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# Effect of surface protection, staining beverages and aging on the color stability and hardness of recently introduced uncoated glass ionomer restorative material

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## ABSTRACT

**Aim:** Evaluation of the effect of coating, staining beverages and aging on the color stability and hardness of recently introduced glass ionomer (GI) restorative material and to determine whether there was a correlation between these two variables.

**Materials and methods:** Two commercially available conventional GI restorative materials were used; Ketac™ Universal Aplicap™ and Ketac™ Fil Plus Aplicap™ GI restoratives. A total of 84 disc-shaped specimens (5 × 2 mm) were prepared and divided into 3 main groups (n = 28). Fifty six specimens were prepared from Ketac Universal Aplicap where half of them was coated (CU) and the other half was uncoated (U) and 28 coated specimens from Fil Plus Aplicap™ (CF) that act as a control group. Coating was performed with Ketac Glaze. Each group was further subdivided into 4 subgroups (n = 7) according to the beverages (tea, coffee, coke and distilled water). Color changes (ΔE) and hardness (MPa) were measured by scanning spectrophotometer and Vickers's hardness (VH) test respectively. Measurements were recorded at the baseline, after 7 and 30 days of aging in each beverage. Chemical analysis of the glass powders was performed by EDXA. Additionally, the filler size was examined by the SEM. The data were statistically analyzed (P ≤ 0.05).

**Results:** The CU subgroups possessed lower ΔE than U subgroups in tea and coffee. The impact of staining beverages and aging on the ΔE was material's dependent. The CU subgroups recorded higher VH than the U and CF subgroups after aging in coke (30 days). Aging of the U subgroups in tea and coke significantly decreased its VH. The SEM revealed smaller average filler size in Ketac Universal Aplicap (7.2 μm) than Ketac Fil Plus Aplicap (17.9 μm).

**Clinical significance:** It is advisable to use the recently introduced uncoated GI restorative material for patients who are not consuming tea and/or coffee but with surface protection to maintain its color acceptability up to 3 year clinically. Not all color changes could be associated with surface degradation.

## 1. Introduction

Glass ionomer was introduced to adhesive dentistry years ago, despite the fact it was commonly named as glass ionomer cement (GIC), it is widely used as a standalone restorative material as well as an intermediary base or liner. This material has been developed since first introduced by Wilson and Kent in 1972 to introduce a flow of new and modified products until recently [1–4]. Modifications and trials were developed to overcome some of the inherent limitations of GICs such as

the snap setting reaction, sensitivity to moisture during the early stages of setting and their inferior mechanical properties when compared to other esthetic restoratives. However, the chemical bonding of GICs to moist tooth structure without an intermediate agent with their fluoride release are continuously encouraging the dentists to use such material more routinely [5–8].

The oral environment is very challenging to both dental professionals and manufacturers. Glass ionomer like any other restoratives materials is exposed to saliva, oral microflora and frequently consumed

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colored food and beverages. The physico-chemical properties of GI especially the conventional type are critical during the early stages of setting and are greatly influenced by the surrounding environment. Any unfavorable surrounding condition might negatively affect the GI restorative material's esthetic and mechanical properties which subsequently will influence its long term clinical performance [9]. This is usually related to the early moisture sensitivity of GI which decreases its structural stability during its initial setting phase [10]. The composition of GICs probably affects their surface roughness and hardness [11]. The deterioration of the surface of the restorative material can lead to esthetic problems. Some studies revealed that conventional GI restoratives suffered the maximum color changes especially in acidic conditions, when compared to other esthetic restorative materials [12–14]. In an attempt to overcome the problem of dehydration and hydration of GICs, there were suggestions to use surface protective materials which could help to maintain the water balance within the material during setting. Additionally, such surface protecting agents could reduce the uptake of stains by the restoration. Protective resin coating of low viscosity showed to be effective with conventional GI restorations [14].

One Recently introduced GI restoration with modified chemical composition has been claimed to be easier in application, stronger than conventional formulations and more stable in the challenging oral environment than other conventional GI restorations, even without the need for a protective coating application. Such material with its adhesive quality and fluoride anticariogenic effect could be considered an ideal solution for many difficult clinical situations. However, the color stability and surface durability of such material (Ketac Universal Aplicap, 3M ESPE, USA) have not been evaluated yet. Therefore, the present study was designed to evaluate the color stability and hardness of the newly introduced GI restorative material with and without coating when subjected to 4 beverages (tea, coffee, coke and water) at different aging periods (at base line, 7 and 30 days) in comparison to coated conventional GI restorative material. Additionally, to determine whether there is a correlation between color changes and hardness alteration of the tested GI restorative materials under all tested conditions. The null hypotheses were that surface protection has no effect on the color stability and hardness of recently introduced uncoated GI restorative material after aging in various beverages.

## 2. Materials and methods

Detailed description of the selected materials as mentioned by the manufacturers is presented in Table 1.

### 2.1. Sample preparation and grouping

The sample size was calculated according to the paper published by Bagheri et al., 2005 [12], using IBM™ SPSS™ SamplePower™ (Version 3.0.1). The criterion for significance (alpha) has been set at 0.05.

**Table 1**

Detailed description of the used conventional glass ionomer restorative materials as mentioned by the manufacturers.

Material and Manufacturer	Lot no.	Shade	Composition
Ketac™ Universal Aplicap™ 3M ESPE Dental Products, St. Paul, USA	582332	A3	Conventional glass ionomer restorative material composed of: Traditional glass ionomer restorative with a self-cure (acid-base) mechanism composed of: Powder: Oxide glass > 95 wt %. Liquid: Water (40–60 wt %), Copolymer of acrylic acid – maleic acid (30–50 wt %), Tartaric acid (1–10 wt) and Benzoic acid (< 0.2 wt %)
Ketac™ Fil Plus Aplicap™ 3M ESPE Dental Products, St. Paul, USA	582527	A3	Conventional glass ionomer restorative material composed of: Traditional glass ionomer restorative with a self-cure (acid-base) mechanism composed of: Powder: Oxide glass (± 100 wt %) Liquid: Water (40–55 wt %), Copolymer of acrylic acid – maleic acid (35–55 wt %), Tartaric acid (5–10 wt)
Ketac™ Glaze, 3M Deutschland GmbH, Germany	538500	–	Varnish for glass ionomer composed of: 2-Propenoic acid, 2-methyl-, [(3-methoxypropyl)imino] di-, 2,1-ethanediy ester (1–5 wt %) and Dicyclopentylidimethylene Diacrylate

