

# Color Stability of Bioactive Restorative Materials After Immersion in Various Media

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## ABSTRACT

**Objectives:** The aim of this article is to assess the color stability of the bioactive restorative materials (Activa Bioactive, Beautifil II) compared with the conventional resin composite and resin-modified glass ionomer cement after immersion in different staining solutions overtime. **Materials and Methods:** This is an *in-vitro* study that investigated four different material groups: (1) bioactive composite (ACTIVA Bioactive-Restorative, Pulpdent, USA), (2) Giomer composite (Beautifil II, SHOFU Dental GmbH, Japan), (3) resin-modified glass ionomer cement (Fuji II, GC Corporation, Japan), and (4) resin composite (Filtek Z350XT, 3M ESPE, USA). One hundred samples ( $n=25$  each group) were fabricated using a custom acrylic mold (1 mm thick  $\times$  10 mm diameter) and then immersed in five different staining solution groups: coffee, black tea, cola, mixed berry juice, and saline. Baseline (T0) shade of samples was recorded using two spectrophotometers: VITA Easyshade Digital Advance and a spectrophotometer. Then shade was recorded at the intervals of 7 (T1), 14 (T2), and 28 (T3) days of immersion. Measurements were obtained and then  $\Delta E$  was calculated for each group at each time point. Three-way analysis of variance tests were used to test the interactions between different variables at the 0.05 significance level. **Results:** All specimens showed a significant color change ( $P<0.001$ ), following 7, 14, and 28 days of storage. Activa Bioactive and Filtek Z350 showed the highest color stability overtime in different staining solutions, whereas Fuji II and Beautifil II showed the least color stability. The most significant color change was noticed in the coffee group and then in the mixed berry juice group. **Conclusion:** Resin-based restorative materials showed higher color stability than glass ionomer-based restorations. Both spectrophotometers gave comparable results for materials' color stability.

**KEYWORDS:** Bioactive materials, color alteration, color stability, composite resin, resin-based materials

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## INTRODUCTION

Currently, restorative materials are expected to meet high esthetic demands by patients specially when placing anterior restorations by mimicking the natural teeth in terms of texture, anatomy, and color.<sup>[1,2]</sup> With regard to the concept of minimally invasive dentistry, an inert restorative material is no longer desired. Therefore, advancement in restorative

biomaterials has been focussed on the production of hybrid restorative materials. These bioactive materials as Gionomers and Compomers combine the properties of high fluoride release from glass ionomer cements and

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outstanding aesthetics of composite resin.<sup>[3]</sup> Moreover, they are expected to induce tissue response and promote remineralization, which is usually permissible through the materials ability to release ions and form apatite structures.<sup>[4]</sup> Compomers are polyacid-modified composites containing the main components of glass ionomer cement; therefore, water sorption is required for the acid–base reaction to take place during materials' maturation. However, this has been found to negatively affect the aesthetics properties of Compomers.<sup>[5]</sup> Giomers are composites containing pre-reacted glass ionomer cements as fillers. The amount of fluoride release from Giomer materials is significantly higher than Compomers with comparable mechanical properties and clinical performance.<sup>[6-8]</sup> The balance between the bioactive properties of these emerging materials and their physical and mechanical properties has been a challenge in their development and a subject of major interest in the dental and biomaterial research.<sup>[3]</sup>

The long-term success of esthetic restorations depends largely on the material's resistance to different stains. Therefore, color stability and translucency of esthetic restorations are critical properties to be considered as these restorations are frequently susceptible to intrinsic and extrinsic stains. It has been reported that food and drinks containing colorant can cause staining of the restorative materials affecting their longevity and long-term success.<sup>[1,9-11]</sup> When assessing restorations' success, the color match is evaluated based on the United States Public Health System (USPHS).<sup>[12,13]</sup> Color mismatch of direct restorations has been reported as one of the reasons leading to patients' dissatisfaction and restoration failure, indicating its replacement.<sup>[14]</sup> Among the latest developments in resin composite materials, bioactive composites are becoming greatly desirable among clinicians and researchers. This is due to the remineralizing and anti-caries properties

of these new composite materials, which add a huge benefit due to their use, in addition to the excellent physical and mechanical properties.<sup>[5]</sup> In contrast, there is an increased esthetic demand and awareness among population. Therefore, it is essential to investigate the optical properties of these modified restorative materials to enable a prediction on their long-term esthetic results.<sup>[15]</sup> The purpose of this study was to investigate the stability in color of different tooth-colored restorative materials based on their type and staining solution over time. The first null hypothesis was that there is no difference in the color stability of the investigated materials based on their type, the staining media, or exposure time. The second null hypothesis was that there is no difference in the assessment of color recordings between two spectrophotometers (VITA Easyshade Digital Advance, VITA and CE7000A, X-Rite, Grand Rapids, MI, USA).

