

# Evaluation of color stability of bulk-fill restorative materials with different properties

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## ABSTRACT

**Aims:** The present study aims to evaluate the color stability of bulk-fill restorative materials with different properties over different time periods when immersed in a coffee solution.

**Methods:** According to the selected restorative materials, the study groups were formed as follows: CNT/light (Alkasite, Cention N, Ivoclar Vivadent, light cure mode), CNT/self (Alkasite, Cention N, self cure mode) EQF (Glass hybrid restorative, Equia Forte HT, GC Corporation), AU (Single-shade bulk fill, Aura Bulk Fill, SDI), Z550 (Filtek Z550, 3M) (control group). A total of 50 samples (n=10) were prepared using standard molds (2mmx10mm). After finishing and polishing procedures, all samples were kept in distilled water at 37°C for 24 hours. The first color measurement ( $L^*a^*b$ ) was performed with Vita Easy Shade V (VITA Zahnfabrik). Samples were added to the coffee solution. Color was measured at times simulating 7 days, 1 month, 6 months and 1 year of clinical service. Color changes ( $\Delta E1$ ,  $\Delta E2$ ,  $\Delta E3$  and  $\Delta E4$ ) were calculated according to the CIE  $L^*a^*b$  system. Normality of the data was examined by D'Agostino & Pearson omnibus normality test. One-way ANOVA analysis and Tukey test were performed. Statistical significance level was set at p 0.05 and statistical analysis software (GraphPad Prism 6.0, GraphPad Software, USA) was used for the analysis.

**Results:** According to  $\Delta E1$  results; clinically unacceptable color change ( $\Delta E > 3.3$ ) values were observed in CNT/light and CNT/self groups. The color change values of EQF, Z550 and AU groups were statistically similar ( $p > 0.05$ ). According to  $\Delta E2$  results; an unacceptable color change ( $\Delta E > 3.3$ ) was observed in all groups except EQF. According to  $\Delta E3$  results; an unacceptable color change ( $\Delta E > 3.3$ ) was observed in all groups. According to  $\Delta E4$  results; an unacceptable color change ( $\Delta E > 3.3$ ) was observed in all groups. The color change values of EQF, Z550 and AU groups were statistically similar ( $p > 0.05$ ). CNT/light group showed the highest color change in all time periods, followed by CNT/self group.

**Conclusion:** The color stability of single-shade bulk fill material and glass hybrid restorative materials is similar to the control group. The color stability of alkasite material needs to be improved.

**Keywords:** Alkasite, bulk fill, cention N, color stability, glass hybrid

This study was presented as an oral presentation at the "Zonguldak Bülent Ecevit University Faculty of Dentistry 1<sup>st</sup> International Congress of Dentistry" (September, 2022) with the title "Comparative Evaluation of Color Stability of Alkasit Restorative Material with Different Restorative Materials".

## INTRODUCTION

The physicochemical characteristics of dental materials have undergone continuous improvements since their initial appearance in the dentistry market. Furthermore, novel products are consistently being introduced to address current problems associated with restorative materials.<sup>1</sup> The rapid application of restorative materials is especially important in hard-to-reach areas and with uncooperative patients. Therefore, in recent years, manufacturers have focused on simplifying techniques.<sup>2</sup>

Composite resin materials are among the most commonly used tooth-colored restoration materials.<sup>2,3</sup> In order to achieve effective light transmission and adequate polymerization, it is recommended that composite resins

are applied by a layering technique with a maximum of 2 mm. However, there are disadvantages of this technique: it is time-consuming as each layer requires separate light polymerization, there is a possibility of air bubbles between layers, and there is a risk of moisture contamination.<sup>1,3</sup> Recently, bulk-fill composite resins have been produced which overcome these problems as it has been stated that these products can be applied up to a thickness of 4-5 mm thickness at one time.<sup>4</sup> Another simplification development in composite resins has been the production of single-shade composite resins that have a "chameleon effect".<sup>2</sup> In this way, time is not wasted with shade selection. Today, some companies have started to produce composite resins with both bulk-fill and single-shade properties.

