EFFECT OF ACCELERATED AGING ON COLOR STABILITY OF DUAL-CURED SELF-ADHESIVE RESIN CEMENTS AND LIGHT-CURED RESIN CEMENT

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ABSTRACT

Introduction: Color stability of resin cements plays an essential role in esthetics of ceramic restorations. Resin cements are widely used in dentistry and have different clinical applications.

Objectives: This study aimed to evaluate the effect of accelerated aging on color stability of dual-cured self-adhesive resin cements and light-cured resin cement.

Material and methods: In this study, 15 disc-shaped specimens (45 specimens in total) were made using a stainless steel mold from three types of cement: two dual-cured self-adhesive resin (G-Cem LinkForce, GC, Corporation, Tokyo, Japan, and Panavia SA, Kuraray, Osaka, Japan), and a light-cured resin (Choice2, Bisco Inc., Schaumburg, IL, USA) cement. Baseline colorimetry was performed using Easy Shade (Vita, Germany) according to CIE Lab system. Samples underwent aging process for 100 hours, after which the final colorimetry of samples was performed. Color differences between samples were measured and analyzed using ANOVA and Tukey tests with SPSS version 22. *P*-value < 0.05 was considered statistically significant.

Results: All resin cements had lower L* values after accelerated aging, but it was not significant in Panavia SA group ($p = 0.121$). Color change in the dual-cured self-adhesive resin cements (G-Cem LinkForce and Panavia SA) was associated with clinically acceptable values of ΔE ($\Delta E < 3.5$).

Conclusions: Color stability of adhesive resin cements after accelerated aging was evaluated, and statistically significant color changes were observed within a clinically acceptable range in dual-cure adhesive resin cements.

Key words: accelerated aging, color stability, dual-cured self-adhesive resin cement, light-cured resin cement.

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INTRODUCTION

Today, the use of all-ceramic restorations is increasing due to excellent esthetics and biocompatibility [1]. All-ceramic crowns and veneers are among the most popular restorations in the anterior area because of their natural and appealing appearance [2]. Many clinical trials have reported long-term success of resin-bonded restorations, such as veneers, inlays, onlays, and all-ceramic veneers. On the other hand, patients' demands for such restorations are on the rise [2]. Since all types of fragile ceramic restorations require a solid infrastructure to protect them against occlusal forces, and bond to the structure of the tooth and restoration at the same time, there is a stronger need to improve their esthetic qualities, with the development of various ceramics types [3]. Resin cements are commonly used to cement all-ceramic restorations because they have good esthetic properties, low

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solubility, high bond strength to teeth, mechanical properties, and ceramic support [4].

Resin cements can be activated chemically, using light, or by both mechanisms (dual-cure) [5]. Dual-cure cements are activated by a combination of light and chemical mechanisms. This type of activation leads to improved mechanical properties, including tensile strength, elastic modulus, and stiffness compared with other curing methods [4]. Another advantage of dual-cured cements is their ability to cure in deeper areas where light does not reach, and the light intensity is low [4, 5]. Despite the advantages of dual-cured cements, their amino catalyst turn yellow after a while, affecting esthetic appearance of the restoration [6]. Dual-cured resin cements have low color stability compared to cements activated by light only [7].

In 2002, self-adhesive resin cements were introduced [3, 8]. These systems are dual-cured, and the lack of preparation with bonding agents has made their clinical application interesting [9]. Self-adhesive cements do not need tooth surface preparation before application, are very easy to use, and like zinc phosphate and polycarboxylate cements, are applied in one clinical stage [8]. Manufacturers of self-adhesive resin cements claim that they are resistant to moisture, release fluoride, and have no post-operative sensitivity [9]. Since the smear layer is not removed, they have no post-operative sensitivity, and unlike zinc phosphate and polycarboxylate cements, they can tolerate moisture and release fluoride like glass-ionomer [8]. Therefore, all these factors simplify the procedure, reduce technical sensitivity, and improve adaptation to clinical needs [9]. In addition, self-adhesive cements are expected to have favorable esthetics, mechanical properties, and excellent dimensional stability [8].

