

Influence of Thermo-Light Curing on the Microhardness and Fluoride Release of Glass-Ionomer Cements

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Glass ionomer cements (GICs) are important restorative materials that are frequently preferred in both primary and permanent teeth. The aim of this study was to investigate the effect of thermo-light curing on the surface microhardness and fluoride ion (F⁻) release of the materials during the curing reactions of high-viscosity GICs. In our study, Equia Fil (EQ-GC), Fuji IX GP Extra (FGP-GC), and Fuji IX Capsule (FC-GC) were prepared in accordance with the manufacturer's instructions, and for 60 and 90 seconds during curing Woodpecker LED-C (WL), GC D-Light Pro (GLC) and BlueLuxcer M-855 Halogen Curing Light (HALO) light instruments formed the experimental groups, and self-curing glass ionomer cement samples formed the control groups. VHN and F⁻ release values of both groups were evaluated on 1st day, 1st week, 1st, 3rd, and 6th months. The Jamovi (Version 1.0.4) program was used for statistical analysis. Significant difference was analyzed by the Tukey's post hoc test ($\alpha = 0.05$). In the 1st month, EQ exhibited significantly higher microhardness than FC ($p < 0.05$) but not with FGP ($p > 0.05$). All materials exhibited higher microhardness during 1st day than in other periods ($p < 0.05$). In terms of F⁻ release, there were significant differences between materials on the 1st day ($p < 0.05$). EQ, FC, and FGP exhibited higher F⁻ release, respectively. In the 1st, 3rd, and 6th months, EQ exhibited released significantly higher F⁻ significantly ($p < 0.05$), whereas this amount was similar between FC and FGP ($p > 0.05$). Within the limitations of this study, it can be concluded that thermo-light application does not cause unacceptable long-term changes in the physical and chemical properties of materials.

Keywords: Fluoride release, Glass-ionomer cement, Microhardness, Thermo-light curing

1. Introduction

Glass ionomer cement (GIC) is an indispensable restorative material frequently used in pediatric dentistry [1,2]. They are preferred for both primary and permanent tooth restorations due to their beneficial properties, such as their anti-cariogenic effects, chemical adhesions to dental tissues, and

biocompatible structures resulting from fluoride ion (F⁻) release [3]. These properties have made GIC a critical material in treating initial carious lesions and pediatric patients with high caries risk [4].

The mechanical and physical properties of GIC are lower than resin-based restorative materials (RBCs) and amalgam. Their sensitivity to moisture

and drying during setting and insufficient physical properties are disadvantages [2]. Their moisture sensitivity and longer setting time result in a lower final strength [5,6]. During the initial stage of curing glass ionomer cements, incomplete chemical reactions and sensitivity to water result in a soft, porous, brittle surface susceptible to crack formation, which reduces wear resistance and fracture strength [7,8].

One of the methods developed to improve the physico-mechanical properties of GICs is to catalyze the reaction by applying heat, especially at the initial stage of the setting reaction. Heat application can be achieved using high-energy LED light sources, halogen lamps, ultrasonic devices, and diode lasers [5,7,9,10]. These applications shorten the maturation process of GICs and improve the setting process. It is because the reduction in the duration of the initial reaction makes clinical application faster and easier, reduces the possibility of restoration failure, and enables the patient to start using the restoration in a shorter time [5,7,9].

It has been reported that the setting time of GIC is 24 hours after mixing [11]. Surface hardness reveals the maturation quality of the GIC. Accelerating the initial setting reaction with an external energy source may lead to a decrease in the ‘bursting effect,’ which is the first process of F⁻ release that occurs in the initial phase of the mixture [5,10]. While improving the physical properties of the material, a decrease in the release of F⁻ is not a desirable property [5,12].

This study aimed to evaluate the effect of radiant heat energy generated by various light sources applied at different times during the initial setting reactions of GICs with different properties on the microhardness and F⁻ release. The null hypotheses of the study are as follows:

- 1) There is no difference between GIC materials in microhardness.
- 2) There is no difference between the light sources in microhardness.
- 3) There is no difference between GIC materials in F⁻ release.
- 4) There is no difference between the light sources in F⁻ release.

2. Experimental

2.1. Materials

Three high-viscosity GIC materials were tested in this study: Equia Fil (EQ) (GC, Japan), Fuji IX GP Extra powder-liquid (FGP) (GC, Tokyo, Japan), and Fuji IX Capsule (FC) (GC, Japan). The technical specifications of the materials are presented in Table 1. For each GIC, 49 disc-shaped specimens (10 mm in diameter and 2 mm in thickness) were prepared using cylindrical Teflon molds. A total of 294 specimens were obtained from 147 each to be evaluated for both surface microhardness and F⁻ release. Capsules with GIC were placed in a Capsule Mixing Unit (RotoMix, 3M ESPE) for the recommended time (10 s for each material) after activation. The mixed capsules were immediately injected into the mold. Before placing the material, a polyester strip is laid on a glass plate. After filling the mold with GIC, a second polyester strip was placed over the mold and pressed with a second glass sheet to achieve a standard surface finish and remove excess material. FGP in powder and liquid form was prepared by the recommended instructions and placed in molds as applied in capsule forms.