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Another product line that manufacturers are working on to simplify procedures is 'self-adhesive' restorative materials. Self-adhesive materials are chemically bonded to hydroxyapatite via monomers that can erode enamel and dentin, and this eliminates the need for a separate adhesive application step. These self-adhesive materials can also be applied as bulk-fill.<sup>5-7</sup>

Recently, a new self-adhesive bulk-fill material Cention N (Ivoclar Vivadent, Schaan, Liechtenstein) has been introduced. This material, defined as alkasite, is essentially a subgroup of composite resin. It is a tooth-colored material, but its mechanical properties have been noted as comparable to an amalgam. It can release fluoride ions and can be polymerized light-cured or self-cured.<sup>8,9</sup>

Another group of self-adhesive bulk-fill restorative materials produced in recent years is "glass hybrid restorative materials".<sup>10</sup> These materials were introduced to the market to overcome the disadvantages of conventional glass ionomer materials and resin composites without compromising their advantages. The latest version of these materials, Equia Forte HT (GC, Tokyo, Japan), manufactured in 2019, can release fluoride ions and can be used for permanent posterior restorations.<sup>10,11</sup>

Today, with increasing aesthetic demands, the color match and color stability of restorative materials have become important in determining the longevity

of a restoration, even in the posterior region. There are, however, few studies on the color stability of the new bulk-fill restorative materials mentioned in the literature.<sup>9,12,13</sup>

The present study aims to evaluate the color stability of bulk-fill restorative materials with different properties over different time periods when immersed in a coffee solution.

The hypothesis formulated for this purpose is "The type of restorative material will not affect color stability".

### METHODS

Only restorative materials were used in this study. It was not tested on humans or animals and no materials derived from humans or animals were used. Therefore, ethics committee approval is not required. All procedures were carried out in accordance with the ethical rules and the principles.

The present study exclusively utilized restorative material in an in vitro setting, which eliminated the need for ethical approval. All specimens and phases of the experiment were handled by the same operator. The manufacturer, classification, content, and application procedure of the materials used in the study are listed in **Table 1**. The flowchart of the experimentation method is illustrated in **Figure 1**.

Table 1. Manufacturer, classification, content, and application procedure of the materials used in the study				
Group	Material	Classification	Content	Application Procedure
CNT/light CNT/self	Cention N, A2 (Ivoclar Vivadent, Liechtenstein)	Alkasite Restorative Material: Self-curing with light-curing option, Bulk fill, Self adhesive	Liquid: UDMA DCP Aromatic aliphatic-UDMA PEG-400DMA Powder: Barium aluminum silicate glass filler Ytterbium trifluoride Isofiller Calcium barium aluminum fluorosilicate glass filler Calcium fluorosilicate (alkaline) glass filler	Mix 2 drops of liquid to 2 measuring spoons of powder until smooth. Condense using a plastic filling instrument. Light cure: for 20 sec  Self-cure :5 min (from the start of mixing)
EQF	Equia Forte HT, A2 & Equia Forte Coat (GC Corporation, Japan)	Glass hybrid restorative material: Bulk fill, self adhesive	Liquid: Polybasic carboxylic acid, water Powder: Fluoroaluminosilicate glass, polyacrylic acid, iron oxide Coat: Methyl methacrylate, colloidal silica, camphorquinone, urethan methacrylate, phosphoric ester monomer	Mechanically mixed for 10 sec Deposited in inside mold and closed on the surface by a mylar strip Wait for 10 min Equia Forte Coat was applied and light-cured for 20 sec
AU	Aura Bulk-fill universal shade (SDI, Australia)	Universal bulk fill composite	Diurethane dimethacrylate (3-20% wt.) Bis-EMA UDMA TEGDMA Bis-GMA Amorphous SIO Barium alumino-borosilicate glass Pre- polymerized filler (81% wt.)	Light cure for 20 sec using a high powered LED curing light
Z550	Filtek Z 550 A2, (3M ESPE, USA)	Nanohybrid resin composite	Bis-GMA UDMA TEGDMA PEGDMA Bis-EMA Silica Zirconia	Light cure for 20 sec using a high powered LED curing light

