

## COLOR STABILITY AND MICROHARDNESS OF DIFFERENT GLASS IONOMER CEMENTS

### FARKLI CAM İYONOMER SİMANLARIN RENK STABİLİTELERİ VE MİKROSERTLİKLERİ

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#### Öz

#### Amaç

Cam iyonomer simanlar sahip oldukları olumlu özelliklerden dolayı diş hekimliğinde birçok farklı tedavi prosedüründe tercih edilmektedir. Bu çalışmanın amacı, farklı cam iyonomerlerin, farklı solüsyonlarda bekletildikten sonraki renk farkı ve mikrosertlik sonuçlarını değerlendirmektir.

#### Gereç ve Yöntem

Equia Forte, AHfil Silver ve Ionofil cam iyonomerlerin her birinden 40 tane olmak üzere toplam 120 örnek hazırlandı. Her cam iyonomere ait örnekler distile su, kola, çay ve kahve solüsyonlarında bekletildi. Solüsyonlara daldırılmadan önce ve bir hafta sonra örneklerin renk ve mikrosertlik ölçümleri yapıldı. Örneklerin renk ölçümleri bir spektrofotometre ile, mikrosertlik değerleri ise Vickers mikrosertlik cihazı ile değerlendirildi. Renk analizi için, solüsyonlara daldırılmadan önce ve bir hafta sonra örneklerin L\*, a\* ve b\* değerleri elde edilerek renk değişimleri ( $\Delta E$ ) hesaplandı. Elde edilen veriler varyans analizi (ANOVA) ve Tukey's testi ile değerlendirildi.

#### Bulgular

AHfil Silver için kolada bekleyen örnekler dışında, tüm materyaller için tüm sıvılarda bekletilen örneklerde

mikrosertlik değerlerinde bir artış gözlemlendi. En yüksek renk değişimi Equia Forte için kahvede, AHfil Silver ve Ionofil için kolada bekletilen örneklerde elde edildi ( $p<0.05$ ). En düşük renk değişimi distile suda bekletilen örneklerde elde edildi ve bu değerler Equia forte ve Ionofil için, kabul edilebilir renk değişimi eşik değerinin altındaydı.

#### Sonuç

Test edilen tüm materyaller, test ortamına daldırıldıktan sonra mikrosertlik ve renk değişim değerlerinde bir farklılık göstermiştir. Mikrosertlik değerindeki artış cam iyonomer simanların zamanla artan maturasyonuna, renk değişimi ise solüsyonların içinde bulunan sarı boyar madde miktarına atfedilebilir.

**Anahtar Kelimeler:** Cam iyonomer siman, Mikrosertlik, Renk stabilitesi,

#### Abstract

#### Objective

Glass ionomers are preferred in many different treatment procedures in dentistry due to their positive features. The aim of this study is to evaluate the color difference and microhardness values of various glass ionomer cement after storing them in different solutions.

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## Material and Method

A total of 120 samples were prepared, including 40 of each of the Equia Forte, AHfil Silver, and Ionofil glass ionomers. Each glass ionomer sample was stored in distilled water, cola, tea, and coffee solutions. Color and microhardness measurements of the specimens were performed before and after a week before it was immersed in solutions. The color measurements of the samples were evaluated with a spectrophotometer and microhardness values were evaluated with Vickers microhardness device. For color analysis, color changes ( $\Delta E$ ) were calculated by obtaining the values of  $L^*$ ,  $a^*$ , and  $b^*$  of the samples before and after a week after being immersed in solutions. The data obtained were evaluated by variance analysis (ANOVA) and Tukey's test.

## Results

Except for specimens stored in cola for AHfil Silver, microhardness values were increased for all materials

immersed in test solutions. The highest color change was obtained in coffee for Equia Forte, in the samples immersed in cola for AHfil Silver and Ionofil ( $p < 0.05$ ). The lowest color change was detected in samples stored in distilled water, and for Equia Forte and Ionofil, the color change was below the acceptable threshold value.

## Conclusion

All the materials tested differ in microhardness and color change values after being immersed in the test environment. The increase in microhardness value can be attributed to the increasing maturation of glass ionomer cement over time, and color change can be attributed to the amount of yellow dyestuff contained in the solutions.