2.2. Experimental

The study groups (control and experiment) were randomly allocated to 7 groups (n = 7) for each GIC material. Group 1 (G1) (control) samples did not undergo any application during the setting and were removed from the Teflon molds after waiting for about 5 minutes. Group 2 (G2) and Group 3 (G3) samples were cured with Woodpecker LED-C (WL) (Woodpecker, China) at 1200 mW/cm² for 60 and 90 s, respectively. Group 4 (G4) and Group 5 (G5) samples were cured with GC D-Light Pro (GLC) (GC, Japan) at 1400 mW/cm² for 60 and 90 s, respectively. Group 6 (G6) and Group 7 samples were cured with BlueLuxcer M-855 Halogen Curing Light (HALO) (Monitex, China) at 1000 mW/cm² for 60 and 90 s, respectively. Before the EQ and FC samples in both control and experimental groups were taken into storage containers, G-Coat (GC, Tokyo, Japan) was applied to the upper surface as specified in the instructions for use and cured with WL for 20 s. The samples were then placed in cylindrical plastic containers

Table 1. The technical characteristic of the tested materials

Material	Category	Manufacturer	Composition
Equia Fil	Conventional GIC (Capsule)	GC Dental	Flouro-alitmia silicate glass, polyacrylic acid, distilled water
Fuji IX GP Extra	Conventional GIC	GC Dental	water, fluoroaluminosilicate glass, polybasic carboxylic acid, polyacrylic acid
Fuji IX Capsule	Conventional GIC (Capsule)	GC Dental	glass, polyacrylic acid, polyacrylic acid
G-Coat	Coating	GC Dental	methyl methacrylate, colloidal silica, phosphoric ester monomer

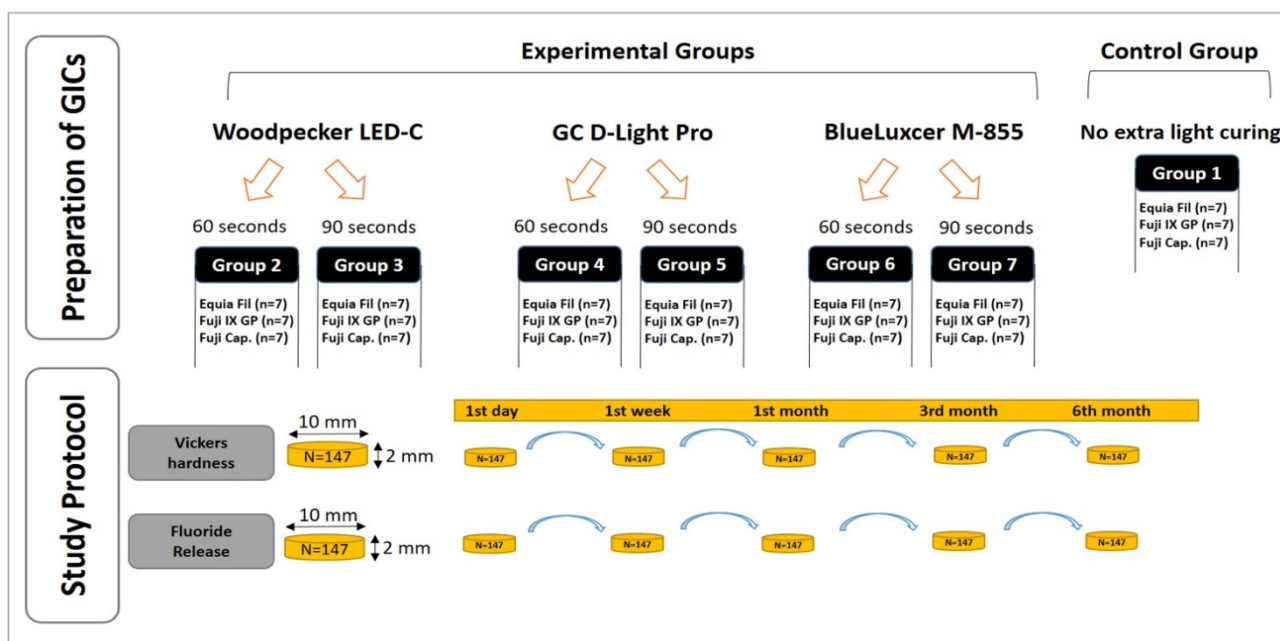


Fig. 1. The preparation process and study protocol.