Resin cements for veneers are usually activated using a light-curing device [7], and light-cured cements have various advantages, including durability, need-based setting, and enhanced color stability. However, the use of light-cured cements is limited to situations, such as veneers or shallow inlays where the thickness and color of the restoration do not affect photo-polymerization ability of the cement [9]. Therefore, color stability of the underlying cements might play an essential role in the long-term success of restorations [2]. Previous studies have shown that luting cements with different colors under porcelain restorations can affect the final color of restoration [7]. Discoloration of the underlying cement might be visible from the edges or through the restoration [7]. Duration of restoration depends on color stability of the material and sometimes the technique of using the cement [9]. However, resin cements have extrinsic and intrinsic color changes [2, 10]. In particular, intrinsic discoloration is a challenge, which is attributed to chemical properties of the material [7]. Internal factors are attributed to the composition of material, the amount of filler, and the type of activator [7]. External factors include surface absorption of substances, such as food and colored beverages as well as UV, saliva, and heat [7]. For this reason, the dentist needs to know these color changes while choosing a suitable cement [10]. Color changes can be assessed visually or with color measuring devices. The use of color measuring instruments, such as colorimeters or spectrophotometers, has become common

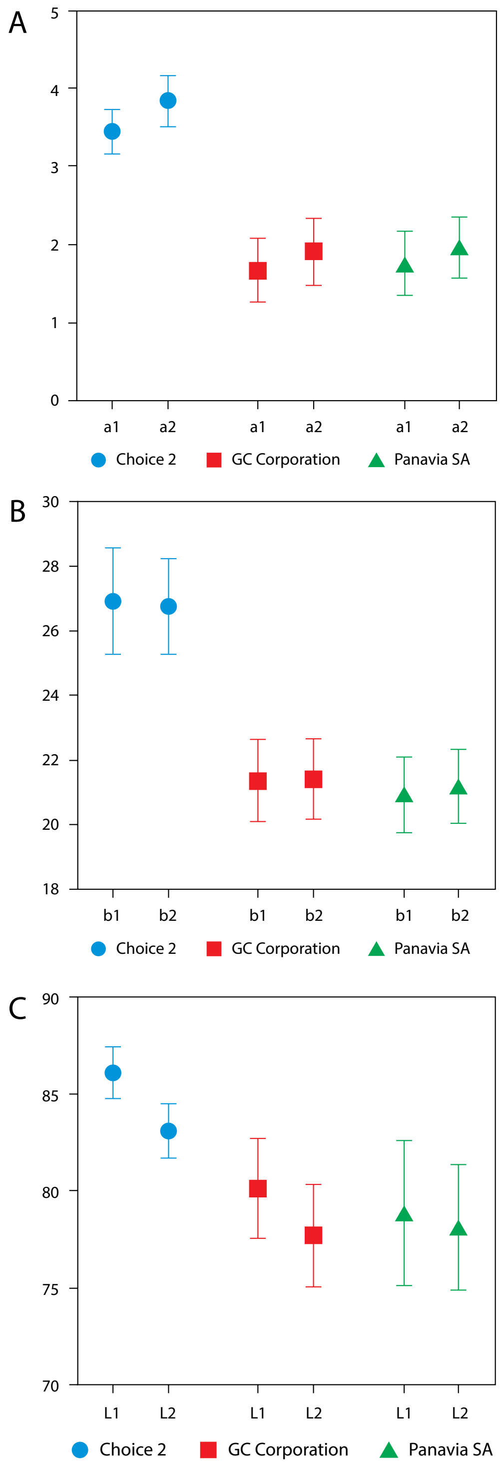

FIGURE 1. Comparison of changes in different color parameters in the three resin cements before and after accelerated aging

due to accuracy, standardization, and quantitative values in reporting the color [11].

In-vitro studies have shown that accelerated artificial aging is an effective way to assess the durability of dental materials. This technique simulates clinical parameters to examine the effects of different conditions on the material, and has been used to evaluate color stability of dental restorations, including resin cements and all-ceramic restorations. These clinical conditions include exposure to ultraviolet light and continuous changes in temperature and humidity [9].

OBJECTIVES

This study aimed to evaluate the color stability of three widely used resin cements after accelerated aging.