## MATERIALS AND METHODS

### SAMPLE PREPARATION

Four types of materials were used in this study [Table 1]: ACTIVA Bioactive-Restorative (Pulpdent, USA), Beautifil II (Shofu Inc., Kyoto, Japan), Fuji II (GC Corporation, Japan), and Filtek Z350 XT (3M/ESPE, St. Paul, MN, USA). The shade of all used materials was A1. This study was reviewed and approved by the Research Ethics Committee at King Abdulaziz University. Ethical approval number was 160-12-20, and this was guaranteed according to the guiding principles for investigational methods found in the Declaration of Helsinki of the World Medical Association.

After a pilot study, the sample size was calculated using a 0.05 alpha value and 80% power to detect a difference of 25%. One hundred samples ( $N=100$ ) were used in this project. Twenty-five discs from each material were fabricated (1 mm thickness  $\times$  10 mm

**Table 1: Summary of the materials used in the study: bioactive composite (ACTIVA Bioactive-Restorative, Pulpdent, USA), Giomer composite (Beautifil II, SHOFU Dental GmbH, Japan), resin-modified glass ionomer cement (Fuji II, GC Corporation, Japan), and resin composite (Filtek Z350XT, 3M ESPE, USA)**

Materials	Types	Shade	Composition	Manufacturer
ACTIVA Bioactive	Reinforced Compomer	A1	Mixture of other methacrylates and diurethane with amorphous silica (6.7%), modified polyacrylic acid (44.6%), and sodium fluoride (0.75%)	Pulpdent, Watertown, MA, USA
Beautifil II	Giomer	A1	BISGMA, TEGDMA, aluminum oxide, silica, aluminofluoro-borosilicate glass filler, pre-reacted glass-ionomer filler, camphoroquinone	Shofu, Kyoto, Japan
Fuji II	RMGIC	A1	58 wt.% Fluoroaluminosilicate, methacrylate, hydroxyethyl, polyacrylic acid and water	GC, Tokyo, Japan
Filtek Z350	Nano-filled composite	A1	Fillers: silica cluster and Zirconia (20 nm). Matrix: Bis-GMA, UDMA, Bis-EMA, PEGDMA, and TEGDMA	Filtek Z350XT, 3M ESPE

UDMA = urethane dimethacrylate

diameter) by injecting the materials into a customized acrylic mold. A mylar strip was used on either side of the mold and the material was gently pressed against a microscopic glass slide and then the excess material was removed.<sup>[16-19]</sup> Each material was then light cured on the top surface using LED curing unit (Elipar FreeLight 2, 3M/ESPE, St. Paul, MN, USA) at 1200 mW/cm<sup>2</sup> power density, activated for a period, following the instructions of the manufacturer for each material. After polymerization, samples were polished with 1200-grit carborundum papers using MetaServ 250 Grinder-Polisher with Vector Power Head-Buehler-USA, under running water.

#### STAINING PROTOCOL

Five samples from each material were assigned to one of the following solutions: 5 mL coffee (Nescafe, Nestle, Syria) (pH 5.1), 5 mL black tea (Lipton, Dubai, UAE) (pH 5.5), 5 mL cola (Coca Cola, Riyadh, Saudi Arabia) (pH 2.7), 5 mL mixed berry juice (Almarai, Jeddah, Saudi Arabia) (pH 4.5), and 5 mL saline (Pharmaceutical Solutions Industry, Jeddah, Saudi Arabia) (pH 7). Each sample was immersed in a separate glass vial and stored separately in an incubator at 37°C (Mettmert, Schwabach, Germany). Staining solutions were replaced daily for 28 days, with the concentration and temperature of the solutions standardized each time they were replaced.<sup>[17,18,20]</sup>