with a diameter of 32 mm and a height of 50 mm and containing 5 ml of deionized water (ph~7). Then, it was kept in an oven at 37 °C (VWR, Pennsylvania, USA) for 24 h. The preparation process and study protocol are illustrated in Fig 1. 90 s, respectively. Group 4 (G4) and Group 5 (G5) samples were cured with GC D-Light Pro (GLC) (GC, Japan) at 1400 mW/cm² for 60 and 90 s, respectively. Group 6 (G6) and Group 7 samples were cured with BlueLuxcer M-855 Halogen Curing Light (HALO) (Monitex, China) at 1000 mW/cm² for 60 and 90 s, respectively. Before the EQ and FC samples in both control and experimental groups were taken into storage containers, G-Coat (GC, Tokyo, Japan) was applied to the upper surface as specified in the instructions for use and cured with WL for 20 s. The samples were then placed in cylindrical plastic containers with a diameter of 32 mm and a height of 50 mm and containing 5 ml of deionized water (ph~7). Then, it was kept in an oven at 37 °C (VWR, Pennsylvania, USA) for 24 h. The preparation process and study protocol are illustrated in Fig 1.

2.3. Evaluation of surface microhardness

Seven samples prepared for each study group (control and experimental groups) were placed in acrylic blocks, and a total of 42 acrylic blocks, 14 for each material, were used. Silicon carbide (SIC) abrasives of 600, 800, 1000, and 1200 grit were used in an automatic polishing device (Buehler, Illinois, United States) to standardize the sample surfaces. The surface hardness of the samples was measured

using a Vickers hardness device (Struers, Germany). A 200 g load was applied to the GIC discs for 15 seconds with a square-bottomed and pyramid-shaped diamond tip. Each sample surface was divided into four equal quadrants, and 4 measurements were made from each quadrant. Measurements were made in 5 different periods: 1st day, 1st week, 1st month, 3rd month, and 6th month. The mean of the measurements was recorded as the Vickers hardness of the material. The Vickers hardness (VHN) of the samples was calculated according to the formula $VHN=1.854 F/d^2$.

2.4. Evaluation of fluoride release amount

Sample discs were diluted with deionized water in 100 ppm standard fluoride solution (Thermo, Orion, Massachusetts, USA). 100 ppm, 10 ppm, 1 ppm, 0.1 ppm, 0.01 ppm solutions were prepared, and the calibration of the F⁻ ion selective electrode (Thermo-Orion, Massachusetts, USA) was performed before the measurement. The diluted samples were measured using a calibrated ion meter (Thermo-Orion, Massachusetts, USA), and the values were recorded. In order to perform the F⁻ measurement in the test samples, the GIC discs were transferred into a new plastic tube and removed from the oven by adding 5 ml of deionized water. 0.5 mL of total ionic strength adjustment buffer Tisab III (Thermo-Orion, Massachusetts, USA) was added to the remaining 5 ml of test sample liquid. The liquid in the tube was mixed with a magnetic stirrer (IKA, Staufen, Germany) and homogenized. Fluoride measurements were made with an ion