MATERIAL AND METHODS

In this *in-vitro* study, two dual-cured self-adhesive resin cements (GC, Corporation, Tokyo, Japan and Panavia SA, Kuraray, Osaka, Japan) and a light-cured resin (Choice2, Bisco Inc., Schaumburg, IL, USA) cement were used. Resin cement compositions are presented in Table 1. From each cement, 15 disk-shaped samples (45 samples in total) with a 1.5 mm thickness and 10 mm diameter (1.5 mm × 10 mm) were prepared with a stainless steel mold. Each material was prepared according to the manufacturer's instructions and placed within the mold. A thin glass slab was placed on the upper surface of the mold to create a smoothest surface. A relatively uniform pressure was applied using two fingers to remove excess cement. Light-curing was performed with a light-cured device (Blue Phase C8, Ivoclar Vivadent, Schaan, Lichtenstein) at 750 mW/cm^2 intensity. To ensure light intensity of the device, the intensity of the device was measured using a radiometer (Bluephase Meter II, Ivoclar Vivadent, Amherst, NY, USA) after every 10 exposure.

The surface of dual-cured self-adhesive resin cement (GC, Corporation, Tokyo, Japan) and Panavia SA (Kuraray, Osaka, Japan) was cured for 20 seconds according to the manufacturer's instructions.

The surface of light-cured resin (Choice2, Bisco Inc., Schaumburg, IL, USA) cement was exposed twice for 40 seconds, according to the manufacturer's instruction to ensure curing of all areas.

The samples were carefully separated from the mold, and the final thickness of each specimen was verified by a digital caliper (Mitutoyo Corp., Tokyo, Japan). The specimens were polished by dental composite resin polishing paper discs (OptiDisc4200, Kerr, Switzerland) in a low-speed handpiece with medium, fine, and superfine discs for 30 seconds each in one direction, respectively. The samples were stored in distilled water in an incubator (SHI 55, SHIMAZ, Iran) at 37°C for 24 hours to complete polymerization [2]. All the samples were coded and prepared for initial colorimetry. Baseline color values of the samples were measured by Easy Shade (Vita, Germany) and according to CIEL*a*b standard system in daylight D65, and a standard viewing angle of 10 degrees [2, 12]. Color parameters were measured based on the values of L, a, and b in a white background, three times for each sample, and their average was reported as the amount of color. 'L' was the lightness or value, 'a' was the amount of red and green, and 'b' was the amount of yellow and blue [12]. Then, the samples were placed in a weathering machine (Xenotest, USA, Atlas, Beta) for 100 hours under irradiance of 50 W/m^2 conditions, 40 minutes of only xenon radiation, and 20 minutes of xenon radiation and water shower at 37°C, with 95% humidity.

The color of the samples was measured again by Easy Shade (Vita, Germany) according to the standard CIEL*a*b system. Moreover, color comparison before and after the procedure was calculated using the following formula:

$\Delta E = [(L2 - L1)\ 2 + (a2 - a1)\ 2 + (b2 - b1)\ 2]^{1/2}$.

The ΔL, Δa, and Δb values in Table 2 show a decrease in brightness (L) and color shift to red (+a) and yellow (+b) in both. Normality of data was ensured to compare the effect of accelerated aging on the color stability of dual-cured self-adhesive resin cements and light-cured resin cement. Results of Shapiro-Wilk test demonstrated data normality. Data were analyzed using Shapiro-Wilk, ANOVA, and Tukey post-hoc tests using SPSS version 22 ($p \leq 0.05$).

TABLE 1. Materials used in the study and their composition according to their manufactures

Manufacturer	Composition	Type	Resin cement
GC Corporation, Tokyo, Japan	Dimethacrylate, UDMA, 4-META, phosphoric ester monomer, fluoro-alumino-silicate glass, camphorquinone	Self-etch-adhesive dual cure	G-Cem
Bisco Inc., Schaumburg, IL, USA	Bis-GMA (bisphenol glycol dimethacrylate), strontium glass, amorphous silica, initiator	Light-cured resin cement	Choice2
Kuraray Noritake Dental	10-Methacryloyloxydecyl dihydrogen phosphate (MDP), Bis-GMA, triethylene glycol dimethacrylate (TEGDMA), hydrophobic aromatic dimethacrylate, silanated barium glass filler, silanated colloidal silica, dl-camphorquinone, benzoyl peroxide, initiators, hydrophobic aliphatic dimethacrylate	Self-etch adhesive dual cure	Panavia SA

RESULTS

Table 2 illustrates the color parameter values before and after ageing in this research. It shows the mean and standard deviation (SD) of differences between the initial and the second measures of each CIE coordinates (ΔL, Δa, and Δb) for each group of specimens.