**Keywords:** Color stability, Glass ionomer cement, Microhardness

## Introduction

Glass ionomer cement, developed and introduced in the early 1970s, has anti-cariogenic properties by releasing fluoride. Additionally, it is chemically bonded to the tooth hard tissues, bioactive, clinically simple to use, and has a thermal expansion coefficient comparable to dentin (1, 2). The mentioned favorable properties have provided glass ionomers a wide range of uses in dentistry as a liner, base, orthodontic bracket adhesive, luting cement, fissure sealant, and primary tooth restoration (3). While conventional glass ionomer cement has many beneficial properties, it also has poor mechanical properties, such as low tensile strength, high surface roughness, low brittleness, and poor fracture toughness (4-6). However, the mechanical properties and clinical survival of restorations are negatively affected by conventional glass ionomers' prolonged setting reaction time (4).

Despite these limitations, clinically significant features such as fluoride release, use of bulk-fill, and no additional bonding procedure during application have led to the development of new glass-ionomer-based materials with robust protective properties (7, 8). Recently, high-viscosity glass ionomer cements with a higher powder:liquid ratio have been developed compared to previous glass ionomers (9). Introduced for atraumatic restorative treatment under the auspices of the World Health Organization, this product has acrylic acid molecules with a higher number of silicates and high molecular weight, which increases

the number of matrix crosslinks and provides greater flexural strength (5, 9). High-viscosity glass ionomers present better physicommechanical characteristics and faster setting times than self-curing materials due to their formulations (10-12). Furthermore, a review of clinical investigations using this cement revealed that the failure rates of posterior restorations performed with high-viscosity glass ionomer cement were comparable to the rates of dental amalgam restorations (13).

After application, the restorations undergo several physical and mechanical changes in the oral environment, including wear and discoloration (14). In particular, prolonged and frequent exposure of dental hard tissues and restorative materials with low pH beverages results in erosive wear on their surfaces, adversely affecting restoration's success (2, 15-18). Hardness is intimately related to the restorative material's proportionate limit, durability, and capacity to abrade opposing teeth or materials (19). Examining the surface hardness of tooth-colored materials provides valuable information about the durability of these materials (5). Considering the increasing use of newly developed glass ionomer cement and the consumption of acidic beverages, especially among young people, it is observed that there is a need for studies investigating the effects of these beverages on the surface properties of glass ionomer cement (4). The aim of this study was to evaluate the effect of frequently consumed beverages on the microhardness and color change of a high-viscosity

glass ionomer by comparing it with a silver-reinforced glass ionomer and a conventional glass ionomer cement. The null hypothesis of the study, there would be no difference between the three different glass ionomer cement used in the study in terms of surface hardness and color change values before and after storage in solutions.

## Material and Method

A high-viscosity (Equia Forte, GC, Tokyo), silver-reinforced (AHfil Silver, AHL, UK), and conventional (Ionofil, Voco, Germany) glass ionomer cement was used in this study. The design of the study is illustrated in Figure 1.

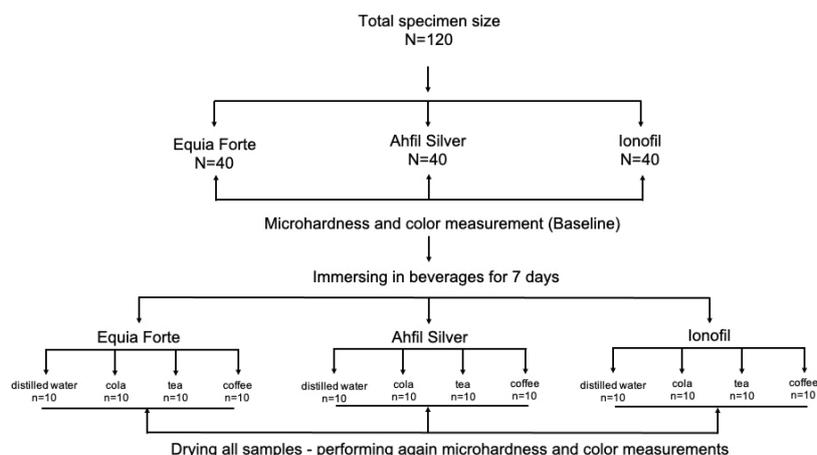
A total of 120 samples were prepared, 40 for each different glass ionomer. Sectional Teflon cylindrical molds with 6mm diameter and 2mm height were used to prepare the specimens. A Mylar strip was placed on the glass plate and a Teflon mold was placed on the top. According to the manufacturer's instructions, the test products were mixed and carefully placed into Teflon molds without creating any air bubbles. A Mylar strip and a glass plate were placed on top of the cement with slight pressure to overflow the excess material and to obtain a smooth surface. The samples were removed from the mold after waiting for the period each company claimed the setting time had been completed. The prepared samples were stored in distilled water at 37°C for one day.