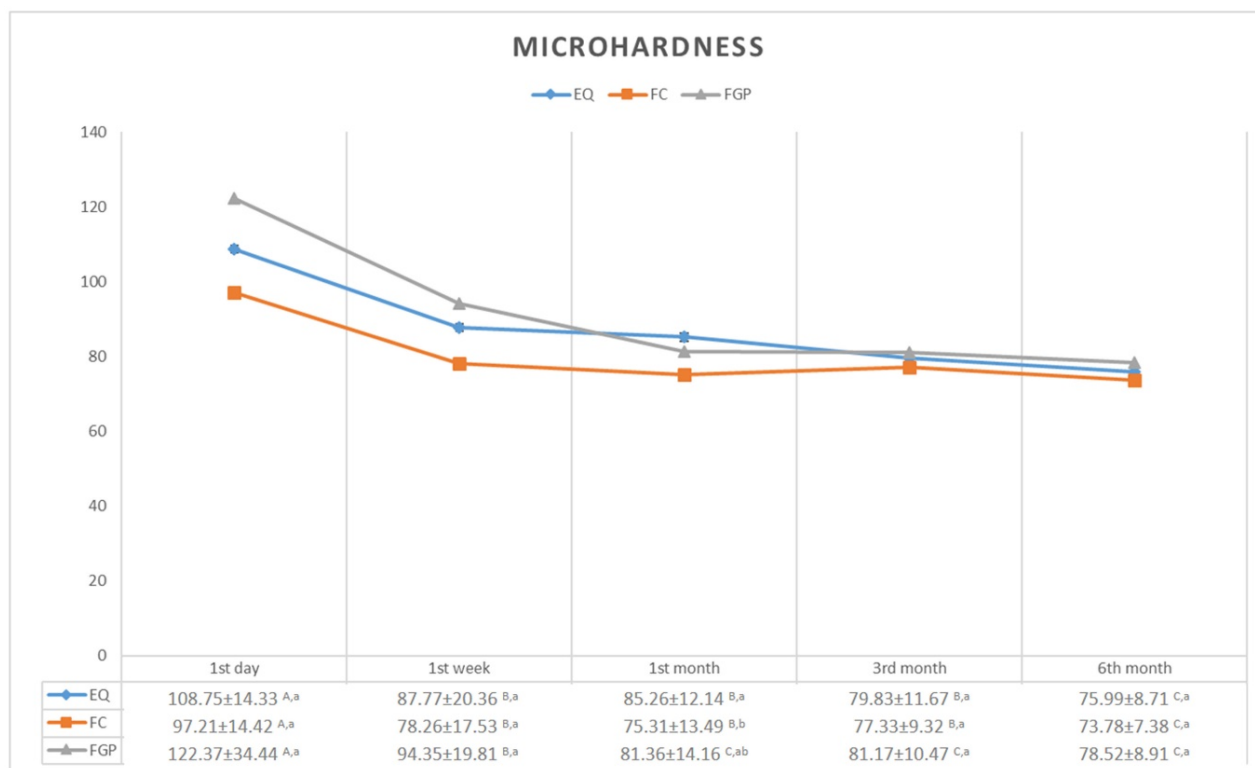


Fig. 2. Variation of microhardness of materials with time.

meter (Thermo-Orion, Massachusetts, USA) at room temperature. Measurements were repeated in 5 different periods 1st day, 1st week, 1st month, 3rd month, and 6th month. The results were recorded as $\mu\text{g}/\text{mm}^2$ by calculating the amount of F⁻ ions released per unit area from the sample surface.

2.5. Statistical analysis

The Jamovi (Version 2.2.5) [ComputerSoftware], as accessed by <https://www.jamovi.org>, software was used for statistical analysis. The normality of data distribution was checked using the Shapiro–Wilk test. Due to normal distribution, Repeated Measure ANOVA was conducted to measure the differences among factors. A significant difference was analyzed by Tukey’s post hoc test. The significance level was set at $p=0.05$.

3. Results and discussion

In terms of microhardness, there was no significant difference between materials in any period ($p>0.05$) except the 1st month. In the 1st month, EQ exhibited significantly higher microhardness than FC ($p<0.05$) but not with FGP ($p>0.05$). All materials exhibited higher microhardness during the 1st day than in other periods ($p<0.05$). EQ and FC exhibited similar microhardness in the 1st week, 1st month, and 3rd month ($p>0.05$), but the least microhardness was

obtained in the 6th month ($p<0.05$). The microhardness that FGP exhibited was significantly higher in the 1st week than in other periods ($p<0.05$) (Fig. 2).

No difference between light curing devices was obtained in any period ($p>0.05$) except the 6th month ($p<0.05$). In the 6th month, the microhardness exhibited by WL 60 and GLC 90 was less than others ($p<0.05$). On the 1st day, higher microhardness was obtained by Control and Halo 60 than in other periods ($p<0.05$), and no difference was obtained between other periods ($p>0.05$). Regarding WL 60, significantly, the highest and least microhardness was significantly obtained in the 1st and 6th month, respectively ($p<0.05$). The microhardness that was obtained by WL 90 and GLC 60 was significantly higher than the 1st, 3rd, and 6th months, significantly ($p<0.05$), except the 1st week ($p>0.05$). Regarding WL 90, significantly least microhardness was obtained in the 6th month ($p<0.05$). The microhardness that was obtained by GLC 90 on the 1st day was significantly higher than other periods ($p<0.05$) and that obtained in the 1st month was higher than the 6th month ($p<0.05$). Regarding Halo 90, no significant difference was found between periods ($p>0.05$) (Fig. 3).

In terms of fluoride release, there were significant differences between materials on the 1st day ($p<0.05$). EQ, FC, and FGP exhibited higher

