetic properties, mechanical strength and low cost (1). However, composite resins have disadvantages such as incremental application, insufficient degree of conversion, weak cavity adaptation, lack of remineralization ability and absence of antibacterial properties (2,3). Glass ionomer cements (GIC), which are routinely used as tooth-colored restorative materials. GICs can be considered as the first-choice materials, especially in the restorations of patients with high cariogenic activity, due to their fluoride release feature that gives them a cariostatic property (4,5). In addition, current restorative materials are being developed by combining the strength and esthetics of composites with the benefits of glass ionomers. One of these materials, alkasite is a bulk-fill tooth-colored resinbased restorative material which uses alkaline filler, capable of releasing acid-neutralizing ions and fluorides (6). Another recent resin-based material, ACTIVA contains bioactive ionic resin, patented rubberized resin, and bioactive ionomer glass (7). Bioactive ionic resin content of the material provides

the properties of moisture tolerant and high release and recharge of calcium, phosphate, and fluoride ions (8).

One of the main goals in the current improvement of the dental restorative materials is to ensure the optical properties of the tooth to reflect its original nature (9). In order to obtain esthetically successful restorative filling materials, they are able to imitate the natural tooth structure and are able to maintain their color and translucency of the initial application (10). Light reaching a tooth surface can be reflected, diffused, absorbed or transmitted (11). Also, restorative materials should exhibit similar behavior in order to provide acceptable esthetics. Translucency is defined as the ability of a material to transmit light and it was reported to be the most essential parameter after primary color properties (12). Also, it is one of the key factors affecting the esthetic performance of the dental restorations (13). Translucency parameter (TP) and contrast ratio (CR) are two commonly used indicators to determine translucency values

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Content of this journal is licensed under a Creative Commons Attribution-NonCommercial 4.0 International License. and changes (14). CR is a wavelength dependent parameter based on the luminescence and reflection calculations (15).

A change in color or shade can also affect translucency and change the initial optical properties (16) The longevity of the restorative materials in the oral cavity was extended by the improvements in their structures. One of the important factors in restoration renewal is the loss of color harmony between the restoration and the tooth (17). Therefore, longterm color stability of dental materials is a necessity (18). With the increasing esthetic demands of the patients, the color match and the stability of the restorative materials became important in determining the longevity of the restoration even in the posterior region. Color changes in composites occur due to the internal and external factors. Internal coloration occurs in the inner layers as a result of the structure of the composite resins and incomplete polymerization, while external coloration occurs as a color change on the outer surfaces of the composite resins (19).

It was determined that the coloration caused by diet containing coloring pigment and chemical coloring agents is more prominent than the coloration caused by intrinsic factors due to water absorption in polymerized materials (20). Moreover, the pH value of the aging solution can also affect the surface degradation, causing changes in water absorption (21). Also, it was reported that the beverages containing coloring pigments such as tea, coffee, cola, red wine, fruit juices and energy drinks, which were widely consumed, caused discoloration in resin-based materials (1, 20). It was also reported that when the resin-containing glass ionomers were subjected to immersion in various solutions, more discoloration occurred comparing the composites (22,23). A recent study evaluating the translucency of the different GICs found that they had low translucency (13).

Table 1. The Brand and the contents o	of the restorative materials
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However, there is not enough information in the literature about the color stability and translucency of recent restorative materials, whose demand for use was increased with the progress in their structure. The aim of this study is to evaluate the time-dependent changes in optical properties of recent fluoride-released tooth-colored restorative materials in commonly used colorant beverages by comparing them with composite. In this study, two null hypotheses were tested: (i) that the type of restorative materials and (ii) the type of the beverage and the duration of the immersion would not affect the color, translucency and contrast of the materials.

# 2. METHODS

## 2.1. Preparation of Specimens

This study was found medically appropriate with the ethics committee report numbered 2021/108.43 and date 21.02.2021 of Çukurova University, Faculty of Medicine, Clinical Research Ethics Committee. The specification, composition and filler ratio of the materials (Alkasite, ACTIVA, Equia Forte, Zirconomer and composite) used in this study were given in Table 1. The microhybrid composite was included as the control group. A total of 200 discshaped specimens were fabricated for 40 of each material. In accordance with the manufacturer's instructions, all materials were prepared in disk-shaped Teflon molds with a diameter of 8 mm and a height of 2 mm, by pressing gently from the top with mylar strip and glass to promote removal of the excess material and to obtain a standard surface. The thickness of each disc was measured with a digital caliper (Liaoning MEC, Dalian, China) placed in the center of the material. Only samples with a thickness of 2 mm were used in the study.