After accelerated ageing, L* values, which measured brightness, tended to decrease in all the cements, and statistically significant differences were found in all the tested cements ($p < 0.05$). In all the tested cements, statistically significant differences in a* values, representing redness index and inclination toward red color, were found in the three groups ($p < 0.05$). After accelerated ageing, b* values, which indicated yellowness index, were elevated to incline towards yellow color in the G-Cem group and the Panavia SA group, with a statistically significant difference in both the groups ($p < 0.05$). After accelerated ageing, b* values in Choice2 tended to decline towards blue color, but there were no statistically significant changes ($p = 0.304$).

Calculating the numeric values of color, as indicated in Table 3, revealed the results of change in color (ΔE). The ΔE* values for color difference ranged from 1.69 to 3.08. Panavia SA had the lowest ΔE* values after accelerated ageing, G-Cem was the second, and Choice2 was the third. The results of ANOVA test showed a significant difference between the case of ΔE in the three study groups ($F(2.42) = 23.418$, $p < 0.001$).

Tukey test was employed to compare all the parameters of the three groups. The results of Tukey test demonstrated a significant difference between the means of the three groups ($p < 0.05$). The ΔE values of the three resin cement brands were compared using Tukey's post-hoc test. The results revealed significant differences between all the groups, as presented in Table 4.

Based on *p*-value, the null hypothesis on the similarity of color change in dual-cured self-adhesive resin cements (G-Cem and Panavia SA) and light-cured resin cement (Choice2) was rejected. In other words, in the present study, the color changes in the cements were different.

DISCUSSION

Today, resin cements are widely used for dental restorations. A major issue in the esthetics of dental restorations is the color stability of these cements, which has been addressed in many studies [13]. Therefore, the current research evaluated the color stability of two dual-cured self-adhesive resin (GC Corporation, Tokyo, Japan, and Panavia SA, Kuraray, Osaka, Japan) and a light-cured resin (Choice2, Bisco Inc., Schaumburg, IL, USA) cements after accelerated aging because physicochemical reactions, such as visible and ultraviolet

TABLE 2. Descriptive statistics (mean ± SD) for ΔL, Δa, and Δb values in the groups

Group	*n*	Mean	Standard deviation
Choice2 light-cure			
ΔL	15	–3.02	0.33
Δa	15	0.39	0.07
Δb	15	–0.15	0.55
G-Cem dual-cure			
ΔL	15	–2.45	0.18
Δa	15	0.24	0.06
Δb	15	0.02	0.04
Panavia SA dual-cure			
ΔL	15	–0.72	1.71
Δa	15	0.21	0.27
Δb	15	0.25	0.31

TABLE 3. ΔE value of adhesive resin cements

	n	Mean	Standard deviation	Minimum	Maximum
Choice2 light-cure	15	3.08	0.41	2.53	4.05
G-Cem dual-cure	15	2.46	0.18	2.20	3.02
Panavia SA dual-cure	15	1.69	0.85	0.23	3.40
Total	45	2.41	0.79	0.23	4.05

TABLE 4. Comparison of ΔE values of the study groups after aging

(I) group ΔE	(J) group ΔE	Mean difference (I-J)	Standard error	Sig.
Choice2 light-cure	G-Cem dual-cure	0.62221*	0.20433	0.011
	Panavia SA dual-cure	1.39562*	0.20433	0.000
G-Cem dual-cure	Choice2 light-cure	–0.62221*	0.20433	0.011
	Panavia SA dual-cure	0.77341*	0.20433	0.001
Panavia SA dual-cure	Choice2 light-cure	–1.39562*	0.20433	0.000
	G-Cem dual-cure	–0.77341*	0.20433	0.001

**Mean difference significant at 0.05 level.*

light, temperature, and heat, can all cause intrinsic color changes in cements over time [14]. The accelerated aging process exposes samples to xenon light with moisture and light cycles, to simulate oral conditions [2]. Complete model of the clinical conditions is impossible [15], but it can stimulate the oxidation of amines, which are the activator of resin cements [16]. Almeida *et al.* [17] investigated the effect of accelerated aging on the darkening of samples, and showed that even if the samples were not the subject to an extrinsic color change, the cause of intrinsic color change were oxidation and the presence of unreacted components during polymerization process. Therefore, color stability can best be evaluated via accelerated aging.