Forty samples prepared for each glass ionomer cement tested were randomly divided into 4 subgroups (n=10) to be immersed in distilled water, cola, tea, and coffee for 7 days. The determination of microhardness values

and color measurements were performed before the samples were immersed into the beverages. Microhardness measurements were carried out using a microhardness device (TTS Matsuzawa HWMMT-X3, Tokyo, Japan) with the application of a 50 g load for 10 seconds. The mean of three measurements taken from each sample was accepted as the microhardness value (V<sub>1</sub>) of that sample. Only one operator carried out the microhardness analyses. A clinical spectrophotometer (Spectroshade Micro, MHT, Italy) was used for color measurements. Before color measurement, the samples were washed with distilled water for 10 s and dried with mild air pressure for 5 s. Three measurements were obtained from each specimen by only one operator. The color measurement of each sample was determined by calculating the mean of these three measurements. Color measurements taken from the samples were recorded as L\*, a\* and b\* values (L<sub>1</sub>, a<sub>1</sub>, b<sub>1</sub>).

The samples, for which the initial microhardness and color measurements were made, were immersed in the distilled water, cola, tea, and coffee solutions and kept in an incubator at 37°C for 7 days. Tea was prepared by putting a 2X2 prefabricated tea bag in 300 ml of boiled water for 10 minutes. Coffee was prepared by diffusing 3.6 g of coffee powder into 300 ml of boiled water. The solutions were changed daily to inhibit bacterial growth. The samples were dried at the end of the 7-day periods, and microhardness (V<sub>2</sub>) and color measurements (L<sub>2</sub>, a<sub>2</sub>, b<sub>2</sub>) were again performed. Color change (ΔE) was calculated using the following formulation.

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



**Figure 1**  
Design of the study

Statistical analyses were performed with the SPSS Program, version 29.0 (Statistical Package for the Social Sciences; SPSS, Chicago, USA). The Kolmogorov-Smirnov test was applied to verify if the data were normally distributed, and the data were found to have a normal distribution. The data were also statistically homogenous based on the Levene test. The data were analyzed using a two-way analysis of variance (ANOVA) followed by post-hoc Tukey's tests to compare the means between groups. A t-test analyzed the differences in microhardness values of the materials, evaluating the aging. The p-value less than 0.05 was considered statistically significant for all statistical analyses.

### Results

The ΔE values obtained from the specimens stored in different solutions for one week are shown in Table 1. When the ΔE of the glass ionomer cements tested in the study were analyzed by Tukey HSD multiplex comparison, only the difference between AHfil Silver and Ionofil was significant.

The highest and lowest ΔE values for Equia Forte were obtained from samples stored in coffee and distilled water, respectively. The ΔE values of the Equia Forte samples stored in distilled water were statistically lower than the ΔE values of the specimens stored in tea and coffee (p<0.05). Furthermore, the ΔE values of the samples stored in the coffee differed from those in tea and cola.

The maximum color change (ΔE) in Ahfil Silver samples was obtained in samples kept in cola. While the color change values that occurred when Ahfil Silver samples were stored in distilled water, cola, and tea were different from each other, the ΔE values of the specimens stored in coffee differed only with cola samples.

The highest data on color change values obtained from Ionofil samples belonged to cola. The delta values of each of the Ionofil samples stored in the solutions evaluated in the study were statistically different from each other (p<0.05).

When the initial microhardness values (MH<sub>1</sub>) of the glass ionomer cements tested in the study were analyzed with the Tukey HSD multiple comparison test, no statistical difference was found between Equia Forte and Ionofil (p>0.05). When the microhardness values (MH<sub>2</sub>) obtained after the samples were stored in solutions for one week were compared, no difference was found between Equia Forte and Ionofil again (p>0.05). The microhardness values obtained in the initial state and after storage for one week of the samples are summarized in Table 2.

When the MH<sub>1</sub> and MH<sub>2</sub> values for Equia Forte were compared, the microhardness values of the samples stored in distilled water and tea increased, and this difference was found to be significant (p<0.05). No significant difference was found when MH<sub>2</sub> values

**Table 1** The mean values and standard deviations of the color change (ΔE) of the glass ionomer cements stored in staining solutions.