Materials	Specification	Composition	Filler ratio (weighted)
Alkasite (Cention N; Ivoclar Vivadent, Schaan Liechtenstein) Lot: Z0054T	Alkasite, self-adesive restorative material	Powder: Ca fluorosilicate glass (25-35%, Ba-Al silicate glass (20-30%), Ca-Ba-Al fluorosilicate glass (10-20%), ytterbium trifluoride (5-10%), iso-fillers, initiators and pigments. Liquid: UDMA (95-97%), DCP, Aromatic aliphatic – UDMA, PEG-400 DMA, initiators (hydroperoxide – self cure), stabilizers, additives and mint flavour	78.4%
ACTIVA (Pulpdent, Watertown, MA, USA) Lot: 191212	Bioactive glass ionomer	Blend of diurethane and other methacrylates with modified polyacrylic acid (44.6%), Bioactive glass filler (21.8%), patented rubberized resin (Embrace), amorphous silica (6.7%), and sodium fluoride (0.75%)	56%
Equia Forte Fil (GC, Corp., Tokyo, Japan) Lot: 1803121	High viscosity glass ionomer cement (HVGIC)	Fluoroalumino silicate glass (95%), hybrid glass particles, polyacrylic acid powder, 5% polyacrylic acid, polybasic carboxylic acid, distilled water	75%
Zirconomer (Shofu, Kyoto, Japan) Lot: 10200185	High viscosity glass ionomer cement (HVGIC)	Powder: Fluoro-Aluminosilicate glass, Zirconium oxide, pigments and others Liquid: polyacrylic acid solution and Tartaric acid	15 – 20%
Filtek Z250 (3M ESPE, St.Paul, MN, USA) Lot: NA53476	Microhibrit composite resin	TEGDMA (1 – 5%), Bis-GMA (1 – 5%), Bis-EMA (5 – 10%), UDMA (5 – 10%), Zirconia/silica inorganic fillers 60%	60%

Ca: Calcium, Ba: Barium, Al: Aliminum, UDMA: Urethane dimethacrylate, DCP: Tricyclodecan-dimethanol dimethacrylate, Aromatic aliphatic – UDMA: Tetramethyl-xylylendiurethane dimethacrylate, PEG-400 DMA: Polyethylene glycol 400 dimethacrylate, TEGDMA: Trietyhlenglycol dimetacrylate, Bis-GMA: Bisphenol-A-glycidylmethacrylate, Bis-EMA: Bisphenol-A polyethylenglycol dietherdimethacrylate **Group 1 (Alkasite):** One measuring spoon of powder and one drop of liquid, corresponding to a powder/liquid weight ratio of 4.6g to 1g, were manually mixed until a smooth consistency. The mixing time was completed in 60s and the prepared material placed in the mold. The setting time for the self-curing mode was achieved in 5min.

**Group 2 (ACTIVA):** The material applied into the mold with a gun and polymerized with a 1200 mW/cm<sup>2</sup> LED curing unit (Freelight Elipar II, 3M, St. Paul, MN, USA) for 20s.

**Group 3 (Equia Forte):** Capsule of the material was activated just before mixing and was placed in the amalgamator immediately. The restorative material mixing time was 10s and the setting time of the material was 2min 30s after placing into the mold. Equia Forte Coat (GC, Tokyo, Japan) was applied to the surfaces using a disposable microtip applicator. The coated surface was light-cured with the same curing unit.

**Group 4 (Zirconomer):** Two measures of powder and one drop of liquid with a powder liquid ratio of 3.6 g/1.0 g were placed on the mixing paper. The first half of 2 equal parts of powder was added to the spread liquid by using a plastic spatula and mixing in 5-10s. Then the remaining half was added and mixed until a paste-like consistency was achieved. Mixing was completed in a total of 30s. The prepared material was placed in the mold and the 3min curing time was completed.

**Group 5 (Composite):** The material was applied into mold and was polymerized with a 1200 mW/cm<sup>2</sup> LED curing unit for 20s.

The upper surfaces of all samples were polished with medium, thin and superfine aluminum oxide impregnated discs (Sof-Lex, 3M, St. Paul, MN, USA), respectively, using a low-speed handpiece rotating at 12,000rpm, according to manufacturer's instructions. The samples were kept in distilled water at 37°C for 24h in an incubator (Memmert, Schwabach, Germany) to complete their polymerization. Then, the baseline color measurements of the materials were determined.