In the present study, the samples were exposed to heat, humidity, and radiation for 100 hours in an atmospheric simulator (Xenotest Beta+, Atlas, USA) to investigate accelerated aging, because typically, placing the samples for 300 hours in a weather simulator is equivalent to one year of clinical use [18]. However, Kokhar *et al.* [4] noted that discoloration occurs mostly in the first 100 hours of accelerated aging. Therefore, in this study, 100 hours of accelerated aging was selected to show the initial color change of resin cement.

Easy Shade device and CIE Lab system were used to evaluate optical properties more accurately. Although the color of dental restorative materials can be measured visually [19], such a measurement is preferred with precise tools to provide summarized and reliable results [2]. Johar *et al.* [27] used the Easy Shade device to measure the color of samples due to its' reliability.

In the current study, after measuring the color of samples, three values of L, a, and b, and as a result, ΔE was obtained. $\Delta E < 3.5$ was considered clinically acceptable, which is consistent with Vichi *et al.* [20] findings, where ΔE values of < 1 are not detectable by human eyes and values of 1-3.5 can only be detected by dentists and are clinically acceptable, whereas values of > 3.5 can be detected by any individual and are considered clinically unacceptable.

The present study showed that the ΔE^* values for color difference ranged from 1.69 to 3.08. Panavia SA had the lowest ΔE^* values after accelerated ageing, G-Cem was the second, and Choice2 was the third, which had clinically acceptable ΔE ($\Delta E < 3.5$) values for all tested resin cements. A color difference of $0.5 \leq \Delta E \leq 1$ cannot be clinically perceived, while color difference of $1 \leq \Delta E \leq 2$ can be perceived only by 50% of observers. Therefore, most of the studies defined perceptibility threshold (PT) of color difference as $\Delta E = 1$. A value of $\Delta E \leq 3.7$ can be perceived by 100% of observers. Hence, all the tested resin cements had clinically acceptable ΔE ($\Delta E < 3.5$) values. Nevertheless, the color change of the light-cured resin (Choice2, Bisco Inc., Schaumburg, IL, USA) was close to the clinical limit ($\Delta E > 3.5$). The unacceptable amount of ΔE might be attributed to the thickness of the cement disks (approximately 2 mm), and was higher than the thickness used in the clinic. Various studies have used samples thicker than normal for the clinic to evaluate color stability [16]. Lu and Powers [21] and Smith *et al.* [22] reported unacceptable color changes for dual-cured and light-cured resin due to the thickness of the cement disks used in these studies (1-2 mm). However, in clinical conditions, the cements are about a few tenths of a millimeter in thickness, and only the edge of the cement is exposed to the oral environment, and the rest is under ceramic restorations. Tabatabai *et al.* [19] observed acceptable color change, and reported that any color change in a thin layer of cement in clinical conditions is less detectable than the same amount of color change in the cement with thicker layers in laboratory conditions.

In the present study, all the cements except G-Cem showed a shift toward yellow color, which is consistent with a study of Kilinc *et al.* [2], who reported that in all resin cements after aging, there was an increase in parameters a and b. The change in the Δa and Δb values of the samples placed in atmospheric conditions simulator, is due to the difference in the concentration and the characteristics of the optical primers, the resin composition, the type and size of the fillers, and fewer amines [23]. Changes in yellow values can be partially attributed to changes in camphorquinone levels due to increased polymerization. Camphorquinone is an initiator in most composite resins and has a yellow color that becomes almost colorless after polymerization. However, since the degree of polymerization of the composite cannot reach 100%, some yellowness remains in the restoration systems [24]. In addition, the physicochemical properties of the monomers used in the resin matrix might affect resistance to discoloration [25]. Another explanation for the tendency to yellow color can be attributed to Bis-GMA exposure to UV light, humidity, and temperature, because the hydroxyl and ester groups of Bis-GMA molecules in resin cements absorb water and are hydrolyzed, and these hydrolytic effects have been reported to play an important role in changing the color of base resins [26]. UDMA-based and Bis-GMA-based materials modified with UDMA are more resistant to discoloration than those with a Bis-GMA base [35]. Therefore, in this study, the cause of severe discoloration of light-cured samples (Choice2, Bisco Inc., Schaumburg, IL, USA) might be the presence of Bis-GMA in their composition, which causes discoloration after exposure to temperature and humidity.