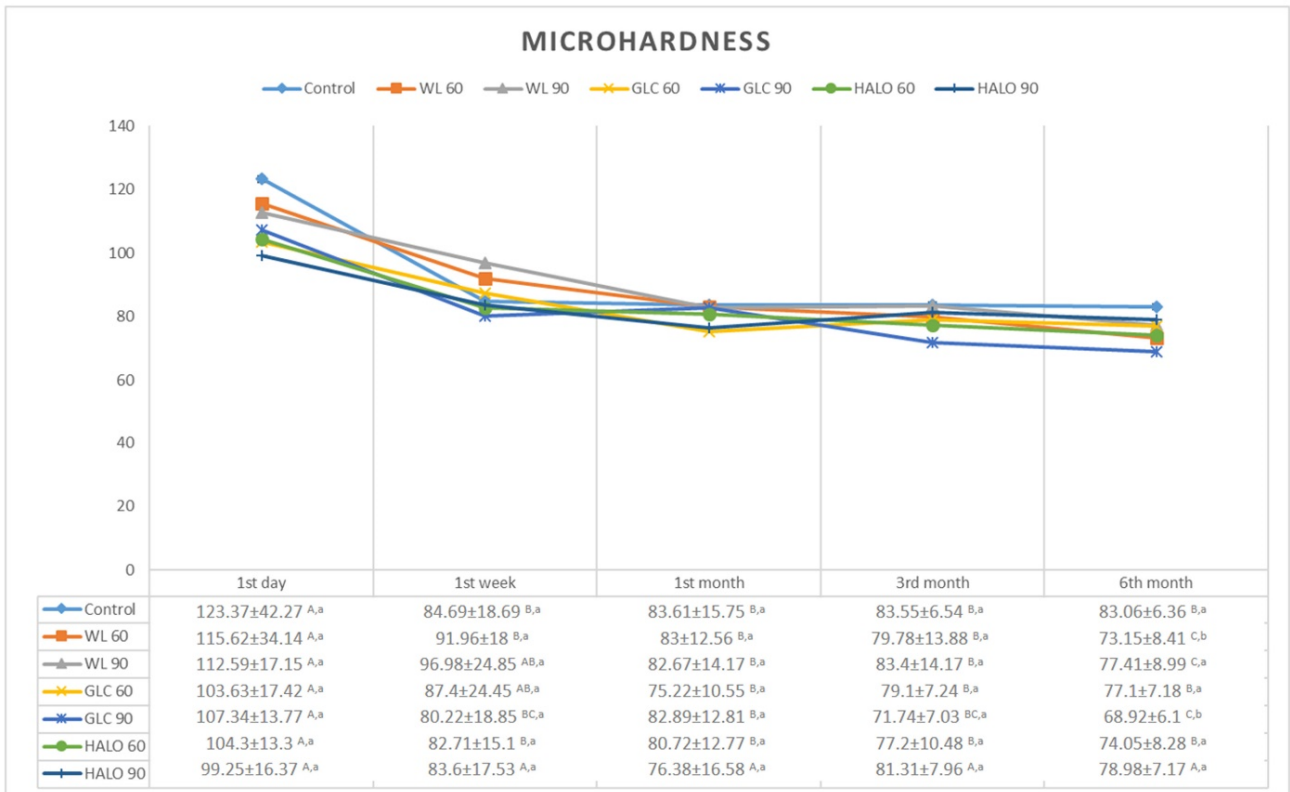


Fig. 3. Variation of microhardness of groups with time.

fluoride release, respectively. In the 1st week, the amount of fluoride released by EQ and FC was similar ($p>0.05$), whereas it was higher than FGP ($p<0.05$). In the 1st, 3rd, and 6th months, EQ exhibited released significantly higher fluoride significantly ($p<0.05$), whereas this amount was

similar between FC and FGP ($p>0.05$). For all materials, the fluoride release amount was significantly higher in the 1st month than in other periods ($p<0.05$), except in the 3rd month ($p>0.05$) (Fig. 4).

No difference between light curing devices

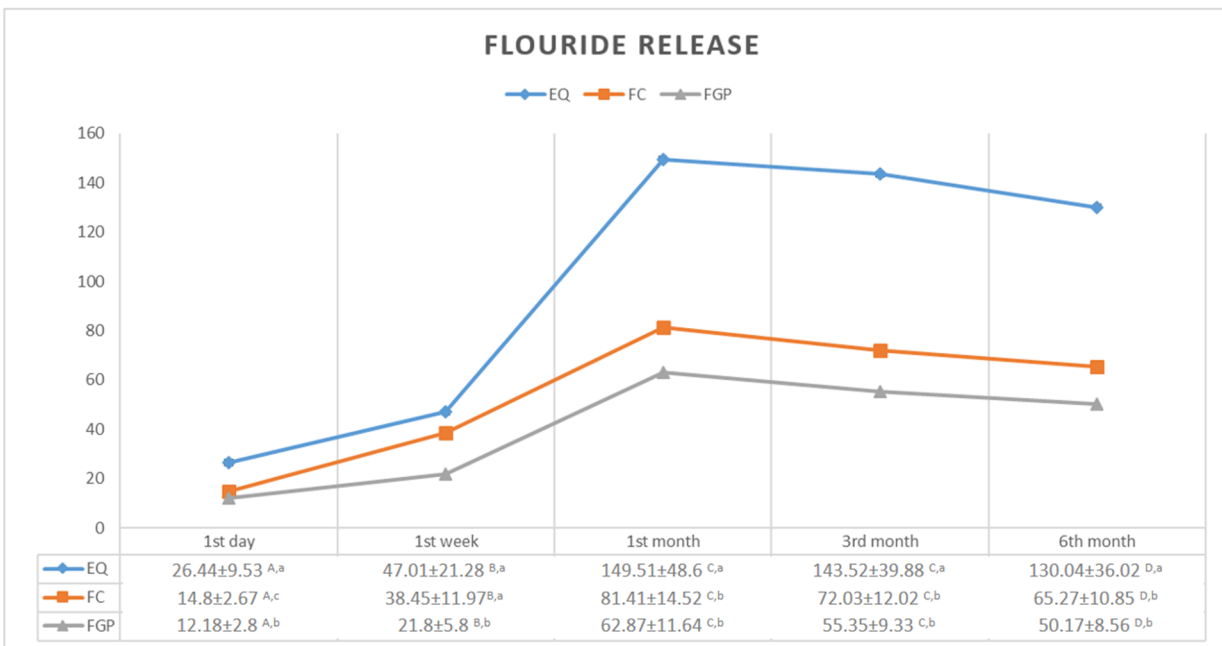


Fig. 4. Variation of fluoride release of materials with time.