#### 2.2. Storage in Colored Beverages and Artificial Saliva

The groups of the restorative materials were divided into four subgroups (n=10) according to the type of the solution (coffee, cola, ice tea and artificial saliva). The composition and pH values of the coloring agents used in this study and the salivary fluid used as the control group were given in Table 2. The specimens were immersed for 7, 14, 28 days in four different solutions in the incubator at 37°C. The specimens were positioned inside 48-well plates containing 3 mL of solution during the immersion process. All solutions were renewed every 24h. Before putting the samples into the newly prepared solution, they were washed with 5ml of distilled water and dried with blotting paper.

# 2.3. Measurement of the Color Change, Translucency Parameter and Contrast Ratio

The optical analyzes were carried out by using a spectrophotometer (VITA Easyshade<sup>\*</sup> V; VITA Zahnfabrik, Bad Sackingen, Germany). A saturated sucrose solution (refractive index n=1.5 approximately) was used as contact between samples and background. The values of CIELAB coordinates (L\*, a\*, b\*) were determined by using the CIE D65 illuminator and the CIE 2° standard colorimetric observation at initial, 7, 14 and 28 days on a white background [24]. It was completed by taking three consecutive measurements from each sample. The color change ( $\Delta E_{00}$ ) was calculated for each sample according to the CIEDE2000 formula [25,26]:

$$\Delta E_{00} = \left[ \left( \frac{\Delta L'}{K_L S_L} \right)^2 + \left( \frac{\Delta C'}{K_c S_c} \right)^2 + \left( \frac{\Delta H'}{K_H S_H} \right)^2 + R_T \left( \frac{\Delta C'}{K_c S_c} \right) \left( \frac{\Delta H'}{K_H S_H} \right) \right]^{1/2}$$

where  $\Delta L'$ ,  $\Delta C'$  and  $\Delta H'$  in the CIEDE2000 system are the differences in lightness, chroma and hue, respectively. The weighting functions (SL, SC, and SH) determine the total color difference for variation in the location. The parametric factors (KL, KC and KH) are correction terms for experimental conditions. For this study, each KL, KC and KH were taken as 1.0. A rotation function (RT) defines the interaction between chroma and hue differences in the blue region.

Table 2	2. Th	e brand.	content and	pH value	of the	solutions
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Solutions	Composition	рН
Cola (The Coca Cola, İstanbul Turkey)	Carbonated water, sugar, caramel color, phosphoric acid, natural flavors, caffeine	2.53
Ice-Tea (Unilever, Lipton, İstanbul, Turkey)	Water, sugar, fructose, acids (citric acid, malic acid), black tea extract (0.14%), peach juice concentrate (0.1%), acidity regulator (trisodium citrate), flavorings (peach flavor), antioxidant (ascorbic acid), sweetener (steviol glycosides).	3.8
Coffee (Nescafe Classic, Nestle, Vevey, Switzerland)	10g coffee / 200 ml of boiling water and cooled to 60°C	4.50
Artificial saliva	1.160 g/l sodium chloride, 0.600 g/l calcium chloride, 0.600 g/l potassium phosphate, 1.491 g/l potassium chloride, 0.050 g/l sodium fluoride, trace of sodium hydroxide	6.93

#### **Optical Properties of Recent Restorative Materials**

$$TP_{00} = \left[ \left( \frac{L'_B - L'_W}{K_L S_L} \right)^2 + \left( \frac{C'_B - C'_W}{K_C S_c} \right)^2 + \left( \frac{H'_B - H'_W}{K_H S_H} \right)^2 + R_T \left( \frac{C'_B - C'_W}{K_C S_C} \right) \left( \frac{H'_B - H'_W}{K_H S_H} \right) \right]^{1/2}$$

where subscripts w and b refer to measurements on white and black backgrounds, respectively. The change in translucency parameter ( $\Delta TP_{00}$ ) at the end of the 28th day was calculated according to the formula following:

$$\Delta TP_{00} = TP_{28} - TP_0$$

The calculation of CR was carried out by using the L\* values measured on white and black backgrounds. The spectral reflectance, Y values were calculated for black and white background as following formula and YN value was taken as 100 (15):