#### COLOR MEASUREMENT

For each sample, the first color measurement was taken as a baseline before immersion in staining solution. The baseline color values were recorded using two spectrophotometers: VITA Easyshade Digital Advance, VITA against white background and CE7000A, X-Rite, Grand Rapids against black background. After 1 week of immersion, specimens were removed from solutions and washed with distilled water. Later, they were placed in an ultrasonic cleaner in separate vials filled with saline (POWERSONIC 405, Hwashin Technology Co.) for 5 min and then dried for 10 s to be ready for color measurement. According to the manufacturer's instructions of VITA Easyshade spectrophotometer, the device was calibrated prior to each color measurement and the probe tip was placed perpendicular and flush to the tooth surface during color measurement. Regarding X-Rite spectrophotometer, the device was also calibrated for reflectance mode with a 6 mm aperture plate according to manufacturer's instructions prior to measurements. This protocol for color measurements using both devices was followed to record color values at 7-, 14-, and 28-days intervals.<sup>[16]</sup> Data were recorded using the Commission Internationale d'Eclairage (CIE) L\*a\*b\* system, and then they were used for

$\Delta E$  calculation from both devices for each time point following the below equation<sup>[21,22]</sup>:

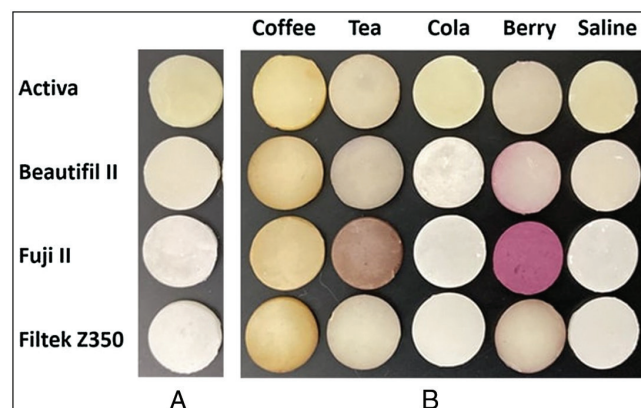
$$\Delta E = \sqrt{(L_{post} - L_{baseline})^2 + (a_{post} - a_{baseline})^2 + (b_{post} - b_{baseline})^2}.$$

#### STATISTICAL ANALYSIS

The mean and standard deviation values were calculated for each group in each test. Data were explored for normality using Kolmogorov–Smirnov and Shapiro–Wilk tests. Data showed parametric (normal) distribution. The paired sample *t*-test was used to compare two groups in related samples. Then repeated-measures analysis of variance (ANOVA) was used to compare between more than two groups in related samples, whereas one-way ANOVA followed by Tukey's *post hoc* test was used to compare between more than two groups in non-related samples. Three-way ANOVA tests were used to test the interactions among different variables. The significance level was set at  $P \leq 0.05$ . Statistical analysis was performed with IBM® SPSS® Statistics Version 20 for Windows (IBM Corp. Released 2011, IBM SPSS Statistics for Windows, Version 20.0, IBM Corp., Armonk, NY, USA).

#### RESULTS

The results of the study revealed that visual color changes were visible for all samples along the storage time at 7, 14, and 28 days with exception of the control group (saline) [Figure 1]. From both color-measuring devices, repeated-measures ANOVA test results indicated that storage time, type of material, and staining solutions have a statistically significant effect on  $\Delta E$  ( $P < 0.001$ ). The most affecting staining solution was found to be coffee (average  $\Delta E = 18.57$ ), followed by mixed berry juice (average  $\Delta E = 14.6$ ) and tea (average  $\Delta E = 12.3$ ) across all groups, except the GIC (Fuji II) group, which was the most significantly affected group



**Figure 1:** Samples show visible differences between baseline (A) and after 28 days (B) in different staining solutions