Coating will include 3 levels, with 28 cases per level. The effect size (f) is 0.40, which yields power of 0.87. Storage media will include 4 levels, with 21 cases per level. The effect size (f) is 0.70, which yields power of 1.00. Accordingly, a total of 84 disc-shaped specimens (56 Ketac Universal Aplicap and 28 Ketac Fil plus Aplicap) were prepared and divided into 3 main groups (n = 28) as follows: coated Ketac Universal Aplicap (CU), uncoated Ketac Universal Aplicap (U) and coated Ketac Fil Plus Aplicap (CF). Teflon mold with 2 central holes (5 mm in diameter and 2 mm in thickness) was utilized for specimen's preparation. These holes were first placed over a microscopic glass slide topped with a Mylar strip. The encapsulated glass ionomer restoratives were mixed by the RotoMix apparatus (3M-ESPE, Seefeld, Germany) and fabricated at room temperature, according to the manufacturer instructions of each material. The holes were filled with the GI restorative material and immediately the surface of the material was covered by a Mylar strip and pressed (weight of 200 g) with a microscope slide to obtain a smooth and flat surface. Seven minutes later, the glass slide and matrix were removed. The top and bottom surfaces of the specimens receiving the protective coating (CU and CF) were covered by the Ketac™ Glaze while the U specimen's group were left uncoated. Another Mylar strips was gently pressed on the coated surfaces of the specimens followed by light curing of the coating for 20 s (LED curing unit (Radii Plus, High power 1500 mW/cm<sup>2</sup>, SDI, Australia). After curing, the coated specimens were removed from the Teflon mold and the sides of the specimens were additionally coated with the ketac™ Glaze to ensure complete coverage of the coated specimens. All the prepared specimens were stored in distilled water for 24 h at 37 °C in the incubator (Cbm. Torre Picenardi (CR), Model 431/V., Italy). Each main group (n = 28) was further subdivided into 4 subgroups (n = 7) according to the storage media (tea, coffee, coke and distilled water). All the specimens were prepared under standard conditions by the same operator to eliminate human variables. The sequence of specimens allocated to groups was determined using a computer generated random sequence table ([randomn.org](http://randomn.org)) which was generated by another author. In order to implement the allocation sequence, numbered containers were used and the sequence was concealed till the day of the intervention (before aging of the specimens in the different staining beverages) and chosen by an independent co-worker. The operator was blinded and unaware of the type of materials tested during color stability and hardness measurements. The same specimens were used for the measurements of the color stability and the hardness to be able to correlate the results. The bottom surface of each specimen was used for color stability while its top surface was marked to be used for the measurements of the hardness throughout the study.

### 2.2. Preparation of staining solutions

The solutions were prepared and their pH values were measured by the pH meter (pHep, Pocket-sized pH Meter, Hanna instrument, Rhode Island, USA). The tea solution was prepared by immersing 5 teabags

(Lipton Yellow Label Tea, Unilever Mashreq-Tea Company, Egypt) into 1000 ml of boiled water (pH 4.9). To prepare the coffee solution, 20 g of coffee (Nescafe Classic, Nestle Egypt) was poured into 1000 ml of boiled water (pH = 4). Both solutions were stirred every 30 min for 10 s until they cooled down to room temperature (25 °C), and then filtered through a filter paper. The third staining beverage was 355 ml coke (Coca-Cola, Coca-Cola Co., Egypt) stored at room temperature (pH = 2.7). The fourth subgroup of specimens were aged in distilled water (pH = 7.14). The specimens were immersed into 20 ml of each beverage and kept in the incubator (Cbm. Torre Pecenardi (CR), Model 431/V., Italy). The solutions were freshened and stirred once daily to reduce the precipitation of particles in the staining beverages [15].

### 2.3. Color measurements

Scanning spectrophotometer (UV-VIS-NIR Shimadzu 3101 PC, Japan) was used to measure the color changes of the tested materials. The base line color measurement of all specimens was performed after storage in distilled water for 1 day from the bottom surface. Then the specimens were aged in the staining beverages (tea, coffee, coke, and distilled water). Subsequent color measurements were taken after 7 and 30 days of aging in each beverage. All the specimens were kept in the incubator at 37 °C between measurements. Before each measurement, the specimens were removed from the beverages and rinsed thoroughly with distilled water for 120 s. Excess water on the surfaces was removed with tissue papers and the specimens were allowed to dry. Prior to measurement, the spectrophotometer was calibrated according to the manufacturer's instructions by using the supplied white calibration standard. The specimens were placed in the center of the measuring head of a spectrophotometer with the aid of a black metallic attachment. This attachment was used in order to provide repetitive measurements for each specimen from the same region. Furthermore, this setup prevented any external light source from entering the system. Three measurements were taken at a time for each specimen from one fixed predetermined point on the bottom surface of each specimen. Color changes were characterized using the Commission International d'Eclairage L\*a\*b\* color space (CIE L\*a\*b\*). Total color differences were expressed by the formula:

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

where,  $\Delta L$ ,  $\Delta a$ , and  $\Delta b$  are differences in L\*, a\*, and b\* values after storage in distilled water for 24 h (Base line) and then after aging in the 4 beverages (7 and 30 days). In this 3-D color space, the 3 axes are namely L\*, a\*, and b\*. The L\* value is a measure of the whiteness or brightness of an object. The a\* value is a measure of redness (positive a\*) or greenness (negative a\*). The b\* value is a measure of yellowness (positive b\*) or blueness (negative b\*). Furthermore, the color difference thresholds have been taken into consideration to correlate between the obtained in vitro results and the clinical situations. Recently, the 50:50% perceptibility threshold (PT) and the 50:50% acceptability threshold (AT) of the CIE in dentistry were found to be  $\Delta E_{ab} = 1.2$  and  $\Delta E_{ab} = 2.7$  respectively [16]. Therefore, qualitatively,  $\Delta E > 2.7$  can be considered unacceptable, and  $\Delta E < 1.2$  imperceptible to the normal observer.

### 2.4. Hardness test

The hardness of the marked top surface of each specimen was measured by the Vickers microhardness instrument (HMV Microhardness Tester, Shimadzu, Japan). A 200-gf load was applied through the indenter with a dwell time of 15 s. Three readings were taken for each specimen, and the average Vickers Hardness (VH) value was recorded (MPa). The base line hardness measurements of all the specimens were taken after 1 day of storage in distilled water. Then the measurements were taken after 7 and 30 days of aging in each beverage

(tea, coffee, coke, and distilled water). All the specimens were kept in the incubator at 37 °C between the measurements. Distilled water was used to thoroughly rinse each specimen for 120s. Afterward, each specimen was blotted dry using a filter paper then subjected to the microhardness measurement test.