$$Y = (L + 16/116)^3 x Y_N$$

The values of CR were obtained as follows:

$$CR = Y_B / Y_W$$

The change in contrast ratio ( $\Delta$ CR) at the end of the 28th day was calculated according to the formula following:

$$\Delta CR = CR_{28} - CR_0$$

#### 2.4. Statistical Analysis

Statistical analyzes were performed by using the SPSS program for Mac version 26 (IBM SPSS, Chicago, IL, USA). Homogeneity of the data was evaluated by using the Shapiro Wilks test. Intergroup and intragroup comparisons of the color change, TP and CR were analyzed by one-way analysis of variance (ANOVA) and Tukey post hoc test. The correlation between CR and TP of the materials was assessed with the Pearson correlation test. Time-dependent color changes of the materials were compared by using repeated measure ANOVA and paired sample t-test, respectively. A three-way ANOVA (restorative material at five levels, immersion solution at four levels and time at three levels) evaluated the effects of material type, storage solution and time as well as their interaction, on  $\Delta$ EOO values. All statistical testing was performed at a preset alpha of 0.05.

#### **3. RESULTS**

The mean color change ( $\Delta E_{00}$ ), standard deviations, comparison of the materials according to the type of immersed liquid and comparison of the immersion times were shown in Table 3. Comparison of the effect of type of immersed liquid on color change of the material was shown in Figure 1.  $\Delta E_{00}$  increased with time in all solutions and the difference was statistically significant in all groups (p≤0.002). There was a significant difference between 7 and 28 days for all groups in pairwise comparisons. All solutions caused a perceptible color change in all materials after 28 days.

Alkasite, ACTIVA and composite materials, which are resinbased materials, showed the highest  $\Delta E_{00}$  in coffee and ice tea solutions. Cola caused significantly less color change compared to coffee and ice tea for alkasite at the measurement times of the 14 and 28 days, for ACTIVA at all measurement times and for composite at the measurement of the 7th days. (p<0.007) Coffee and cola caused more coloration of Equia Forte than ice tea and saliva. The difference among groups was statistically significant for all the measurement times. (p<0.001). Cola caused more coloration of Zirconomer than coffee and ice tea for all the measurement times (p<0.004). Saliva caused the lowest  $\Delta E_{00}$  at all measurement times and for all materials (p<0.001). Three-way ANOVA test (Table 4) revealed difference in the mean value of  $\Delta E_{00}$  that was significantly affected by the material type, solution type and time (p<0.001).