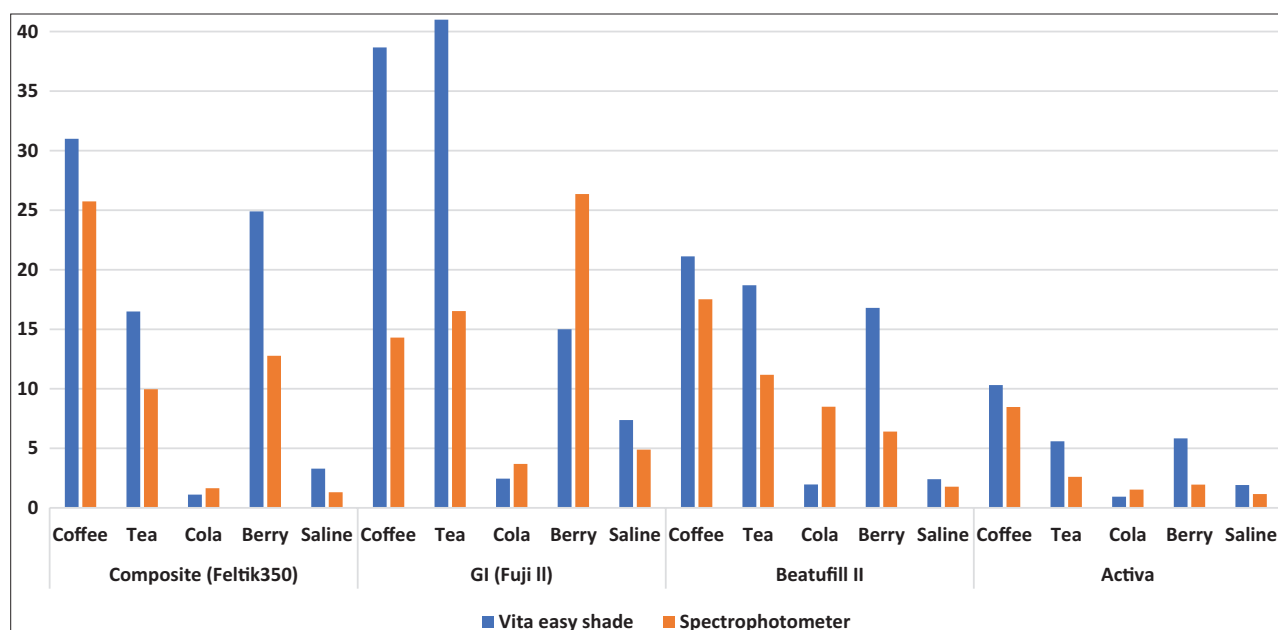


Figure 2:  $\Delta E$  values change after 4 weeks of immersion in different staining solutions that recorded using two devices VITA Easyshade

by mixed berry juice [Figures 2 and 3]. Comparing  $\Delta E$  values of different staining solutions showed significant differences between the materials in terms of color stability ( $P < 0.001$ ). Multiple comparisons of the materials' effect for both devices showed that the GIC (Fuji II) recorded significantly higher  $\Delta E$  values, followed by Beautifil II and then composite ( $P < 0.001$ ) [Table 2 and Figure 3]. The storage time had a significant effect starting from week 1, followed by a significant increase up to week 4, shown on Figure 3. The extent of the effect, however, was determined largely by the materials' type followed by staining solution and the least was storage time [Figure 3].

Comparable results between the two devices were found based on the effect of materials' type and storage time as represented. Both devices showed that resin-based materials have better color stability than GIC-based materials. Moreover, both devices showed that color changes are related to storage time in all groups, as seen in Table 2. On the contrary, significant changes were found when comparing the devices in the same storage media [Figure 2 and Table 2], where  $P$ -values compare the two devices within the samples in each group. Vita Easyshade resulted in a slightly higher  $\Delta E$  value than X-Rite spectrophotometer in all groups.