### 2.5. Chemical analysis and imaging

Representative samples of the glass powder of the 2 tested materials were chemically analyzed with the scanning electron microscope (SEM, Model Quanta 250 FEG, Field Emission Gun, accelerating voltage 30 KV. FEI Company, Netherlands) fitted with the energy dispersive X-ray analyses (EDX), using accelerating voltage 30 K.V., magnification  $14\times$  up to 1000000 and resolution for Gun.1n). Additionally, the SEM was used to detect the filler size range of the glass powder of each tested GI restorative material.

### 2.6. Statistical analysis

Numerical data were explored for normality by checking the data distribution and using Kolmogorov-Smirnov and Shapiro-Wilk tests. All data showed non-parametric (non-normal) distribution. Data were represented as median and range values. Kruskal-Wallis test was used to compare between the effect of protective coating, staining beverage and aging period. Friedman's test was used to compare between the color changes ( $\Delta E$ ) values at different storage times periods. Dunn's test was used for pair-wise comparisons. The significance level was set at  $P \leq 0.05$ . Statistical analysis was performed with IBM (IBM Corporation, NY, USA), SPSS Statistics Version 20 for Windows (SPSS, Inc., an IBM Company). Additionally, Spearman's correlation coefficient was used to determine the correlation between micro-hardness and color change.

## 3. Results

### 3.1. Color change ( $\Delta E$ )

The effect of the **protective coating** on the color changes ( $\Delta E$ ) of the tested materials after aging in the different staining beverages is presented in [Table 2](#). The results revealed that there were no statistically significant differences in the median color changes ( $\Delta E$ ) between the CU, CF and U subgroups when aged in **distilled water** from base line to 7 days and from base line to 30 days. However, from 7 to 30 days of aging in distilled water, the CU and U subgroups were not significantly different and both have significantly higher median  $\Delta E$  value than the CF subgroup. When the tested subgroups were aged in **tea**, the U subgroup possessed the statistically highest median  $\Delta E$  from base line to 7 days and from base line to 30 days. However, there was no statistically significant difference between the median  $\Delta E$  values of the CU and CF subgroups at these aging periods. Furthermore, the U subgroup showed the highest significant median  $\Delta E$  values while the CF subgroup revealed the lowest when they were aged in tea from 7 days to 30 days.

Additionally, there was no statistically significant difference between the median  $\Delta E$  values of the tested materials when aged in **coffee** from base line to 7 days. Meanwhile, the U subgroup showed the highest significant median  $\Delta E$  values from base line to 30 days of aging in coffee. However, there were no statistically significant differences between the median  $\Delta E$  values of the CU and CF subgroups; both showed the lowest significant median ( $\Delta E$ ) values. Furthermore, the value of the  $\Delta E$  of the CU and U subgroups after aging (from 7 to 30 days) in coffee were not significantly different and both were higher than those of the CF subgroup. Finally, there was no statistically significant difference between the median  $\Delta E$  values of the CF and U subgroups when they were aged in coke from base line to 7 days; both showed statistically higher significant median  $\Delta E$  values than the CU subgroup. Meanwhile, there were insignificant differences between the

**Table 2**  
Effect of protective coating on the median color change values ( $\Delta E$ ) of the tested materials.

Staining beverages	Time	CU		CF		U		P-value
		Median	Range	Median	Range	Median	Range	
Distilled water	Base line–7D	2.38	0.77–6.26	3.51	2.60–3.87	3.13	1.78–5.70	0.070
	Base line –30D	5.14	3.86–6.13	3.87	2.83–5.83	5.24	2.73–8.26	0.270
	7D–30D	5.80 <sup>A</sup>	5.23–7.37	1.77 <sup>B</sup>	0.91–2.64	6.48 <sup>A</sup>	5.72–8.06	0.001*
Tea	Base line–7D	3.33 <sup>B</sup>	1.97–4.19	3.97 <sup>B</sup>	2.11–7.26	8.18 <sup>A</sup>	5.83–8.80	0.002*
	Base line –30D	9.52 <sup>B</sup>	7.00–10.54	5.06 <sup>B</sup>	4.13–7.74	24.44 <sup>A</sup>	21.13–26.37	< 0.001*
	7D–30D	10.18 <sup>B</sup>	8.56–11.23	3.13 <sup>C</sup>	1.90–8.71	16.49 <sup>A</sup>	14.99–18.42	< 0.001*
Coffee	Base line–7D	3.19	1.52–4.53	3.27	1.49–4.12	3.17	0.99–6.41	0.996
	Base line –30D	5.12 <sup>B</sup>	4.05–6.30	3.73 <sup>B</sup>	2.67–3.94	10.53 <sup>A</sup>	8.37–16.18	< 0.001*
	7D–30D	7.79 <sup>A</sup>	6.36–8.67	0.96 <sup>B</sup>	0.52–2.48	10.16 <sup>A</sup>	5.23–13.25	0.001*
Coke	Base line–7D	0.91 <sup>B</sup>	0.40–3.00	4.81 <sup>A</sup>	2.19–6.90	6.07 <sup>A</sup>	3.34–10.15	0.003*
	Base line –30D	7.46 <sup>A</sup>	6.78–9.05	4.94 <sup>B</sup>	3.67–5.50	8.67 <sup>A</sup>	6.91–13.19	0.001*
	7D–30D	6.65 <sup>A</sup>	4.72–8.35	2.21 <sup>B</sup>	0.76–3.33	4.84 <sup>A</sup>	3.80–11.92	0.001*

\*: Significant at  $P \leq 0.05$ , Different superscripts in the same row are statistically significantly different.

$\Delta E$  values of the CU and U subgroups when aged in coke from base line to 30 days and from 7 to 30 days; both showed the highest significant median  $\Delta E$  values. The CF subgroup showed the lowest significant median  $\Delta E$  values at these aging periods.

The effect of the **staining beverages** on the color changes ( $\Delta E$ ) of the tested materials at the different aging periods is presented in Table 3. Regarding to the CU subgroup, there was no statistically significant difference between the median  $\Delta E$  values when they were aged from base line to 7 days in the 4 staining beverages. However, aging from base line to 30 days in tea and coke were not significantly different and both produced significantly higher  $\Delta E$  values for CU subgroups than those recorded after aging in distilled water and coffee. From 7 to 30 days of aging; the specimens aged in tea showed the highest significant median  $\Delta E$  values while there were no significant differences between the other 3 beverages.

Surprisingly, there was no statistically significant difference between the median  $\Delta E$  values of the CF subgroups after aging in the 4 beverages. Concerning the behavior of the U subgroups, there was no statistically significant difference between the median  $\Delta E$  values when they were aged in tea and coke from base line to 7 days; both showed the highest significant median  $\Delta E$  values. Additionally, there was insignificant difference between the specimens aged in distilled water and coffee; both showed the lowest significant median  $\Delta E$  values. From base line to 30 days of aging in tea, the recorded  $\Delta E$  for the U subgroups were significantly the highest values while there was no statistically significant difference between those recorded when specimens were aged in coffee and coke; both showed lower significant median  $\Delta E$  values. Distilled water showed the statistically significantly lowest

median  $\Delta E$  values. From 7 days to 30 days; tea showed the highest statistically significant median  $\Delta E$  values followed by the coffee. Finally, there was no statistically significant difference between aging in distilled water and coke; both showed the lowest significant median  $\Delta E$  values.