		00					
Liquids	Days	Alkasite	ACTIVA	Equia Forte	Zirconomer	Composite	р
Coffee	7	12.31 ± 2.99 <sup>A, a</sup>	6.32 ± 1.48 <sup>A, b</sup>	7.30 ± 2.65 <sup>A, b</sup>	3.60 ± 0.73 <sup>A, c</sup>	6.67 ± 1.94 <sup>A, b</sup>	< 0.001
	14	16.63 ± 2.90 <sup>B, a</sup>	7.89 ± 1.70 <sup>A, c</sup>	11.27 ± 2.61 <sup>B, b</sup>	4.80 ± 0.95 <sup>B, d</sup>	8.18 ± 1.69 <sup>B, c</sup>	< 0.001
	28	21.18 ± 3.34 <sup>C, a</sup>	10.78 ± 2.15 <sup>B, c</sup>	14.88 ± 4.77 <sup>C, b</sup>	$6.09 \pm 1.08^{C, d}$	10.09 ± 2.33 <sup>C, c</sup>	< 0.001
р		<0.001	<0.001	<0.001	< 0.001	<0.003	
Cola	7	8.90 ± 1.63 <sup>A, a</sup>	2.24 ± 0.94 <sup>A, d</sup>	6.70 ± 1.66 <sup>A, b</sup>	5.73 ± 1.36 <sup>A, bc</sup>	4.60 ± 1.43 <sup>A, c</sup>	< 0.001
	14	11.91 ± 4.01 <sup>AB,a</sup>	3.14 ± 1.16 <sup>B, c</sup>	$9.46 \pm 2.64^{B, ab}$	6.76 ± 1.61 <sup>A, b</sup>	6.64 ± 1.34 <sup>B, b</sup>	< 0.001
	28	13.12 ± 2.33 <sup>B, ab</sup>	5.12 ± 1.55 <sup>C, d</sup>	15.54 ± 2.14 <sup>c, b</sup>	10.65 ± 2.22 <sup>B, ac</sup>	8.41 ± 1.90 <sup>B, c</sup>	< 0.001
р		<0.007	<0.001	<0.001	< 0.001	<0.001	
Ice tea	7	10.09 ± 2.69 <sup>A, a</sup>	6.48 ± 1.44 <sup>A, b</sup>	2.56 ± 0.92 <sup>A, c</sup>	2.78 ± 1.15 <sup>A, c</sup>	6.39 ± 1.09 <sup>A, b</sup>	< 0.001
	14	17.85 ± 3.05 <sup>B, a</sup>	7.98 ± 1.3 <sup>B, b</sup>	4.36 ± 1.32 <sup>B, c</sup>	4.88 ± 1.29 <sup>B, c</sup>	$7.63 \pm 1.78^{A, b}$	< 0.001
	28	23.33 ± 3.36 <sup>C, a</sup>	9.83 ± 2.09 <sup>C, bc</sup>	7.69 ± 1.06 <sup>C, bc</sup>	7.45 ± 1.83 <sup>C, c</sup>	10.25 ± 1.8 <sup>B, b</sup>	< 0.001
р		< 0.001	<0.003	<0.001	< 0.001	< 0.001	
Saliva	7	$4.68 \pm 0.83^{A, a}$	1.58 ± 0.42 <sup>A, b</sup>	1.73 ± 0.39 <sup>A, b</sup>	1.55 ± 0.38 <sup>A, b</sup>	$1.48 \pm 0.63^{A, b}$	< 0.001
	14	5.62 ± 1.78 <sup>AB, a</sup>	$2.62 \pm 0.74^{B, b}$	$3.05 \pm 0.71^{B, b}$	$2.25 \pm 0.28^{B, b}$	$2.01 \pm 0.58^{B, b}$	< 0.001
	28	7.40 ± 1.18 <sup>B, a</sup>	3.45 ± 0.92 <sup>B, bc</sup>	4.27 ± 0.62 <sup>C, c</sup>	2.73 ± 0.34 <sup>C, b</sup>	2.60 ± 0.64 <sup>C, b</sup>	< 0.001
р		< 0.001	< 0.001	<0.001	<0.001	< 0.001	

**Table 3.** Mean color differences ( $\Delta E_{\alpha\nu}$ ), standard deviations and statistical analysis for specimens immersed in different liquids at different times

Different capital letters in each column indicate statistical differences depicted by paired samples t test and repeated measures ANOVA (p=0.05). Different small letters in each row indicate statistical differences depicted by one-way ANOVA and Tukey's post-hoc test (p=0.05).

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**Table 4.** Three-way ANOVA test for the influence of material, liquid and time on the color difference ( $\Delta E_{an}$ )

Source	Type III Sum of Squares	df	Mean Square	F	р
Corrected Model	13743.080ª	59	232.934	65.869	<0.001
Intercept	32779.824	1	32779.824	9269.485	<0.001
material	4705.701	4	1176.425	332.670	< 0.001
Liquid	3913.547	3	1304.516	368.891	< 0.001
Time	2084.243	2	1042.121	294.691	< 0.001
material * Liquid	2131.820	12	177.652	50.236	< 0.001
material * Time	318.747	8	39.843	11.267	< 0.001
Liquid * Time	257.850	6	42.975	12.152	< 0.001
material * Liquid * Time	331.172	24	13.799	3.902	<0.001

a. R Squared=0.878 (Adjusted R Squared=0.865)

**Table 5.** Mean  $\Delta TP_m$  values and standard deviations of restorative materials after 28 days of immersion in liquids

ΔΤΡ	Alkasite	ACTIVA	Equia Forte	Zirconomer	Composite	р
Coffee	4.6 ± 1.9 <sup>A, a</sup>	3.9 ± 1.0 <sup>A, a</sup>	1.5± 0.6 <sup>A, b</sup>	1.3 ± 0.6 <sup>A, b</sup>	3.5 ± 1.8 <sup>A, a</sup>	< 0.001
Cola	3.1 ± 1.3 <sup>AB, a</sup>	$2.0\pm0.9^{\text{B, ab}}$	1.6 ± 0.9 <sup>A, b</sup>	1.5 ± 0.8 <sup>A, b</sup>	3.2 ± 1.3 <sup>A, a</sup>	< 0.001
Ice tea	4.6 ± 1.5 <sup>A, a</sup>	$3.4 \pm 1.0^{A, ab}$	$1.0 \pm 0.4^{A, c}$	1.1 ± 0.4 <sup>AB, c</sup>	3.3 ± 1.0 <sup>A, b</sup>	< 0.001
Saliva	$2 \pm 0.4^{B, a}$	$1.4 \pm 0.6^{B, b}$	0.9 ± 0.2 <sup>A, c</sup>	0.5 ± 0.2 <sup>B, c</sup>	0.8 ± 0.2 <sup>B, c</sup>	< 0.001
р	<0.001	<0.001	<0.053	<0.005	<0.001	