Bis-EMA: Bisphenol A ethoxylate dimethacrylate; Bis-GMA: Bisphenol A glycerolate dimethacrylate; PEGDMA: Polyethylene glycol dimethacrylate; TEGDMA: Triethylene glycol dimethacrylate; UDMA: Urethane-dimethacrylate

### Specimen Preparation

G\*power software (version 3.1.9.4) was used to determine the sample size. The minimum number of specimen required for each group was determined to be 9, with 95% confidence (1-α) and a f=0.655 effect size. Considering the possible specimen loss, the study was planned with 10 specimens for each group.

The study groups were formed as follows:

- CNT/self: Alkaside restorative material cured / self-cure mode (Cention N)
- CNT/light: Alkaside restorative material cured / light-cure mode (Cention N)
- EQF: Glass hybrid restorative material (Equia Forte HT)
- AU: Single-shade bulk-fill composite resin (Aura Bulk Fill)
- Z550: Control group, nanohybrid composite resin (Filtek Z550)

A total of 50 samples (n=10) were prepared and placed in standard (2 mm × 10 mm) disk-shaped metal molds. A mylar strip was placed to cover the surface of the sample and then a glass slide was added to create pressure. This allowed excess material to be forced out of the slide. The groups to be cured as self-cure (CNT/self and EQF) were left to set as specified in the manufacturer's instructions. The light polymerized specimens were cured using an LED lamp (SmartLite Focus, Dentsply, USA) for 20 sec from a distance of 1 mm (1000 mW/cm<sup>2</sup>). After the glass slide was removed, additional polymerization was performed for 10 seconds from the top surface of the samples. Then, the top surfaces of all specimens were polished with course (10 sec), medium (10 sec), fine (10 sec), and super fine (10 sec) aluminum oxide impregnated discs (OptiDisc, Kerr Corporation, USA) respectively, with a micromotor of 10,000 rpm set and with linear movements under dry conditions.<sup>14</sup> A new disk was used for each specimen. A surface sealant (Equia Coat, GC, Japan) was applied to the EQF group according to the manufacturer's instructions and polymerized with the same LED lamp. The specimens were kept in distilled water at 37°C for 24 h to allow the polymerization to complete.

### Staining of the Specimens

To prepare the coffee solution, a soluble granulated coffee was chosen. It was prepared using 2 g of coffee per 200 ml of boiling water and allowed to stand until the solution temperature reached 37°C. 1.5 mm Eppendorf tubes were used to hold the specimens separately in the solution and these tubes, in which the solution and specimen were placed, were kept at 37°C to replicate oral conditions. The specimens were kept in the coffee solution for a total of 12 days representing 1 year clinically.<sup>15</sup> The solutions in the tubes were changed daily.

### Color Measurement

Color measurement was performed at 5 time points: T0 (initial), T1 (336min), T2(24h), T3 (144h), T4 (12 days). (Figure 1). The color measurement at T0 was taken before the staining procedures. At time points T1, T2, T3, and T4, the stained specimens were removed from each Eppendorf tube and washed with distilled water for 10 sec, and then dried with paper for 10 sec. Color measurement was then performed immediately and this procedure was repeated for each specimen individually. Color measurement was performed on a gray card with a contact-type spectrophotometer (VITA EasyShade V, VITA Zahnfabrik, Germany) using the CIE L\*a\*b\* system. The 3-point measurement mode was selected, and the instrument was re-calibrated after each measurement. The averages of the obtained L\*, a\*, and b\* values were recorded. The total color difference (ΔE) for each specimen was calculated using the following equation:

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

$$\Delta L=L2^*-L1^*$$

$$\Delta a=a2^*-a1^*$$

$$\Delta b=b2^*-b1^*$$

The colour changes between T1 and T0, T2 and T0, T3 and T0, and T4 and T0 were named ΔE1, ΔE2, ΔE3, and ΔE4 respectively.