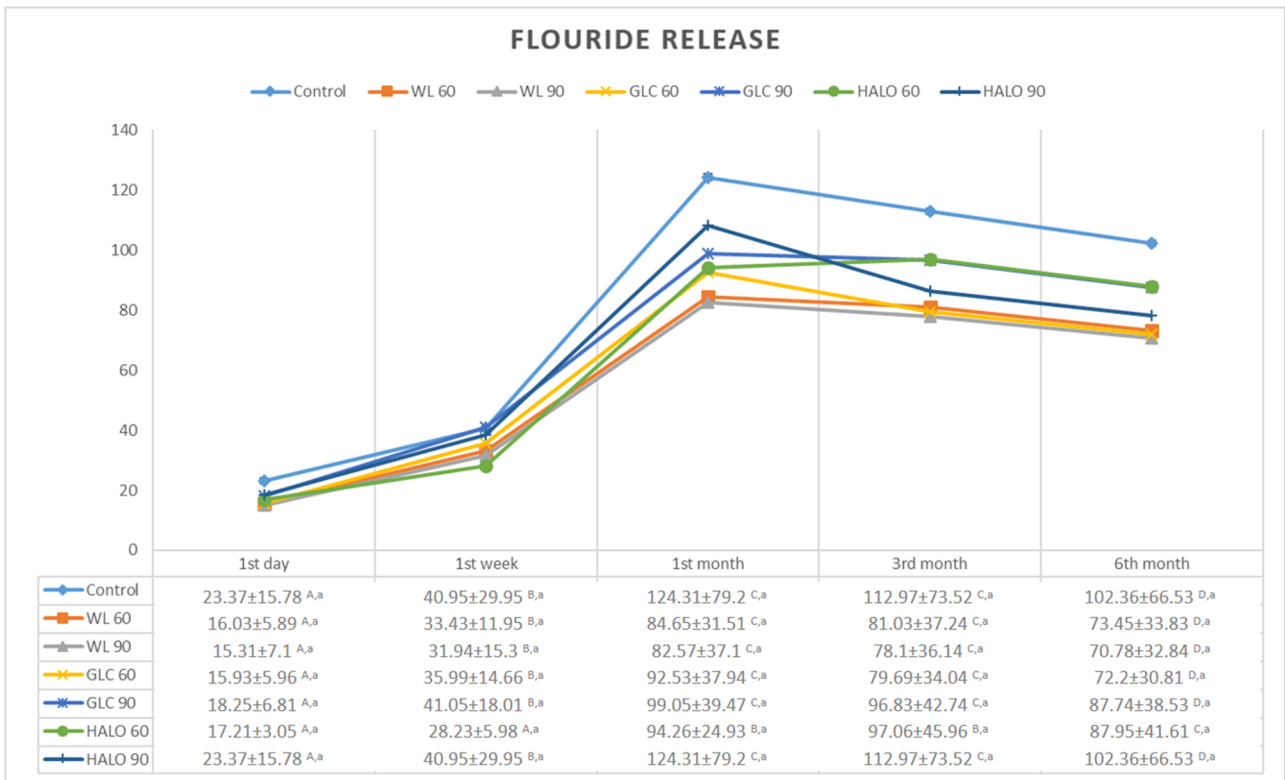


Fig. 5. Variation of fluoride release of the groups with time.

was obtained in any period regarding fluoride release ($p>0.05$). For all light curing devices except Halo 60, the fluoride release was significantly higher in the 1st month than in other periods ($p<0.05$), except the 3rd month ($p>0.05$) (Fig. 5).

Detailed analyses (Material type * Light cure * Time) are shown in Table 2 and Table 3 for microhardness and fluoride release, respectively.

GICs are one of the most preferred materials, especially in pediatric dentistry. While it has the

Table 2. Repeated Measure ANOVA posthoc comparisons (Material type*Light cure*Time) to detect differences in terms of Fluoride release.