Materials	Solutions	DE ± SD	p
Equia Forte	Distilled water	2.08 ± 0.91 <sup>a</sup>	0.000
	Cola	4.65 ± 1.29 <sup>ab</sup>	
	Tea	7.25 ± 2.84 <sup>b</sup>	
	Coffee	10.77 ± 2.98 <sup>c</sup>	
AHfil Silver	Distilled water	3.57 ± 1.97 <sup>a</sup>	0.000
	Cola	11.46 ± 3.04 <sup>b</sup>	
	Tea	6.45 ± 1.61 <sup>c</sup>	
	Coffee	5.89 ± 1.63 <sup>ac</sup>	
Ionofil	Distilled water	0.66 ± 0.40 <sup>a</sup>	0.000
	Cola	9.53 ± 1.50 <sup>b</sup>	
	Tea	3.89 ± 1.29 <sup>c</sup>	
	Coffee	7.02 ± 1.16 <sup>d</sup>	

Table 2

The means and standard deviations of glass ionomers' microhardness (MH) values initially (MH<sub>1</sub>), and after storing different staining solutions (MH<sub>2</sub>)

Materials	Solutions	MH <sub>1</sub> ± SD	p <sub>1</sub>	MH <sub>2</sub> ± SD	p <sub>2</sub>
Equia Forte	Distilled water	31.14 ± 7.59 <sup>aA</sup>	0.993	41.03 ± 5.76 <sup>aB</sup>	0.778
	Cola	30.16 ± 9.93 <sup>aA</sup>		37.27 ± 18.32 <sup>aA</sup>	
	Tea	31.25 ± 7.36 <sup>aA</sup>		36.75 ± 6.09 <sup>aB</sup>	
	Coffee	30.75 ± 8.65 <sup>aA</sup>		37.10 ± 5.83 <sup>aA</sup>	
AHfil Silver	Distilled water	20.36 ± 2.70 <sup>aA</sup>	0.991	21.60 ± 2.04 <sup>aA</sup>	0.002
	Cola	20.34 ± 3.18 <sup>aA</sup>		19.53 ± 2.49 <sup>aA</sup>	
	Tea	20.42 ± 2.84 <sup>aA</sup>		25.85 ± 5.45 <sup>bB</sup>	
	Coffee	20.67 ± 2.76 <sup>aA</sup>		21.83 ± 2.33 <sup>abA</sup>	
Ionofil	Distilled water	33.77 ± 4.23 <sup>aA</sup>	0.999	39.14 ± 2.08 <sup>aB</sup>	0.019
	Cola	33.71 ± 4.98 <sup>aA</sup>		33.46 ± 5.05 <sup>bA</sup>	
	Tea	33.63 ± 4.53 <sup>aA</sup>		38.91 ± 2.33 <sup>aB</sup>	
	Coffee	33.92 ± 4.31 <sup>aA</sup>		37.76 ± 6.26 <sup>abA</sup>	

\*Lowercase letters show differences between rows. Uppercase letters show differences between columns.

were compared for Equia Fort ( $p > 0.05$ ). When the MH<sub>1</sub> and MH<sub>2</sub> values for AHfil Silver were compared, there was a significant change in the microhardness values of only the samples stored in tea. When the MH<sub>2</sub> values were compared for the same glass ionomer cement, the microhardness values of tea were different with cola and distilled water.

When the MH<sub>1</sub> and MH<sub>2</sub> values for Ionofil were evaluated, it was determined that there was a significant change in the microhardness values of the samples stored in distilled water and tea. When the MH<sub>2</sub> values were analyzed for Ionofil in itself, the values obtained from cola were different from those obtained from tea and distilled water.

## Discussion

For the materials to be used for restoration purposes to be clinically successful, they have good internal characteristics, as well as be able to withstand environmental factors in the environment they are exposed to (20, 21). In addition, the human oral cavity is a moist environment in which the applied restorative material is in contact with saliva at any time, and factors such as the low pH of acidic foods and beverages can affect the mechanical and physical properties of restoration (22). In the study, tea, coffee, and cola were known as the most common types of beverages; these fluids were preferred for test

solutions. Based on some studies, distilled water was chosen as a control group (23-25).

Recently, devices like spectrophotometers have been developed to analyze color changes in dental materials. These devices eliminate the subjective aspects of color assessment and provide the analysis of color differences with the CIELAB system (26-29). For this reason, a spectrophotometer was used to detect even the most critical color differences. In addition, the fact that the color measurements obtained by spectrophotometer be able to repeat was the other reason we prefer this device. According to many studies, the clinically acceptable color change threshold value is accepted as 3.3 (26-29). For samples that are kept in distilled water, Equia Forte and Ionofil are under this threshold (2.8 and 0.66 respectively), and for AHfil Silver, this value (3.57) is above the threshold value. Since distilled water does not contain colorants, occurred the higher color change on AHfil Silver than this threshold value is remarkable. However, this change can be attributed to the amount of water absorbed by glass ionomer cement over time (4).