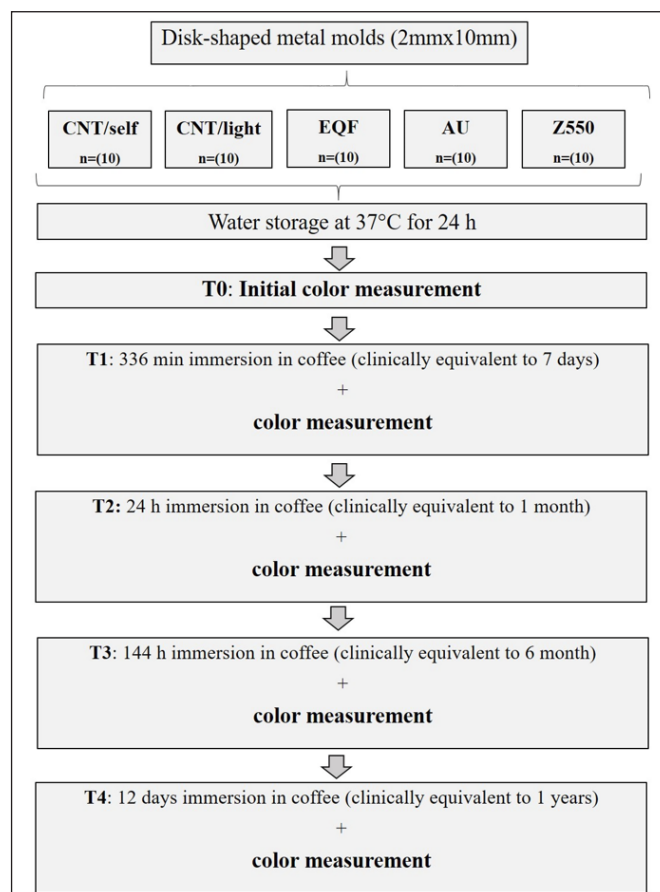


Figure 1. The flowchart of the experimentation method.

### Statistical Analysis

The normality of the data was examined with the D'Agostino & Pearson omnibus normality test. After it was determined that the data were suitable for normal distribution, the One-way ANOVA analysis and Tukey test were performed. The statistical significance level was accepted as  $p < 0.05$  and a piece of statistical analysis software (GraphPad Prism 6.0, GraphPad Software, La Jolla, CA, USA) was used for analysis.

### RESULTS

The  $\Delta E1$ ,  $\Delta E2$ ,  $\Delta E3$ , and  $\Delta E4$  color change values, together with their statistical similarities and differences are summarized in Table 2.

Group	$\Delta E1$ Mean $\pm$ SD	$\Delta E2$ Mean $\pm$ SD	$\Delta E3$ Mean $\pm$ SD	$\Delta E4$ Mean $\pm$ SD
CNT/light	12.2 $\pm$ 1.496 <sup>a</sup>	15.53 $\pm$ 1.768 <sup>a</sup>	25.03 $\pm$ 3.278 <sup>a</sup>	40.79 $\pm$ 4.671 <sup>a</sup>
CNT/self	8.635 $\pm$ 1.09 <sup>b</sup>	13.64 $\pm$ 1.947 <sup>b</sup>	19.05 $\pm$ 1.435 <sup>b</sup>	30.82 $\pm$ 3.077 <sup>b</sup>
EQF	2.006 $\pm$ 0.366 <sup>c</sup>	3.22 $\pm$ 0.6878 <sup>c</sup>	4.891 $\pm$ 0.492 <sup>c</sup>	16.24 $\pm$ 2.455 <sup>c</sup>
Z550	2.703 $\pm$ 0.5753 <sup>c</sup>	4.86 $\pm$ 0.7523 <sup>cd</sup>	6.477 $\pm$ 0.936 <sup>cd</sup>	18.23 $\pm$ 1.98 <sup>c</sup>
AU	3.07 $\pm$ 0.6107 <sup>c</sup>	4.974 $\pm$ 0.859 <sup>d</sup>	8.173 $\pm$ 0.833 <sup>d</sup>	19.26 $\pm$ 1.78 <sup>c</sup>