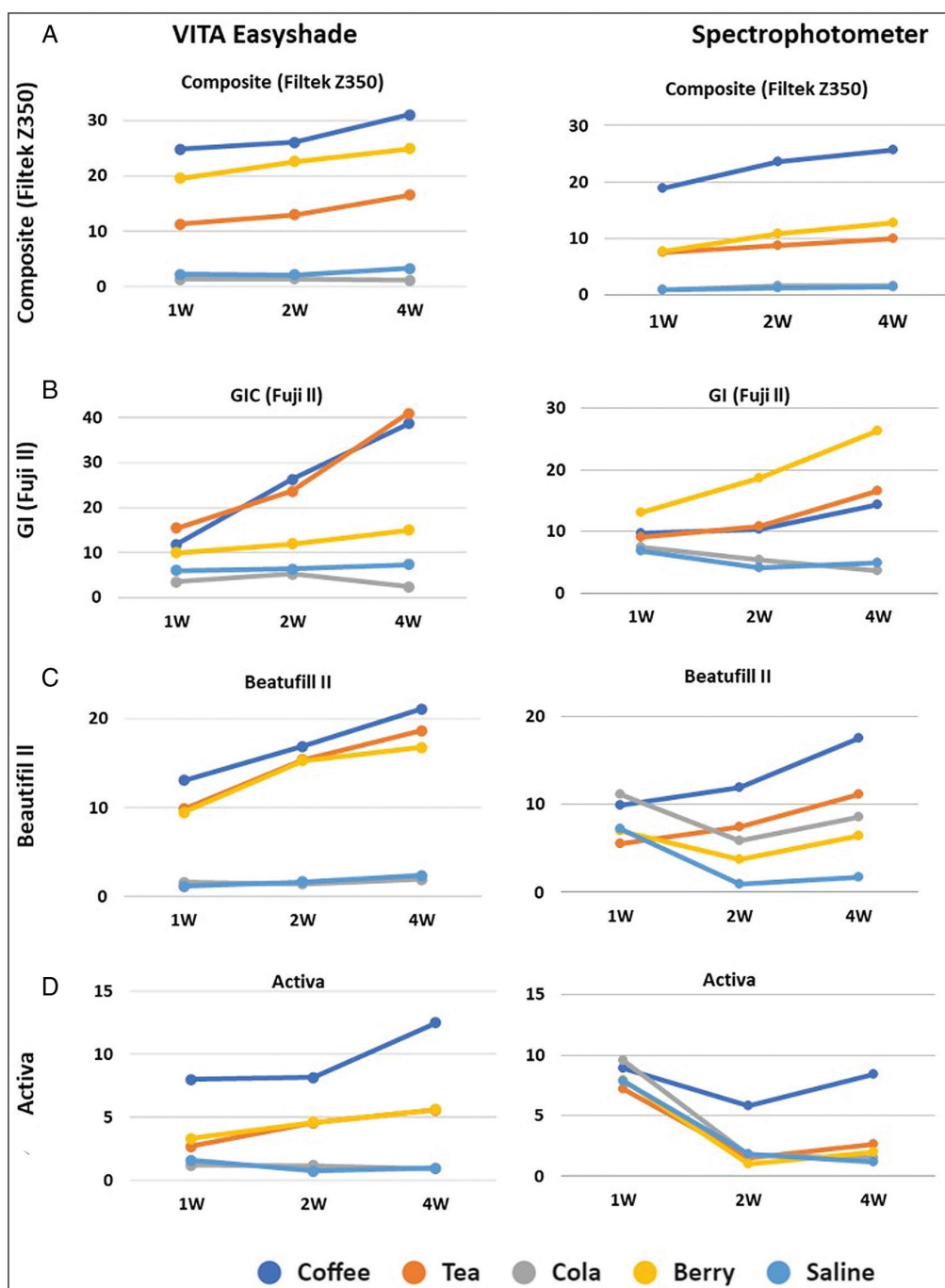
## DISCUSSION

Surface discoloration of esthetic restorative materials is a major problem affecting the durability of these restorations, which indicates the importance of an evaluation of their color stability.<sup>[14]</sup> In the current

study, the color stability of multiple emerging bioactive restorative materials was compared with regular composite and GIC restorative materials. The results indicated that all tested materials had color changes at a period of 28 days; therefore, the first null hypothesis was rejected. Moreover, when comparing two different spectrophotometers, significant changes were found regarding storage media; hence, the second null hypothesis was also rejected.

In order to assess the colorimetric qualities of dental materials in an objective manner, the CIE  $L^*a^*b^*$  system is frequently used. This reduces subjective color perception variability and allows for a consistent method of determining color changes over time. A digital spectrophotometer can reliably detect minor changes in material's brightness as well as hue; however, the significance of these readings should be addressed, as only values equal to or greater than 3.7 can be regarded visually changed and may necessitate restorations' replacement.<sup>[10,22,23]</sup> Regarding the storage media used in this study, staining solutions were chosen on the basis of the most stain-causing and commonly used beverages in the society with distilled water as control media.<sup>[18]</sup>

The results of the current study of all tested materials had color changes at a period of 28 days. Color stability of all tested materials was affected by the staining media and by the materials' type. Coffee and berry juice had the most potent effect on color stability of all samples. Coffee showed significant changes on all axes ( $L^*$ ,  $a^*$ , and  $b^*$ ), resulting in darker samples'