A study reported that the staining in restorations took place within the first week and that stain penetration could reach up to 5µ (30). In this study, samples were stored in solutions for one week. All specimens stored in colored solutions (tea, coffee, cola) showed a color

change above the acceptable threshold value. In the study, the highest color change was observed in Ahfil Silver samples stored in cola. When glass ionomer cement is stored in acidic environments, it has been reported to release more fluoride than those kept in neutral or basic ambient. Continuous exposure to different pH can cause ion change and significant color change (31). Another research that evaluates the color change in glass ionomer cement similar to our study also found the highest color change is in the group stored in the cola. Researchers have attributed this finding that the low pH of the cola caused degradation on the material surface and causes excessive coloring (14). In our study, the most color change in Ahfil Silver and Ionofil was observed in the examples stored in cola similarly with the above research.

In Equia Forte samples, the highest color change was detected in samples stored in coffee. Research, which obtained similar findings with this result in the study, observed that coffee causes more color change than other drinks (32-34). Bagher et al. argued that the cola had a low pH and deteriorated the surface of the restorative materials, but it did not cause the high color change as it does not contain yellow dyestuff substances like coffee and tea (35). The high color change in the Equia Forte samples stored in coffee may be a high rate of yellow dyestuff in the coffee. These different findings obtained from similar restorations have shown that color change may be due to many factors such as the composition of the storage environment, titratable acidity, maturation time, and yellow dyestuff absorption/penetration (4).

The concept of surface hardness, which provides information about the harmony of restorative materials with dental hard tissues, is one of the most important factors affecting the clinical success of restorations. In this study, as in many studies, Vickers microhardness test device was used.

The changes in the microhardness values of the materials tested in the study were statistically significant in the samples stored in distilled water and tea for Equia Forte, in tea for Ahfil Silver, and in distilled water and tea for Ionofil. The initial microhardness values ( $MH_1$ ) were lower than the microhardness values obtained after being immersed in solutions for one week ( $MH_2$ ). These results were not in line with some other studies (7,8,15). A study with similar results to our study argued that after 30 days of storage in artificial saliva increased ionic cross-links and due to the formation of an insoluble polysalt matrix increased surface hardness in all tested glass ionomer cement

over time (36). In addition, the setting reaction of glass ionomer cements involves the reaction of  $Ca^{2+}$  and  $Al^{3+}$  ions released from aluminofluorosilicate glass with water-soluble polymeric acid (37). During the maturation of the cement, the  $Al^{3+}$  ions initially in four coordination states progress to six coordination states, which improves the mechanical properties of the cement to some extent. The fact that the glass ionomers become stronger over time can be attributed to additional cross-linking and formation of the silica gel phase (38).

The highest microhardness values were obtained in Equia Forte after storage in solutions. Equia Forte was the only encapsulated glass ionomer cement tested in the study. It is believed that the encapsulated glass ionomers eliminate incorrect powder/liquid ratio adjustment before mixing and the product is mixed mechanically and standardly according to the manufacturer's instructions. In addition, Equia Forte has a higher P/L ratio than other hand-mixed glass ionomers. This increases the initial viscosity and homogeneity of the mixture and improves its mechanical properties (39). Although the encapsulation of glass ionomer cements is not directly related to the surface hardness, it is expected that the mixing efficiency will increase the setting reaction rate and thus cause the surface hardness to increase more rapidly over time (40). The null hypothesis was rejected since the detected differences in both microhardness and color changes of the glass ionomer stored in solutions.

The storage time of the tested glass ionomer in solutions is a week and the low number of immersed solutions can be expressed as a limitation.

In conclusion, among the tested materials, the most color change was detected in the samples stored in coffee and cola. This may be due to the low pH-related ion release in cola and the excess quantity of yellow dyestuff in coffee. Microhardness values of glass ionomer cements stored in different solutions increased. This may be due to the increasing number of ionic bonds over time. The encapsulated glass ionomer cement showed high microhardness values, eliminating user-related errors in mixing.

#### **Conflict of Interest Statement**

The authors have no conflicts of interest to declare.

#### **Ethical Approval**

This article does not contain any studies with human or animal subjects.

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## Availability of Data and Materials

Data available on request from the authors.

## Authors Contributions

UBT: Conceptualization; Data curation; Formal analysis; Investigation; Methodology; Validation; Visualization; Writing-original draft.

ÖKH: Conceptualization; Formal analysis; Funding acquisition; Investigation; Methodology; Project administration; Resources; Supervision; Validation; Writing-review & editing.

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