Material	Light Cure	1 day	1 week	1 month	3 months	6 months
EQ	Control	43.68±9.68 ^{D,a}	79.71±17.64 ^{C,a}	228.03±38.6 ^{AB,a-c}	210.38±34.34 ^{A,a}	190.59±30.69 ^{B,a}
	WL 60	22.71±4.69 ^{C,b-e}	39.11±10.6 ^{C,b-f}	123.31±19.36 ^{AB,a-e}	129.58±18.37 ^{A,b}	117.47±17.07 ^{B,b}
	WL 90	23.32±6.55 ^{C,b-d}	38.98±19.16 ^{C,b-f}	121.85±37.89 ^{AB,a-e}	120.3±31.63 ^{A,bc}	109.06±28.92 ^{B,bc}
	GLC 60	22.85±5.47 ^{D,b-e}	41.57±11.48 ^{C,b-e}	137.49±25.57 ^{A,d-i}	121.97±20.64 ^{A,bc}	110.51±18.58 ^{B,bc}
	GLC 90	25.97±4.97 ^{C,bc}	44.95±22.11 ^{C,b-d}	138.08±44.01 ^{AB,ij}	144.26±38.43 ^{B,b}	130.63±34.15 ^{A,b}
	HALO 60	18.84±3.66 ^{D,c-f}	29.62±5.95 ^{C,c-g}	120.99±15.56 ^{AB,h-j}	153.92±29.59 ^{B,b}	139.47±26.73 ^{A,b}
	HALO 90	27.71±6.38 ^{E,b}	55.13±17.62 ^{D,b}	176.84±41.67 ^{A,e-i}	124.22±15.79 ^{B,b}	112.58±14.29 ^{C,b}
FGP	Control	11.29±1.82 ^{D,f}	20.01±2.01 ^{C,fg}	58.26±4.65 ^{AB,a-f}	57.46±5.75 ^{A,de}	52.09±5.41 ^{B,de}
	WL 60	11.41±1.84 ^{E,f}	21.39±1.01 ^{D,fg}	60.24±3.24 ^{A,ij}	51.51±2.33 ^{B,e}	46.68±2.1 ^{C,e}
	WL 90	10.47±2.84 ^{D,f}	17.6±1.66 ^{C,fg}	53.38±3.69 ^{A,fj}	49.32±3.26 ^{A,e}	44.69±2.77 ^{B,e}
	GLC 60	12.55±1.71 ^{D,f}	18.97±2.46 ^{C,fg}	56.47±4.8 ^{A,ab}	49.02±5.08 ^{A,e}	44.41±4.45 ^{B,e}
	GLC 90	11.96±2.99 ^{C,f}	25.72±8.86 ^{C,d-g}	72.93±16.79 ^{AB,b-h}	58.86±12.02 ^{A,de}	53.38±11.17 ^{B,de}
	HALO 60	15.52±2.39 ^{D,d-f}	26.09±6.46 ^{C,d-g}	71.47±13.64 ^{AB,a-d}	59.87±12.41 ^{A,de}	54.3±11.45 ^{B,de}
	HALO 90	12.07±3.48 ^{C,f}	22.81±7.79 ^{C,e-g}	67.32±11.58 ^{AB,a}	61.38±11.4 ^{A,de}	55.64±10.46 ^{B,de}
FC	Control	15.14±1.67 ^{C,ef}	23.13±6.32 ^{C,e-g}	86.63±10.08 ^{A,a-e}	71.08±9.49 ^{A,de}	64.39±8.42 ^{B,de}
	WL 60	13.97±2.9 ^{D,f}	39.78±10.43 ^{C,b-f}	70.4±15.8 ^{AB,g-j}	62±9.45 ^{A,C,de}	56.2±8.67 ^{B,C,de}
	WL 90	12.15±1.81 ^{D,f}	39.24±7.15 ^{C,b-f}	72.48±14.98 ^{AB,a-g}	64.67±9.46 ^{A,de}	58.61±8.58 ^{B,de}
	GLC 60	12.38±1.24 ^{D,f}	47.41±7.23 ^{C,bc}	83.63±12.36 ^{AB,c-h}	68.07±8.4 ^{A,de}	61.69±7.6 ^{B,de}
	GLC 90	16.82±1.68 ^{D,d-f}	52.47±8.33 ^{C,b}	86.14±14.41 ^{AB,j}	87.38±7.42 ^{A,cd}	79.21±6.98 ^{B,cd}
	HALO 60	17.27±2.33 ^{D,d-f}	28.99±5.8 ^{C,c-g}	90.32±13.62 ^{AB,h,j}	77.38±13.21 ^{A,de}	70.07±11.68 ^{B,de}
	HALO 90	15.88±1.88 ^{D,d-f}	38.15±9.28 ^{C,b-f}	80.25±13.17 ^{AB,a-h}	73.64±8.7 ^{A,de}	66.72±7.75 ^{B,de}

Different uppercase and lowercase letters indicate the significant difference in rows and columns, respectively ($p<0.05$).

Table 3. Repeated Measure ANOVA posthoc comparisons (Material type*Light cure*Time) to detect differences in terms of microhardness.