The effect of **aging** on the color changes ( $\Delta E$ ) of the tested materials in the different staining beverages is presented in Table 4. Regarding to the CU subgroups, there was no statistically significant difference between the median  $\Delta E$  values from base line to 30 days and 7–30 days of aging in all beverages; both aging periods showed the highest statistically significant median  $\Delta E$  values. Concerning the behavior of the CF subgroups, after aging from base line to 7 days and from base line to 30 days in distilled water, coffee and coke; there were no statistically significant difference between the median  $\Delta E$  values; both aging periods showed the highest statistically significant median  $\Delta E$  values. However, the median  $\Delta E$  values of the CF subgroup did not differ significantly at the different aging periods in tea. Finally, the aging of the U subgroups from base line to 30 days and from 7 to 30 days in distilled water and coffee revealed insignificant difference between the median  $\Delta E$  values; both showed the highest significant median  $\Delta E$  values while the lowest significant median  $\Delta E$  values were found from base line to 30 days. On the other hand, aging in tea from base line to 30 days produced the highest significant median  $\Delta E$  values while the lowest significant median  $\Delta E$  was found from base line to 7 days. Aging in coke from base line to 30 days revealed the highest significant median  $\Delta E$  values. There was no statistically significant difference between the median  $\Delta E$  values from base line to 7 days and from 7 to 30 days; both aging periods showed the lowest significant median  $\Delta E$  values.

**Table 3**  
Effect of staining beverages on the median color change values ( $\Delta E$ ) of the tested materials.

Material	Time	Distilled water		Tea		Coffee		Coke		P-value
		Median	Range	Median	Range	Median	Range	Median	Range	
CU	Base line–7D	2.38	0.77–6.26	3.33	1.97–4.19	3.19	1.52–4.53	0.91	0.40–3.00	0.155
	Base line–30D	5.14 <sup>B</sup>	3.86–6.13	9.52 <sup>A</sup>	7.00–10.54	5.12 <sup>B</sup>	4.05–6.30	7.46 <sup>A</sup>	6.78–9.05	< 0.001*
	7D–30D	5.80 <sup>B</sup>	5.23–7.37	10.18 <sup>A</sup>	8.56–11.23	7.79 <sup>B</sup>	6.36–8.67	6.65 <sup>B</sup>	4.72–8.35	< 0.001*
CF	Base line–7D	3.51	2.60–3.87	3.97	2.11–7.26	3.27	1.49–4.12	4.81	2.19–6.90	0.283
	Base line–30D	3.87	2.83–5.83	5.06	4.13–7.74	3.73	2.67–3.94	4.94	3.67–5.50	0.055
	7D–30D	1.77	0.91–2.64	3.13	1.90–8.71	0.96	0.52–2.48	2.21	0.76–3.33	0.077
U	Base line–7D	3.13 <sup>B</sup>	1.78–5.70	8.18 <sup>A</sup>	5.83–8.80	3.17 <sup>B</sup>	0.99–6.41	6.07 <sup>A</sup>	3.34–10.15	0.002*
	Base line–30D	5.24 <sup>C</sup>	2.73–8.26	24.44 <sup>A</sup>	21.13–26.37	10.53 <sup>B</sup>	8.37–16.18	8.67 <sup>B</sup>	6.91–13.19	< 0.001*
	7D–30D	6.48 <sup>C</sup>	5.72–8.06	16.49 <sup>A</sup>	14.99–18.42	10.16 <sup>B</sup>	5.23–13.25	4.84 <sup>C</sup>	3.80–11.92	< 0.001*

\*: Significant at  $P \leq 0.05$ , Different superscripts in the same row are statistically significantly different.

**Table 4**  
Effect of aging periods on the median color change values ( $\Delta E$ ) for the tested materials.

Material	Storage medium	Base line–7D		Base line–30D		7D–30D		P-value
		Median	Range	Median	Range	Median	Range	
CU	Distilled water	2.38 <sup>B</sup>	0.77–6.26	5.14 <sup>A</sup>	3.86–6.13	5.80 <sup>A</sup>	5.23–7.37	0.028*
	Tea	3.33 <sup>B</sup>	1.97–4.19	9.52 <sup>A</sup>	7.00–10.54	10.18 <sup>A</sup>	8.56–11.23	0.002*
	Coffee	3.19 <sup>B</sup>	1.52–4.53	5.12 <sup>A</sup>	4.05–6.30	7.79 <sup>A</sup>	6.36–8.67	0.002*
	Coke	0.91 <sup>B</sup>	0.40–3.00	7.46 <sup>A</sup>	6.78–9.05	6.65 <sup>A</sup>	4.72–8.35	0.002*
CF	Distilled water	3.51 <sup>A</sup>	2.60–3.87	3.87 <sup>A</sup>	2.83–5.83	1.77 <sup>B</sup>	0.91–2.64	0.002*
	Tea	3.97	2.11–7.26	5.06	4.13–7.74	3.13	1.90–8.71	0.311
	Coffee	3.27 <sup>A</sup>	1.49–4.12	3.73 <sup>A</sup>	2.67–3.94	0.96 <sup>B</sup>	0.52–2.48	0.002*
	Coke	4.81 <sup>A</sup>	2.19–6.90	4.94 <sup>A</sup>	3.67–5.50	2.21 <sup>B</sup>	0.76–3.33	0.018*
U	Distilled water	3.13 <sup>B</sup>	1.78–5.70	5.24 <sup>A</sup>	2.73–8.26	6.48 <sup>A</sup>	5.72–8.06	0.021*
	Tea	8.18 <sup>C</sup>	5.83–8.80	24.44 <sup>A</sup>	21.13–26.37	16.49 <sup>B</sup>	14.99–18.42	0.001*
	Coffee	3.17 <sup>B</sup>	0.99–6.41	10.53 <sup>A</sup>	8.37–16.18	10.16 <sup>A</sup>	5.23–13.25	0.006*
	Coke	6.07 <sup>B</sup>	3.34–10.15	8.67 <sup>A</sup>	6.91–13.19	4.84 <sup>B</sup>	3.80–11.92	0.039*

\*: Significant at  $P \leq 0.05$ , Different superscripts in the same row are statistically significantly different.