Superscript lowercase letters compare the means in each column. There is no statistically significant difference between means shown with the same superscript letter ( $p > 0.05$ ).

When the results of  $\Delta E1$  were evaluated (Figure 2):

CNT/light (12.2 $\pm$ 1.496) group showed significantly higher color change than the other groups. Clinically unacceptable color change ( $\Delta E > 3.3$ ) values were observed in CNT/light and CNT/self (8.635 $\pm$ 1.09) groups. The color change values of EQF (2.006 $\pm$ 0.366), Z550 (2.703 $\pm$ 0.5753) and AU (3.07 $\pm$ 0.6107) groups were statistically similar ( $p > 0.05$ ).

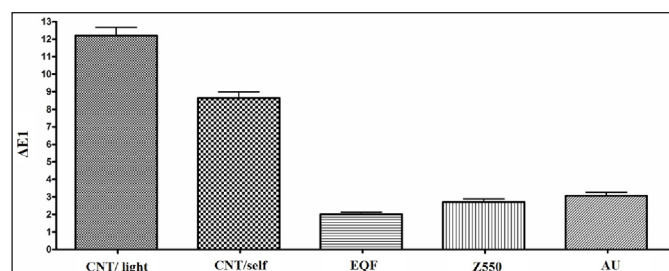


Figure 2.  $\Delta E1$ : Mean color change values according to groups between T1-T0.

When  $\Delta E2$  results were evaluated (Figure 3):

Although the color change values of groups EQF (3.22 $\pm$ 0.6878) and Z550 (4.86 $\pm$ 0.7523) were statistically similar, an unacceptable color change ( $\Delta E > 3.3$ ) was observed in all groups except EQF. CNT/light (15.53 $\pm$ 1.768) showed significantly higher color change than all other groups ( $p < 0.05$ ).

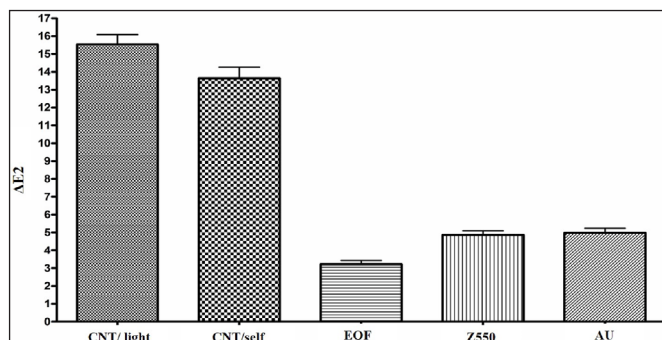


Figure 3.  $\Delta E2$ : Mean color change values according to groups between T2-T0.

When  $\Delta E3$  results were evaluated (Figure 4):

An unacceptable color change ( $\Delta E > 3.3$ ) was observed in all groups. CNT/light (25.03 $\pm$ 3.278) showed significantly higher color change than all other groups. The color change value of EQF (4.891 $\pm$ 0.492) was significantly lower than CNT/light, CNT/self (19.05 $\pm$ 1.435), AU (8.173 $\pm$ 0.833) and similar to Z550 (6.477 $\pm$ 0.936).