Different capital letters in each column indicate statistical differences depicted by one-way ANOVA and Tukey's post-hoc test (p=0.05). Different small letters in each row indicate statistical differences depicted by one-way ANOVA and Tukey's post-hoc test (p=0.05).

Table 6. Mean *ACR* values and standard deviations of restorative materials after 28 days of immersion in liquids

			•		•	
ΔCR	Alkasite	ACTIVA	Equia Forte	Zirconomer	Composite	р
Coffee	$0.19 \pm 0.08^{A_{,a}}$	0.12 ± 0.05 <sup>A, b</sup>	0.11± 0.05 <sup>A, b</sup>	0.04 ± 0.02 <sup>A, c</sup>	$0.10 \pm 0.5^{A, b}$	< 0.001
Cola	$0.10 \pm 0.04^{B, a}$	$0.08 \pm 0.05^{A, a}$	0.10± 0.4 <sup>A, a</sup>	0.05 ± 0.03 <sup>A, b</sup>	0.07 ± 0.04 <sup>AB, a</sup>	<0.037
Ice tea	$0.13 \pm 0.07^{AB, a}$	$0.11\pm0.06^{\text{A, a}}$	$0.08 \pm 0.02^{\text{A, ab}}$	$0.03 \pm 0.01^{AB, b}$	$0.11 \pm 0.05^{A, a}$	<0.001
Saliva	0.08 ± 0.04 <sup>B, a</sup>	$0.6 \pm 0.2^{B, ab}$	$0.02 \pm 0.01^{B, cd}$	$0.01 \pm 0.00^{B, d}$	$0.04 \pm 0.01^{B, bc}$	<0.001
р	<0.001	<0.031	< 0.001	<0.004	<0.007	

Different capital letters in each column indicate statistical differences depicted by one-way ANOVA and Tukey's post-hoc test (p=0.05). Different small letters in each row indicate statistical differences depicted by one-way ANOVA and Tukey's post-hoc test (p=0.05).



**Figure 1.** Comparison of the effect of solution type on the color change of the material. Different letters indicate statistically significant differences among the solutions in the same measurement time and material.



*Figure 2.* Pairwise comparison of translucency parameters at the initial and 28 days. Different letters denote statistically significant difference between the initial and 28 days in the same solution and material.



*Figure 3.* Pairwise comparisons of contrast ratio at the initial and 28 days. Different letters denote statistically significant difference between the initial and 28 days in the same solution and material.

Translucency parameters and pairwise comparisons at the beginning and at the end of the 28th day were shown in Figure 2. There were significant differences for all groups (p<0.05). TP values of all materials decreased after immersion in solutions. The statistical analysis of mean and standard deviations of  $\Delta TP_{00}$  according to the type of the immersion liquid and material were represented in Table 5. Equia Forte and Zirconomer had lower initial translucency than the other materials.  $\Delta TP_{00}$  of these materials congruently was lower than the other materials immersed in coffee and ice tea solutions. Alkasite, ACTIVA and composite showed similar  $\Delta TP_{00}$  in all liquids except saliva.

The values of CR and pairwise comparisons of CR at the initial and 28 days were shown in Figure 3 and significant differences

were found for all groups except the Equia Forte group, which was immersed in saliva (p<0.05). HVGIC-based materials had significantly higher initial CR than the resin-based materials, consistent with TP results (p<0.001). The mean CR change ( $\Delta$ CR), standard deviations, statistical analysis according to the immersed solution type and the material were shown in Table 6. Zirconomer showed statistically significant lower  $\Delta$ CR than the other materials in all investigated solutions (p<0.05). Alkasite showed the highest  $\Delta$ CR value. However, the change in coffee and cola was not statistically significant compared to the other resin-based materials (p>0.05). The Pearson correlation test revealed a strong (r=-0.71) and statistically significant negative correlation between TP and CR.