### 3.2. Hardness (VH)

The effect of the **surface protection** on the VH values (MPa) of the tested groups is presented in Table 5. The results revealed that there were no significant differences between the VH values (MPa) of CU, CF and U subgroups at the base line (measured after storage in distilled water for 1 day) and after aging of the subgroups in distilled water and tea for 7 and 30 days. Moreover, there was insignificant difference between the CU and U subgroups after aging for 7 and 30 days in coffee and after aging in coke for 7 days; both exhibited significantly higher VH (MPa) values than those of the CF subgroups. Finally, the median VH (24.4 MPa) value of the U and CF (29.0 MPa) subgroups were not significantly different after aging in coke for 30 days while both were significantly lower than the median VH (36.4 MPa) values of the CU subgroup.

The effect of the **staining beverages** on the VH values (MPa) of the tested subgroups is presented in Table 6. Aging in the four beverages did not significantly affect the median VH values (MPa) of all tested subgroups (CU, U and CF) except when the U subgroup was aged for 30 days. Aging of U subgroup for 30 days in distilled water recorded the highest significant median VH value (54.0 MPa) followed by its aging in coffee (41.2 MPa). However, aging of the U subgroup in coke for 30 days revealed the lowest significant median VH value (24.4 MPa).

The effect of the **aging** (7 and 30 days) on the median micro-hardness of the tested materials is presented in Table 7. The results

showed that aging of the CU subgroups in all staining beverages and the CF subgroups in distilled water and tea has no statistically significant effect on their median VH values (MPa). Meanwhile, aging of CF subgroup in coffee and coke for 7 and 30 days significantly lowered their median VH values (MPa) than their corresponding VH values at base line. However, the VH values (MPa) of the CF subgroup after aging for 7 days were not significantly different than those after 30 days when they were aged in coffee and coke. Regarding to the U subgroups, aging in distilled water and coffee for 7 and 30 days did not significantly affect their median VH values (MPa). On the other hand, aging of the U subgroup for 30 days in either tea or coke recorded lower significant VH values (MPa) than those recorded at base line and after aging for 7 days which were not significantly different from each other.

### 3.3. Correlation between micro-hardness and color change

There was no statistically significant correlation between color change ( $\Delta E$ ) and the hardness of all tested GI restorative materials under all tested conditions.

### 3.4. Chemical analysis and imaging

The EDX analysis pattern with the elemental percentages (Wt. % and At. %) of the representative glass powders of each tested material is presented in Fig. 1-A and B. According to the site of measurements,

**Table 5**  
Effect of protective coating on the median Vickers hardness (VH) values (MPa) of the tested materials.

Staining beverages	Time	CU		CF		U		P-value
		Median	Range	Median	Range	Median	Range	
Distilled water	Base line	36.7	23.7–70.5	37.4	32.8–54.7	43.0	35.4–56.1	0.508
	7 days	36.9	25.2–71.4	33.7	21.4–47.0	47.3	34.3–60.1	0.058
	30 days	33.9	25.7–64.5	30.3	26.3–62.3	54.0	35.2–60.4	0.114
Tea	Base line	45.8	28.3–66.0	44.3	24.8–79.8	49.6	30.5–55.5	0.992
	7 days	41.7	23.4–64.7	38.6	26.6–51.5	43.6	33.3–51.4	0.739
	30 days	39.1	22.6–65.1	28.6	23.0–43.7	30.1	22.5–35.0	0.324
Coffee	Base line	38.3	26.5–62.9	40.9	28.5–59.7	42.0	31.5–65.6	0.745
	7 days	35.7 <sup>A</sup>	27.3–88.8	27.1 <sup>B</sup>	21.2–37.7	46.1 <sup>A</sup>	25.5–61.8	0.010*
	30 days	44.1 <sup>A</sup>	19.4–65.4	28.5 <sup>B</sup>	23.7–36.7	41.2 <sup>A</sup>	27.9–45.2	0.048*
Coke	Base line	44.9	28.0–63.4	50.8	44.7–55.2	38.9	34.9–49.5	0.052
	7 days	33.9 <sup>A</sup>	26.0–66.9	30.4 <sup>B</sup>	24.4–50.3	42.7 <sup>A</sup>	38.2–60.4	0.040*
	30 days	36.4 <sup>A</sup>	26.6–49.5	29.0 <sup>B</sup>	17.3–39.7	24.4 <sup>B</sup>	22.0–29.7	0.009*

\*Significant at  $P \leq 0.05$ , Different superscripts in the same row are statistically significantly different.

**Table 6**  
Effect of staining beverages on the median Vickers hardness (VH) values (MPa) of the tested materials.

Material	Time	Distilled water		Tea		Coffee		Coke		P-value
		Median	Range	Median	Range	Median	Range	Median	Range	
CU	Base line	36.7	23.7–70.5	45.8	28.3–66.0	38.3	26.5–62.9	44.9	28.0–63.4	0.970
	7 days	36.9	25.2–71.4	41.7	23.4–64.7	35.7	27.3–88.8	33.9	26.0–66.9	0.825
	30 days	33.9	25.7–64.5	39.1	22.6–65.1	44.1	19.4–65.4	36.4	26.6–49.5	0.898
CF	Base line	37.4	32.8–54.7	44.3	24.8–79.8	40.9	28.5–59.7	50.8	44.7–55.2	0.495
	7 days	33.7	21.4–47.0	38.6	26.6–51.5	27.1	21.2–37.7	30.4	24.4–50.3	0.137
	30 days	30.3	26.3–62.3	28.6	23.0–43.7	28.5	23.7–36.7	29.0	17.3–39.7	0.641
U	Base line	43.0	35.4–56.1	49.6	30.5–55.5	42.0	31.5–65.6	38.9	34.9–49.5	0.359
	7 days	47.3	34.3–60.1	43.6	33.3–51.4	46.1	25.5–61.8	42.7	38.2–60.4	0.626
	30 days	54.0 <sup>A</sup>	35.2–60.4	30.1 <sup>C</sup>	22.5–35.0	41.2 <sup>B</sup>	27.9–45.2	24.4 <sup>D</sup>	22.0–29.7	< 0.001*

\*: Significant at P ≤ 0.05, Different superscripts in the same row are statistically significantly different.

higher amount of Al, F and Sr with lower amount of Si were found in ketac Fil Plus Aplicap than in Ketac Universal Aplicap. The SE micrographs of Ketac Universal Aplicap glass powder showed smaller averages particles sizes (7.2 μm) as compared to the 17.9 μm average filler size presented in the SE micrograph of ketac Fil Plus Aplicap powder (Fig. 2-A and B respectively).