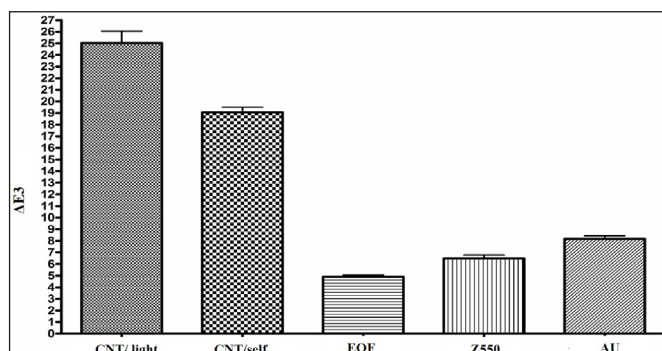


Figure 4.  $\Delta E3$ : Mean color change values according to groups between T3-T0.

When  $\Delta E4$  results were evaluated (Figure 5):

An unacceptable color change ( $\Delta E > 3.3$ ) was observed in all groups. Group CNT/light (40.79 $\pm$ 4.671) showed significantly higher color change than all other groups ( $p < 0.05$ ). Group CNT/self (30.82 $\pm$ 3.077) showed higher color change than groups EQF (16.24 $\pm$ 2.455), Z550 (18.23 $\pm$ 1.98) and AU (19.26 $\pm$ 1.78) ( $p < 0.05$ ). The color change values of EQF, Z550 and AU groups were statistically similar ( $p > 0.05$ ).

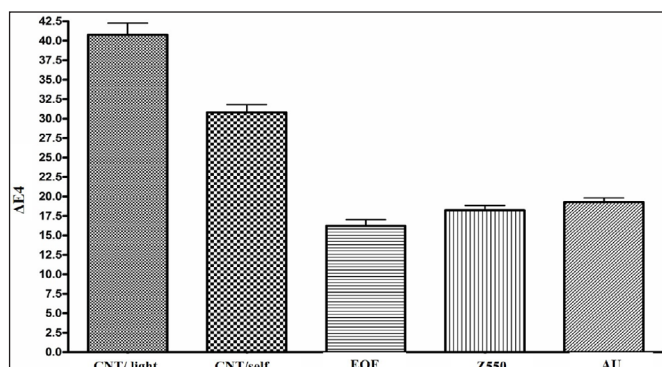


Figure 5.  $\Delta E4$ : Mean color change values according to groups between T4-T0.

## DISCUSSION

In the present study, the color stability of current, bulk-fill restorative materials with different properties was investigated by comparing them with a commonly used nanohybrid composite resin. In the color analysis of all time periods, both light-cure and self-cure modes of the alkasite restorative material showed significantly higher color change than the other groups. Therefore, the hypothesis "The type of restorative material will not affect color stability" was rejected.

Restorative materials are constantly exposed to food and beverages. In the literature, the most commonly used solutions to evaluate the color stability of composite resins are coffee, tea, red wine, and cola.<sup>16</sup> There is no definite application procedure regarding the temperature of the coloring solutions, the residence time of the materials in the solutions, and the frequency of changing the solutions. However, there are studies in the literature in which the coloring solution was used at different temperatures, such as 37°C, room temperature, or at the recommended consumption temperature. The most preferred method is to use the solution at 37°C to simulate the oral environment as used in the present study.<sup>16</sup>

In studies investigating the color stability of restorative materials, immersion times in coloring solutions also varies. It has been stated that a cup of coffee is consumed in about 15 min. and the average daily coffee consumption per person is 3.2 cups, and in many studies, soaking in coloring solutions was made based on this average value.<sup>15,17</sup> When composite resin materials are in contact with liquids, most of the water absorption by the organic matrix occurs in the first 4 days and the highest absorption occurs during the first week. Color pigments in colorant solutions can enter the resin matrix through water. Since the colorant solution also has a tendency to follow water absorption, most coloration occurs in the first week.<sup>18</sup> The most commonly evaluated immersion time in studies is clinically 7 days.<sup>16</sup> In the present study, immersion times corresponding to 7 days, 1 month, 6 months, and 1 year were selected to detect early and relatively later color changes.