**Figure 3:** Line graphs showing  $\Delta E$  values from two devices: VITA Easyshade and Spectrophotometer X-Rite CE7000A, for the four tested materials after immersion in different solutions for 7, 14, and 28 days. (A) Filtek Z350, (B) GIC (Fuji II), (C) Beautifil II, and (D) Activa Bioactive composite

color. Mixed berry juice was most positively affected in color alteration by increased  $a^*$ , leading to reddish samples. Tea, on the contrary, changes mainly on  $b^*$  leading to more yellowish hue of the samples. After 28 days, all samples resulted in a color change greater than 3.7 which is found to be unacceptable clinically [Table 2]. The current results of the study agree with previous studies in which coffee resulted in the greatest

color alteration, whereas cola had the lowest color alteration.<sup>[17,18,20]</sup> There are multiple factors related to the staining solutions that may contribute to the resulted color alteration in the tested material, such as temperature and acidity. Both factors can significantly affect the stability of the color of the restorative materials, resulting in an unpleasant esthetic outcome, and can necessitate restorations' replacement.<sup>[18,24,25]</sup>

**Table 2: Means and standard deviation of  $\Delta E$  values of material groups after 4 weeks' immersion in different staining solutions ( $P$ -values comparing both spectrophotometers)**

Material Device	Composite (Filtek Z350)						GIC (Fuji II)						Beautifil II						Activa					
	VITA			X-Rite			VITA			X-Rite			VITA			X-Rite			VITA			X-Rite		
	Easyshade			CE7000A			Easyshade			CE7000A			Easyshade			CE7000A			Easyshade			CE7000A		
	Mean	SD		Mean	SD	$P$ -value	Mean	SD		Mean	SD	$P$ -value	Mean	SD		Mean	SD		Mean	SD		Mean	SD	$P$ -value
Coffee	31.01	3.89	15.74	5.31	<0.003		38.66	4.92	24.30	2.39	0.003		21.12	3.13	17.52	1.55	0.003		10.31	0.92	8.47	0.41	0.002	
Tea	16.49	2.94	9.97	0.63	<0.001		41.86	3.64	16.53	3.96	<0.001		18.70	1.98	11.18	1.46	<0.001		5.59	0.87	2.61	0.80	<0.001	
Cola	1.12	0.52	1.65	0.46	0.062ns		5.33	0.61	3.69	1.01	0.260ns		1.97	0.91	8.50	1.68	0.260ns		0.94	0.35	1.54	0.83	0.090ns	
Berry	24.90	2.80	8.77	0.68	<0.001		NA	NA	26.36	4.78	<0.001		16.80	2.63	6.41	0.93	<0.001		5.84	1.07	1.96	0.49	<0.001	
Saline	3.30	0.35	1.32	0.55	<0.001		3.53	0.81	4.89	0.30	0.067ns		2.41	0.65	1.78	0.59	0.067ns		1.92	0.48	1.16	0.33	0.010	

It was explained previously that the temperature of coffee may act as an aging factor and the coffee itself is known to cause significant staining, and the grape juice discoloration was minimal compared with coffee.<sup>[17]</sup> Regarding solutions' acidity, the current findings imply that color changes after immersion in various staining solutions are not pH-related alterations. Cola, which had the lowest pH (2.38), had no noticeable color changes, and coffee, which was just mildly acidic (pH 6.28), had the most color changes.<sup>[18]</sup> This is in agreement with a previous study that resulted in cola having the least color effect on tested materials.<sup>[18]</sup> However, to allow for a standardized monitoring of color along this study, both temperature and acidity were controlled by standardizing the temperature of hot beverages (tea and coffee) and the commercial brand of all beverages along the experiment. Moreover, the solution was replaced every 2 days with a fresh beverage simulating continuous consumption of these beverages. In the clinical practice, it is important to report a careful history including habits and diet, which should be taken from the patients as it has been reported that using some medications, certain diets which are full of flavors and dyes, smoking, and other external factors would positively affect the severity of staining of resin composites. The detailed knowledge of the patients' daily routine would result in a better expectation on the longevity and esthetic outcome of the restorative materials.<sup>[17]</sup>