**4. Discussion**

The constant evolution of restorative materials and techniques has been always targeted toward achieving an optimal combination of adequate mechanical properties and satisfactory esthetics [17]. Hardness is a surface mechanical property that could provide an indication of wear resistance and durability in the oral environment [18] On the other hand, color is an esthetic physical property that always grasping patient's attention. Clinically, GIC is widely used as an easy, quick restorative solution for many clinical situations including class V lesions (site frequently subjected to routine tooth brushing abrasion). Knowing that restoratives discoloration is usually related to surface adsorption and absorption of the colorants [12,13,19–21], surface durability could be an important material quality that affects the color stability of esthetic restorations. Accordingly, the color stability and hardness were evaluated for the recently introduced uncoated GI restorative material with and without protective coating as compared with conventional GI that was used with the same resin coating as recommended by the manufacturer; after being exposed to different staining beverages commonly used by wide range of patients (tea, coffee, coke and distilled water) for different aging periods (7 and 30 days). Furthermore, the

correlation between color changes and hardness of the investigated materials were evaluated under all tested conditions.

The null hypothesis of the current study was rejected as significant differences were found between the evaluated material's subgroups under certain testing conditions regarding the color changes and the hardness values.

In an attempt to explain the obtained color stability and hardness results, EDX chemical analysis of the glass powder of the two GI tested materials was performed (Fig. 1A and B). It has been observed that the ketac Fil Plus Aplicap possessed higher Sr and Al: Si ratio content than those of Ketac Universal Aplicap. There are two main factors that control the kinetics of the setting reaction; the extraction rate of ions from the glass, which is controlled by the composition of the glass and liquid, and the binding of cations (e.g. Sr<sup>2+</sup>) to the polyanion chain. The number and type of anions and cations released from the glass particles will determine the extent of crosslinking of the polysalt matrix and the cement properties [22]. It has been found that strontium (Sr) presented delayed onset of setting reaction. This is most likely due to the slow Sr<sup>2+</sup> ion binding to the polyacid chains and thus could delay the maturation of ketac Fil Plus Aplicap as compared with the recently introduced Ketac Universal Aplicap GI restorative materials. This view is in agreement with earlier investigations [23–25]. Additionally, the rate of maturation of GI restoratives can affect its mechanical and physical behavior in the different staining beverages. Presence of benzoic acid (< 0.2 wt %) in the co-polymeric acid of ketac Universal Aplicap as stated by the manufacture (Table 1) could produce a mechanical interlocking effect upon hardening. Benzoic acid containing cements have been reported to have proper chemical resistance in the

**Table 7**  
Effect of aging periods on the median Vickers hardness (VH) values (MPa) of the tested materials.

Material	Staining beverages	Base line		7 days		30 days		P-value
		Median	Range	Median	Range	Median	Range	
CU	Distilled water	36.7	23.7–70.5	36.9	25.2–71.4	33.9	25.7–64.5	0.686
	Tea	45.8	28.3–66.0	41.7	23.4–64.7	39.1	22.6–65.1	0.724
	Coffee	38.3	26.5–62.9	35.7	27.3–88.8	44.1	19.4–65.4	0.990
	Coke	44.9	28.0–63.4	33.9	26.0–66.9	36.4	26.6–49.5	0.386
CF	Distilled water	37.4	32.8–54.7	33.7	21.4–47.0	30.3	26.3–62.3	0.205
	Tea	44.3	24.8–79.8	38.6	26.6–51.5	28.6	23.0–43.7	0.183
	Coffee	40.9 <sup>A</sup>	28.5–59.7	27.1 <sup>B</sup>	21.2–37.7	28.5 <sup>B</sup>	23.7–36.7	0.015
	Coke	50.8 <sup>A</sup>	44.7–55.2	30.4 <sup>B</sup>	24.4–50.3	29.0 <sup>B</sup>	17.3–39.7	0.001*
U	Distilled water	43.0	35.4–56.1	47.3	34.3–60.1	54.0	35.2–60.4	0.223
	Tea	49.6 <sup>A</sup>	30.5–55.5	43.6 <sup>A</sup>	33.3–51.4	30.1 <sup>B</sup>	22.5–35.0	0.003*
	Coffee	42.0	31.5–65.6	46.1	25.5–61.8	41.2	27.9–45.2	0.144
	Coke	38.9 <sup>A</sup>	34.9–49.5	42.7 <sup>A</sup>	38.2–60.4	24.4 <sup>B</sup>	22.0–29.7	< 0.001*

\*: Significant at P ≤ 0.05, Different superscripts in the same row are statistically significantly different.