Similar to the present study, the clinically acceptable threshold value  $\Delta E \leq 3.3$  has been accepted in many studies when color change is calculated with the CIE  $L^*a^*b^*$  system.<sup>9,16</sup>

The alkasite restorative material groups (CNT/light and CNT/self) showed clinically unacceptable color change at all color analysis times. In addition, the CNT/light group showed significantly more discoloration than the CNT/self-group at all time points. The authors have found only two studies in the literature comparing the color stability of different curing modes of alkasite restorative material.<sup>12,19</sup>

In one of these studies, specimens were immersed in cherry juice, iced tea, and distilled water. For cherry juice and iced tea, the self-cured Cention N group showed less coloration than the light-cured group. The groups kept in distilled water showed similar color changes. In addition, a different color change calculation system, the CIEDE 2000 system, was used in the aforementioned study.<sup>12</sup> Despite the methodological differences, we can conclude that the mentioned study does not support the present study.

In the other study on the subject, the specimens were kept in distilled water without using coloring solution. After 28 days of immersed in distilled water, the light-cure Cention group showed higher color change than the self-cure Cention group.<sup>19</sup> Discoloration of restorative materials in distilled water without a staining solution may be due to changes in the interface between unreacted monomers, fillers and resin matrix, oxidation of the resin matrix and is defined as intrinsic discoloration.<sup>20</sup> Although the aforementioned study<sup>19</sup> provides some information, it cannot be compared to the present study because the present study does not have a methodology where only intrinsic coloration can be evaluated.

In a study investigating the surface roughness and flexural strength values of light-cured and self-cured modes of Cention N, the self-cure mode of Cention N was found to be superior and the authors stated that this was probably due to the slow and prolonged curing of self-cured Cention N.<sup>8</sup> This may explain the higher coloration of the CNT/light group in the present study.

In a study examining the color stability of Cention N and a glass hybrid restorative material (Equia forte, GC, Japan), the color analysis of specimens immersed in a coffee solution at different time periods showed that the color stability of the glass hybrid restorative material was superior to Cention N.<sup>21</sup> Although the aforementioned study used a previous version of the glass hybrid restorative material than that used in the present study, the results of the two studies support each other.

In another study using coffee immersion and thermal aging, Cention N (self-curing) showed lower color stability than nanohybrid composite resin, but higher color stability than glass hybrid restorative material.<sup>9</sup> These results are partially different from the present study. However, the glass hybrid restorative material used in the aforementioned study is a previous version of the one used in the present study.

The lack of additional methods such as brushing simulation and thermal aging are limitations of this study. More extensive studies on the color stability of simplified bulk-fill restorative materials should be conducted.

The authors also believe that one of the reasons for the conflicting results in the literature regarding the color stability of Cention N material is that the material is in powder-liquid form and is mixed by hand. Testing the newly available capsule form may yield more consistent results.

## CONCLUSION

The conclusions reached within the limitations are as follows:

The color stability of glass hybrid restorative material and single-shade bulk-fill composite resin materials is similar to that of conventional nanohybrid composite resin. The color stability of the alkasite restorative material needs to be improved. Alkasite restorative material changes color more when light-cured than when self-cured.

## ETHICAL DECLARATIONS

**Ethics Committee Approval:** Only restorative materials were used in this study. It was not tested on humans or animals and no materials derived from humans or animals were used. Therefore, ethics committee approval is not required.

**Informed Consent:** Only restorative materials were used in this study. Therefore, informed consent is not required.

**Referee Evaluation Process:** Externally peer reviewed.

**Conflict of Interest Statement:** The authors have no conflicts of interest to declare.

**Financial Disclosure:** The authors declared that this study has received no financial support.

**Author Contributions:** All the authors declare that they have all participated in the design, execution, and analysis of the paper, and that they have approved the final version.

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