# 4. DISCUSSIONS

There is a lack of studies in the literature evaluating the color stability and optical properties of recent bulk-fill restorative materials. In this in-vitro study, conventional resin composite and recent bulk-fill restorative materials were tested for  $\Delta E_{\alpha\alpha}$ ,  $TP_{00}$ ,  $\Delta TP_{00}$ , CR and  $\Delta CR$  after immersion in different aging solutions. As a result of this study,  $\Delta E_{_{00}}$ ,  $\Delta TP_{_{00}}$  and  $\Delta CR$  of the materials were found to be different from each other. Alkasite showed the most  $\Delta E_{00}$  and  $\Delta TP_{00}$  while Zirconomer was the least. Thus, the first hypothesis was rejected. The color changes of the materials increased during the immersion periods of 7, 14 and 28 days. At the end of 28 days, TP of the materials decreased and CR increased. While coloration occurred mostly in beverages such as coffee and ice tea with resin-based materials, cola was the beverage that most influenced the HVGIC-based materials. Thus, the second hypothesis of the study, which is the time of immersion in the type of beverage, was rejected.

Tooth-colored restorative materials should have excellent color matching and high color stability during the clinical treatment (28). The immersion in cola and coffee beverages with high staining potential are considered a proper test for predicting the tendency of the materials to change color (29). In this study, the effect of immersion for 7, 14 and 28 days on the color stability of the materials in coffee, cola, ice tea and saliva, which are consumed frequently, was investigated. The CIELab color formula, which is widely used in dentistry, was used to evaluate the colors and color differences between various natural and restorative esthetic materials (24). The CIEDE<sub>2000</sub> formula was shown to define color differences recognized by the human eye superior to the CIELab formula in recent studies (30). The  $\text{CIEDE}_{2000}$  was used to determine the color change in this study. The  $\text{CIEDE}_{_{2000}}$  and color differences were evaluated for detectability and clinical acceptability thresholds. Paravina et al. (31) reported that the color perceptibility threshold was between 0.8 and 1.8. In the study, all materials showed a color change over time in beverages above clinical acceptability.

In this study, the time-dependent color changes were determined mostly in coffee and ice tea.  $\Delta E_{_{00}}$  was occurred especially on Alkasite, ACTIVA and composite. Alkasite is one of the recently introduced tooth-colored materials and is

classified as a subgroup of composite materials (32). ACTIVA, which combines the positive properties of GIC and composite resins, can be an alternative to composite resins in anterior and posterior teeth due to its positive properties such as fluoride release and low polymerization shrinkage (33).

The hydrophilic nature and sensitivity to absorb water of the resin matrix can increase the probability of staining materials. The sensitivity to absorb water is a property of the resin ingredient of the material and the resistance of the resin-filler matrix. Excessive water absorption causes expansion and plasticization of the resin matrix, which results in hydrolysis of the silane and, accordingly, the formation of microcracks. Thus, the life of the composite resin is decreased. Microcracks or interfacial gaps between the filler and the matrix cause color penetration and discoloration (34). Coffee and tea contain yellow colorants of different polarities which could explain the discoloration of composite samples (35, 36). Previous studies were shown that composite resins are sensitive to discoloration when exposed to various coloring beverages (23, 34). Although alkasite is a resin-based material, which is a subgroup of composite resin, it was very sensitive to coloration.

The type and particle size of the fillers can also affect the discoloration of composite resin (19). Composite resins with smaller particles have been reported to have less tendency to stain (37). The fact that the resin matrix type of the alkasite is different from the composite resin and the particle size of the alkasite is larger may explain the greater discoloration than the composite resin. Furthermore, it was stated that all resin composition properties such as chemical differences and concentration of resin monomers, type of initiators and inhibitors, oxidation of unreacted monomers affect the discoloration potential of the composite resins (35). The discoloration in self-cured and dual-cure resins is more pronounced than in light-cured resins because tertiary aromatic amines are more likely to oxidize than aliphatic amines used in light-cured resins (38). In addition, since selfcure initiators can induce colored oxidation products that lead to the staining of resins by the time, self-curing of alkasite may cause more intrinsic coloration than the composite resin and ACTIVA (39).