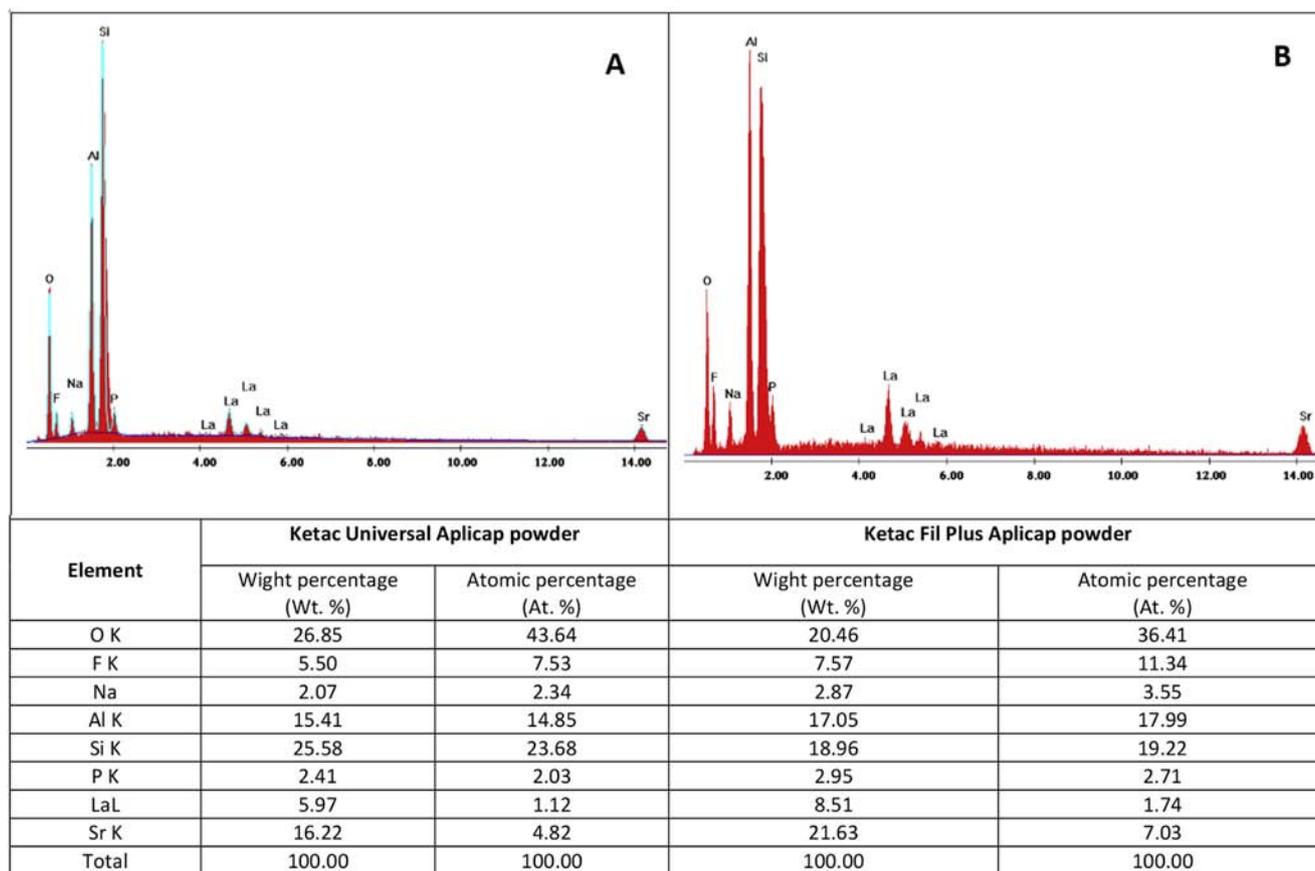


Fig. 1. Energy dispersive X-ray analyses (EDX) pattern with elemental percentages (%) of Ketac Universal Aplicap (A) and ketac Fil Plus Aplicap glass powders (B).

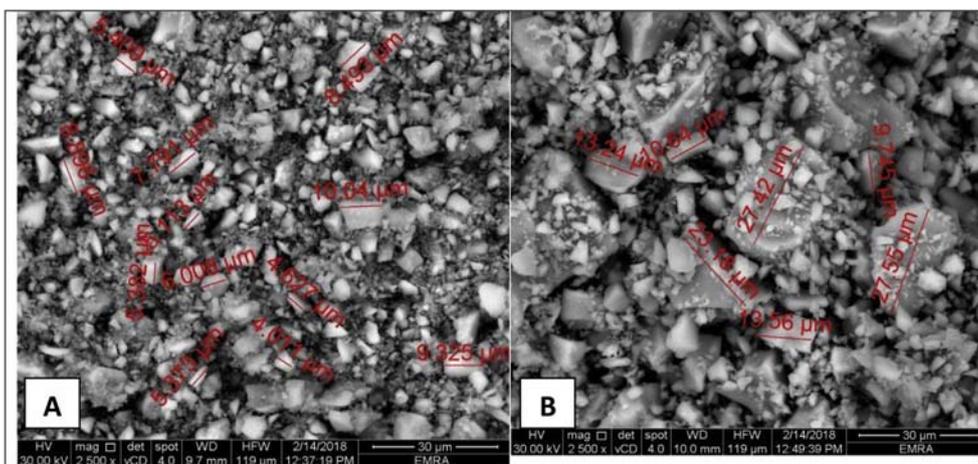


Fig. 2. SE micrograph of the glass power of the tested GI materials: Ketac Universal Aplicap powder revealing a smaller average fillers size of 7.2 μm (A) than the Ketac Fil Plus Aplicap powder of 17.9 μm (B).

oral environment [26]. The ratio of Al: Si ratio in the glass is one of the critical factors that control the behavior of the glass fillers towards the aqueous media to which they are exposed to. It must exceed 1.2:1 by mass to impart the proper reactivity and basicity of the glass fillers [27–29]. It worth mentioning that the majority of silicate glasses are resistant to acid attacks owing to the strong covalent characteristics of Si-O-Si-bonds; though, the glass becomes more sensitive to acid attacks with an increase in the ionic properties of silicate. Silicates, which are attacked by acid, include Al: Si ratio which is sufficiently high (i.e. higher content of the more basic Si-O-Al bonds) [30]. Therefore, any factor that decreases the hydrolytic stability of the GI restorative

material would decrease its hardness and color stability. Therefore, they could enhance their degradation in oral fluids. This has been reflected on the color stability and hardness results of the current study to great extent.

#### 4.1. Color stability

Color of conventional GI restorative materials in the oral environment is very challenging owing to its inherent chemical composition, slow setting reaction and smart behavior under various conditions. Generally, the differences in color stability among restorative materials

can be described in part to the size of the colorant particle and the constituents of the restorative material (water and monomer). In this study CIELAB (Standardized Commission International De Eclairage) color system was used, as recommended by the American Dental Association [9]. Regarding to the obtained color changes values ( $\Delta E$ ) presented in Tables 2–4, all the tested groups under the various testing conditions exceeded the 50:50% acceptability threshold (AT) except for the CU subgroups when aged in distilled water (2.38) and in coke (0.91) for 7 days. Therefore, although there was statistically insignificant differences in  $\Delta E$  values between the CU and U subgroups after their storage in water during all aging periods, in coffee from baseline to 7 days and from 7 to 30 days and in coke from base line to 30 days as well as from 7 to 30 days (Table 2), there is clinical significance that necessitates the application of surface protective coating on the newly introduced GI restorative material to help in the maintenance of its color stability up to 2.8 years clinically. This is based on the following assumption; where 2 cups of tea or coffee or coke are consumed every day by the patients and the beverages remain in contact with the restored teeth for 5 min/cup (10 min/day), then 7 days of specimen's immersion would be relatively equivalent to the cumulative effect of the restoration in clinical service for 2.8 years. Furthermore, the survival of conventional GI restorations up to 2.8 years would be of adequate clinical success rate as they are classified as semi-permanent restorative materials owing to their smart behavior and continuous solubility especially under acidic conditions. Though, a total period of 30 days for specimen's aging was used in the current study because it is typically the period used in most in vitro color stability studies to achieve accumulative staining effect and to obtain distinctive results [31]. However, in the present study statistically significant color changes ( $\Delta E$ ) occurred during the first week of specimen's immersion.

According to the EDX chemical analysis results obtained for the glass powders of the tested materials (Fig. 1A and B), the hydrolytic stability of ketac Fil Plus Aplicap glass powder would be lower than that of Ketac Universal Aplicap. This could explain the higher color changes of the CF subgroups when aged in coke from base line to 7 days as compared to the CU subgroup although, that both subgroups were coated by the same resin coating (ketac™ Glaze), (Table 2). The acidic pH of the coke (2.7) together with the higher Al: Si ratio and fluoride content would increase filler erosion and subsequent surface roughness that facilitate staining adsorption. Consequently, increases color changes when compared to Ketac Univesal Aplicap.