In this study, discoloration fell within the clinically acceptable range ($\Delta E < 3.5$) for dual-cured self-adhesive cements (GC Corporation, Tokyo, Japan). Tabatabaei *et al.* [19], Alamooz *et al.* [5], and Archegas *et al.* [16] showed that ΔE values for dual-cured cements were < 3.5, consistent with the present study. The reason for the more acceptable color stability of dual-cured resin cements than light-cured resin cements might be the complexity of the setting mechanism of light-cured cements.

There are other factors helping a stability, such as those affecting the degree of polymerization at a certain depth from the surface after light-curing, including the concentration of photo-initiator, which must be sufficient to react the appropriate wavelength. Additional factors include the amount of filler and particle size that affect the scattering of light rays, and the distance between the tip of the light source and the exposed surface area, which should be approximately 1 mm to achieve proper exposure. Importantly, the degree of polymerization and the amount of unreacted monomers in these cements is not predictable; therefore, the color change does not follow a specific pattern [25]. Therefore, the reason for greater discoloration of the light-cured cement might be the non-standard distance of the light source from the exposure surface in this study, or the non-standard concentration of photo-initiator and filler and particle size in the cement structure. This is consistent with a study of Tabatabaei *et al.* [19], who reported that dual-cured cements have more color stability than light-cured total-etch cements, and both have more color stability than self-adhesive self-cured cements. They reported that the reason for a higher color stability of dual-cured cements compared with light-cured cements was the complex setting mechanism of light-cured cements.

Numerous other studies have shown that light-cured resins have a lower degree of conversion than dual-cured types; the lower the degree of conversion of the resin, the more prone to discoloration and color instability over time [7, 27]. Yang *et al.* [27] investigated the color stability of new resin cements containing amine or amine-free self-initiators. In their study, one light-cured resin cement and three types of dual-cured cements were used. They concluded that the light-cured cements showed similar color changes like other cements. Papazoglou *et al.* [6] also found that the rate of monomer change and degree of polymerization in dual-cured cements was higher than in light-cured cements.

Contrary to the present study, some studies, including Pissaia *et al.* [28], Kilinc *et al.* [2], and Almeida *et al.* [17] have demonstrated better color stability in light-cured resin cements compared with dual- and self-cured types for the absence of aromatic amines in their composition [4]. This is because the main cause of discoloration of dual-cured resin cements is the oxidation of aromatic amines, which is required to trigger the polymerization process of resins and react with benzoyl peroxide [4]. Aromatic ternary amines (accelerators) make photoreactive by-products that tend to produce discoloration from yellow to red/ brown under the influence of light or heat. Moreover, the presence of unreacted benzoyl peroxide in dual-cured materials leads to more severe discoloration [15]. However, light-cured materials contain only aliphatic amines in their composition, which is the reason for less color change in light-cured cements [16]. Most dual-cured and self-cured cements use benzoyl peroxide and ternary amines to initiate polymerization. However, to prevent discoloration in order to combine suitable mechanical and optical properties, various resin cements are manufactured without amine accelerators [4], such as dual-cured self-adhesive resin (G-Cem and Panavia SA) cements used in the present study. This is the reason for the milder color change of this cement than the light-cured resin cement (Choice2, Bisco Inc., Schaumburg, IL, USA), which consistent with findings of Smith *et al.* [22], Ural *et al.* [29], and Atay *et al.* [30], who reported that resin cements without ternary amine compounds have better color stability.

In this study, a decrease in ΔL was observed in all the samples after aging, which is in line with a study by Archegas *et al.* [16], who reported that resin materials tend to darken after aging.

CONCLUSIONS

The results of the present and previous studies showed color changes of all the resin cements after accelerated aging. However, the color change of dual-cured self-adhesive resin cement G-Cem used in the study showed acceptable values of ΔE (ΔE < 3.5). Moreover, it was found that all the cements showed a color shift towards yellow.

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The study obtained approval of the Bioethics Committee (approval number: IR.AJUMS.REC.1398.587).

CONFLICT OF INTEREST

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

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