Similar to the result of the current study, previous studies also concluded that color stability of the restorative material is highly influenced by their type.<sup>[17,22]</sup> Resin-based materials were found to have more color stability than GIC-based materials, and this is directly related to their composition and filler particle. Gonulol *et al.*<sup>[19]</sup> have compared the color stability of Giomers and nanohybrid Filtek Z550 composite, and they have concluded that the discoloration values were significantly higher in the Giomer (Beautifil) group to a degree that this may have an adverse effect on their esthetic performance. They have referred this observation to the materials' type as Giomers are fluoride-releasing materials and this may have created voids within the matrix and possible roughness, which may contribute to less color stability of Giomer restorative materials.<sup>[19]</sup> Similar findings by Özdaş *et al.*<sup>[16]</sup> were explained by the differences in the materials matrix and particle size, as the hydrophilic resin matrix and larger particle size in Beautifil II resulted in earlier and higher color alternation after 30 days. Moreover, several studies that evaluated Activa Bioactive reported similar results to this study in which Activa Bioactive showed more color stability when

compared with other resin composites.<sup>[17,26]</sup> This can be attributed to the fact that materials containing Bis-GMA have lower color stability compared with those that contain urethane dimethacrylate but with higher water absorption.<sup>[27,28]</sup> This explains the reasons that Aactiva Bioactive performed better than resin composite (Filtek Z350) in the current study, as Aactiva Bioactive contains no Bis-GMA.<sup>[29]</sup> Moreover, the fact that the resin matrix type plays an important role in the stain resistance explains comparable color stability between Beautifil II and resin composite (Filtek Z350) as they both share the same resin matrix Bis-GMA.

Comparing Beautifil II and Fuji II, it was found in this study that Fuji II showed lower resistance to stain. These results are in alignment with an earlier study, which was explained by the higher ion release, as a result of ionic changes between the environment and the material in addition to the larger filler size in Fuji II.<sup>[30]</sup>

Throughout the experimental period,  $\Delta E$  values are found to steadily increase. Considering the measurement of immersion duration alone, it was found that the most apparent color change occurred after 28 days. This time-dependent increase is consistent with prior research, which found that samples exposed to staining solutions for longer periods of time had greater color shifts, regardless of the type of storage media.<sup>[22,31,32]</sup> However, this study was different from previous ones, in that it displayed a patient's daily intake *in vitro* and employed a different methodology.

Regarding the spectrophotometers used in this study, two digital shade-determining devices have been used to evaluate the color change in these materials; spectrophotometers X-Rite CE7000A and Vita Easyshade. So far, both devices were used in previous studies separately with no reports on comparing both devices, and this study is the first to provide a simultaneous comparison of both devices.<sup>[16-18,22]</sup> The results in the current study found that there are significant differences in the values of  $\Delta E$  between both devices. These differences were found when both devices were compared within each group. However, these differences did not change the fact that resin-based materials have better color stability than GIC-based materials. Moreover, the results of storage time effect on the materials' color stability were confirmed by both devices. Therefore, it can be concluded that both devices resulted in different  $\Delta E$  values, but this did not affect the comparison between the color stability performance of the tested materials in this study.

Some limitations in this research include lacking the investigation of the materials' surface roughness and its

effect on the color changes. Also, when solutions were replaced to simulate patients' consumption, samples were not brushed, which could have altered the staining susceptibility of the materials. The impact of surface roughness, brushing, and thermal stresses on the color stability of restorative materials should be studied in future investigations.<sup>[33]</sup>

## CONCLUSIONS

Based on the findings of the current study, color stability of restorative materials was found to be dependent mainly on their type, then staining solution type, and storage time. The most affecting staining solution was coffee, followed by tea and mixed berry juice. Moreover, Aactiva Bioactive and Beautifil II materials showed promising results in color stability when compared with other conventional restorative materials. Both spectrophotometers (VITA Easyshade and X-Rite) gave comparable results for color stability of the materials, regardless of the differences in the calculated  $\Delta E$  values.

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## CONFLICTS OF INTEREST

The authors declare no conflict of interest.

## AUTHORS' CONTRIBUTION

Conceptualization, SS and RA; methodology, SS; AB, TA, RA; software, RA; validation, SS, RA, AB and TA; formal analysis, SS; investigation, AB, TM; resources, RA; data curation, AB, TA; writing – original draft preparation, TA, AB, RA; writing – review and editing, SS; visualization, SS; supervision, SS, RA; project administration, SS, RA; funding acquisition, none.

## ETHICAL POLICY AND INSTITUTIONAL REVIEW BOARD STATEMENT

This study was reviewed and approved by the Research Ethics Committee at King Abdulaziz University (ethical approval number 160-12-20), this was guaranteed according to the guiding principles for investigational methods found in the Declaration of Helsinki of the World Medical Association.

## PATIENT DECLARATION OF CONSENT

Not applicable.

## DATA AVAILABILITY STATEMENT

All data generated or analysed during this study are included in this published article. Detailed datasets are available from the corresponding author on reasonable request.

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