Concerned with the effect of the staining beverages on the color stability, it is generally obvious that tea and coke are the most staining beverages for CU and U subgroups as compared with coffee and water (Table 3). This could be related to the existence of flavonoids (tannic acid) in tea and citric acid in coke. The flavonoids are one of the most important groups of polyphenolic compounds present in tea. Within the flavonoid group, flavanols are the most prevalent. Flavanols are also referred to as tannins, and during oxidation are converted to the aflavins and thearubigins—the compounds responsible for the dark color and robust flavors notably present in black teas [32]. However, the lack of yellow colorant in coke may be the reason why it did not produce as much discoloration as compared to tea especially in the U subgroups when aged from base line to 30 days and from 7 to 30 days (Table 3). Furthermore, the presence of carbonated water could produce erosive effect on the specimen's surface followed by adsorption of the caramel pigment present in the coke.

The effect of the aging on the color stability of the tested materials is presented in Table 4. Generally, aging of the CU subgroups for 7 days in all beverages revealed statistically lower color changes than those after aging from base line to 30 days and from 7 to 30 days. On the other hand, the CF subgroup usually recorded earlier statistically higher significant color changes (during the first week) in all beverages except for tea where there were insignificant differences among all aging periods. This could be related to the lower Sr content in the glass powder and addition of benzoic acid to the liquid of Ketac Universal

Aplicap (Table 1, Fig. 2A and B) that could be reflected on the maturation and interlocking rates of the setting restorative material as explained earlier [23–25]. Additionally, the larger average (17.9  $\mu\text{m}$ ) filler size of Ketac Fil Plus Aplicap as compared with those of Ketac Universal Aplicap (7.2  $\mu\text{m}$ ) could increase its early surface roughness after aging in the staining beverages (after 7 days) especially in the more acidic beverages (coke of pH of 2.7 and coffee of pH of 4) as seen in the SE micrographs (Fig. 2A and B). It seems that the presence of surface coating did not alter the aging behavior of the U subgroups as compared to the CU subgroups in all beverages except coke; revealing the effectiveness of surface protective coating on the newly introduced GI restorative material in highly acidic media.

#### 4.2. Hardness

The effect of the protective coating, staining beverages and aging on the microhardness values of the investigated materials are presented in Tables 5–7 respectively. It has been found that the CU and U subgroups revealed statistically nonsignificant differences after aging in all staining beverages at all aging periods except when the specimens were aged in coke for 30 days (Table 5). The CU subgroup exhibited significantly higher VH value than the corresponding U subgroup and the CF subgroup where the latter were not significantly different from the U subgroup. This could be explained in the light of the modified chemical composition (Table 1, Fig. 1 A, B) and smaller filler size (Fig. 2 A, B) of the newly introduced GI (Ketac Universal Aplicap) as discussed earlier. The obtained hardness results are supported by a previous study reported that premature hydration of GI did not negatively influence the strength of GI restoratives and recommended an opposing advice to the instructions issued by most manufacturers [33]. However, placement of a protective coating over the recently introduced GI restorative material could be beneficial for long term clinical serve especially in acidic medium with abrasive action of tooth brushing occurred in class V restorations. This is in agreement with earlier studies [34–36].

The lower significant hardness values of the CF subgroup as compared with those of the CU and U subgroups when aged in coffee for 7 and 30 days and in coke for 7 days could be related to the fact that the initial setting reaction of GI cements usually takes place in the first 3–4 min; afterwards a slower reaction takes place (maturation) that is associated with various changes in the physical properties, strength and translucency of the materials [37]. This means that inherent characteristics of the material were expressed after the full maturation was reached and not at the baseline where the protective coating is still intact. These findings are in accordance with an earlier study revealed that hardness of different GICs is definitely influenced by compositional variations [11].

Considering the effect of the staining beverages exclusively on the hardness values of the tested materials (Table 6), the U subgroup maintained comparable median hardness values to those of the CU and CF subgroups up to 30 days of aging in all staining beverages except when they were aged in coke where their hardness values were significantly decreased (24.4 MPa); almost to half its value when aged in distilled water for the same aging period (54.0 MPa). This could be referred to the effect of the highly acidic nature (pH = 2.7) of coke that tends to enhance the degradation of the matrix with surface erosion of the fillers [38], hence, dramatically decreases its hardness.

Surprisingly, the effect of aging exclusively on the hardness values of the CU subgroups in all staining beverages was not statistically significant (Table 7). On the other hand, aging for 7 and 30 days in tea and coke significantly decreased its hardness values than its corresponding base line values. This could be attributed to the presence of phosphoric and carbonic acids in coke (pH = 2.7) and the presence of tannic acid in tea that could produce softening effect on the unprotected surfaces in spite of its higher pH value (4.9) [38,39]. Additionally, aging of CF subgroups for 7 and 30 days in coffee and coke decreased significantly their hardness values than its corresponding base line values. This could

**Table 8**

Spearman's correlation coefficient for the correlation between the color change ( $\Delta E$ ) and hardness.

Time	Correlation coefficient	P-value
Base line–1w	0.318	0.152
Base line–4w	0.126	0.475
1w–4w	0.169	0.405

\*: Significant at  $P \leq 0.05$ .

be explained on the basis of the acidic nature of the staining beverage (coke pH = 2.7 and coffee pH = 4) that could attack the matrix and the basic fillers [27–29].

Finally, the absence of statistically significant correlation between the color changes ( $\Delta E$ ) and the surface hardness of all tested GI restorative materials whether coated or not (Table 8), indicated that surface hardness is not the main factor that guarantee the color stability of the GI restorative materials. It worth mentioning that the current study has certain limitations; namely, it does not simulate the role of saliva or oral clearances in retarding the long-term build-up of stains in the oral environment. Therefore, further studies considering the diluting effect of saliva on the staining beverage and oral cleaners in correlation to surface roughness and color stability of the newly introduced uncoated GI restorative material are recommended.

## 5. Conclusions

Surface protection is not the only factor that could maintain the color stability and hardness of GI restoratives; the chemical composition and glass filler size could be of great value. Surface protective coating of the recently introduced GI restorative materials exhibited acceptable color changes only in coke after aging for 7 days (~3 years clinically). Surface protective coating of the newly introduced uncoated GI restorative seems effectiveness in maintaining its hardness in coke up to 3 year clinically. Hardness behavior of all coated and uncoated GI restorative materials was not correlated to their color stability. It is advisable to use the recently introduced uncoated GI restorative material for patients who are not consuming tea and/or coffee but with surface protection to maintain its color acceptability up to 3 year clinically.

## Conflicts of interest

The authors declare no conflicts of interest. The authors alone are responsible for the content and writing of the paper.

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