The resin matrix mostly absorbs water directly, but glass fillers do not show the water absorption into the bulk of the material. It can only absorb water to the surface of the material (35). This situation could explain that the color changes of Equia Forte and Zirconomer with glass filler content in coffee and ice tea were less than that of alkasite, ACTIVA and composite with the resin matrix. When immersion in cola was examined, resin-containing materials did not show discoloration as much as coffee and tea. Although cola has the lowest pH value and damages the surface integrity of the resin materials, it has been reported that it does not cause discoloration as much as coffee and tea, probably because it does not contain yellow colorants (40). However, the most color changes occurred in cola with glass filler Equia Forte and Zirconomer. It can be caused by discoloration, possibly as a result of the low pH damaging the integrity of the materials.

Translucency and contrast are important properties for dental tissues and materials. TP and CR are two indicators commonly used to determine the transparency and opacity values and changes of the material (14). The values of TP range from 0 to 100, indicating that the material is completely transparent and completely opaque, respectively (41). The values for CR range from 0 to 1, indicating that the material is completely transparent and completely opaque, respectively (42). The strong correlation was found between the two values in this study.

The translucency of human teeth should be a reference in the evaluation of the translucency of dental restorative materials (43). The translucency of natural teeth tends to decrease from the cutting edge (TP=15) towards the cervical (TP=5) (44). Mean TP values of 1 mm thick bovine enamel, bovine dentin, human enamel and human dentin were 14.7, 15.2, 18.7, and 16.4, respectively (45). In this study, the values closest to enamel translucency were seen in microhybrid composite resin, while the translucency of HVGIC materials was quite low. The translucency of the flowable and universal resin composites of the same brand prepared with a thickness of 2 mm were compared, and the TP values were found to be between 10-15 for the flowable composite and between 9-12 for the universal composite, respectively (46). Uchimura et al. (13) found the TP values of 18 different conventional glass ionomers after 7 days without immersion in any liquid, varying between 3.9-20. The translucency values of resinmodified glass ionomers varied depending on whether the material was light-cured or cured by acid-base reaction alone (47). Light-cured samples were found to be marginally more translucent than the samples allowed to cure without irradiation.

Quek et al. (48) found that red wine and coffee decreased the translucency of composite restorative materials. Stawarczyk et al. (49) evaluated  $\Delta E$  and TP of five different composite resin materials for different colorant solutions for 14 days and reported that the internal structure and composition of the material affected the translucency of the materials. Barutçugil et al. (14) showed that immersion of three different materials in red wine and coffee caused significant changes in color and translucency compared to immersion in water. In the present study, the solutions affected  $\Delta E$ ,  $\Delta TP$  and  $\Delta CR$  of the materials and the changes were higher in coffee, cola and ice tea compared to saliva.

The detectability and acceptability thresholds of  $\Delta$ TP were reported as 0.62 and 2.62, respectively (16). Considering the  $\Delta$ TPs of the materials, the changes caused by the beverages at the end of 28 days showed unacceptable changes in the translucency of the resin-containing ones, while the changes in HVGIC-based materials are not clinically significant. This study stands out with its evaluation of  $\Delta$ E,  $\Delta$ TP and  $\Delta$ CR values as a result of time-dependent aging of current restorative materials in different solutions. In a study evaluating the CR of different GICs, the contrast values were found to be between 0.7-1 (13). A study evaluating time-dependent of  $\Delta$ CR found that all materials tested statistically increased their opacity during the 1-month immersion period (14). Similar to the above studies, CR values of the materials were found to be between 0.73-0.90 in this study, while their values approached 1 at the end of 28 days. These results may present a poor clinical picture due to the opacities of the materials. These materials may be preferred for clinical uses where masking with low thickness is required.

Limitations of this study involve allowing both sides of the material to be exposed to coloring agents, unlike clinical staining conditions. In the oral cavity, restorative materials are constantly exposed to colorants from foods and beverages and are rinsed with saliva and are cleaned with oral hygiene procedures. The present study experimented to simulate the clinical setting but did not comprise the effect of cleaning and thermal change on restorative materials and specimens were in beverages nonstop during the immersion period.

#### 5. CONCLUSIONS

Within the limitations of this in vitro study, exposing recent restorative materials to different beverages produced significant changes in color, translucency and contrast. Resinbased alkasite, ACTIVA and composite showed the highest change the optical properties in coffee and ice tea. HVGICbased materials, Equia Forte and Zirconomer were mostly affected by cola and coffee. HVGICs presented very low TP and high CR values, which remains thought-provoking in supplying esthetic demands.

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Analysis of data for the study: CK

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