Material	Light Cure	1 day	1 week	1 month	3 months	6 months
EQ	Control	103.57±12.13 ^{A,cd}	93.44±25.3 ^{A,a-c}	92.64±19.1 ^{A,a}	82.64±10.3 ^{A,a-e}	84.29±10.32 ^{A,a-c}
	WL 60	114.8±18.24 ^{A,b-d}	87.51±26.29 ^{AB,a-c}	79.29±6.44 ^{B,ab}	95.2±9.22 ^{A,a}	82.27±5.74 ^{B,a-e}
	WL 90	103.16±12.4 ^{A,cd}	90.2±14.11 ^{A,a-c}	91.16±12.12 ^{A,a}	89.04±12.23 ^{A,ab}	83.07±6.43 ^{A,a-e}
	GLC 60	104.57±14.13 ^{A,cd}	103.26±19.7 ^{A,ab}	86.09±3.43 ^{AC,ab}	73.96±6.17 ^{BC,b-e}	72.87±5.03 ^{B,d-h}
	GLC 90	112.41±11.56 ^{A,b-d}	68.79±15.87 ^{BC,bc}	85.61±11.04 ^{B,ab}	70.63±8.1 ^{BC,c-e}	66.73±5.27 ^{C,h}
	HALO 60	108.79±15.25 ^{A,b-d}	77.54±4.13 ^{BC,a-c}	82.64±11.78 ^{A-C,ab}	71.8±3.34 ^{B,c-e}	70.04±3.64 ^{C,gh}
	HALO 90	113.96±16.27 ^{A,b-d}	93.66±14.9 ^{AC,a-c}	79.39±12.61 ^{BC,ab}	75.51±3.8 ^{B,b-e}	72.64±3.03 ^{B,e-h}
	Control	170.87±40.53 ^{A,a}	85.24±14.28 ^{B,a-c}	84.27±11.43 ^{B,ab}	84.19±3.88 ^{B,a-e}	81.96±3.4 ^{A,f}
FGP	WL 60	140.89±44.31 ^{A,ab}	95.43±13.89 ^{AC,ab}	78.87±11.14 ^{BC,ab}	69.04±4.22 ^{B,de}	66.63±4.76 ^{B,h}
	WL 90	120.73±14.32 ^{A,bc}	110.29±36.03 ^{A,a}	80.01±9.95 ^{B,ab}	77.21±18.83 ^{B,b-e}	70.66±10.28 ^{B,f-h}
	GLC 60	120.94±6.55 ^{A,bc}	98.54±13.81 ^{B,ab}	74.1±9.14 ^{B,ab}	85.21±3.59 ^{B,a-d}	84.66±3.58 ^{B,ab}
	GLC 90	113.59±11.07 ^{A,b-d}	89.21±18.23 ^{AB,a-c}	87.2±14.48 ^{B,ab}	77.39±5.63 ^{B,b-e}	75.4±3.81 ^{B,b-h}
	HALO 60	105.46±14.05 ^{A,cd}	96.31±16.42 ^{A,ab}	81.01±17.34 ^{AB,ab}	85.39±6.82 ^{B,a-c}	83.54±4.11 ^{B,a-d}
	HALO 90	84.14±5.83 ^{A,d}	85.43±10.47 ^{A,a-c}	84.04±22.94 ^{A,ab}	89.73±7.51 ^{A,ab}	86.8±5.91 ^{A,a}
	Control	95.67±13.5 ^{A,cd}	75.37±11.39 ^{A,bc}	73.91±11.23 ^{A,ab}	83.81±4.46 ^{A,a-e}	82.94±3.67 ^{A,a-e}
	WL 60	91.19±12.1 ^{AB,cd}	92.93±12.55 ^{A,a-c}	90.84±15.9 ^{AB,a}	75.09±10.1 ^{B,b-e}	70.54±5.04 ^{B,gh}
FC	WL 90	113.89±20.95 ^{A,b-d}	90.44±15.88 ^{AB,a-c}	76.83±17.06 ^{B,ab}	83.96±9.13 ^{B,a-e}	78.5±5.51 ^{B,a-g}
	GLC 60	85.39±5.44 ^{A,d}	60.41±11.23 ^{B,c}	65.49±5.08 ^{B,b}	78.13±6.92 ^{A,b-e}	73.79±5.78 ^{B,c-h}
	GLC 90	96.01±12.28 ^{A,cd}	82.66±18.6 ^{AB,a-c}	75.86±11.29 ^{AB,ab}	67.21±2.24 ^{B,e}	64.63±2.41 ^{B,h}
	HALO 60	98.66±9.86 ^{A,cd}	74.27±11.99 ^{BC,bc}	78.51±9.71 ^{AB,ab}	74.43±13.68 ^{A-C,b-e}	68.57±6.3 ^{C,gh}
	HALO 90	99.66±8.7 ^{A,cd}	71.73±20.17 ^{BC,bc}	65.71±4.38 ^{B,b}	78.69±3.27 ^{C,b-e}	77.49±2.53 ^{C,a-g}

Different uppercase and lowercase letters indicate the significant difference in rows and columns, respectively (p<0.05).

advantage of protecting against dental caries with fluoride release, its mechanical properties are lower than several materials, such as dental composites [13]. The poor mechanical properties lead to the development of new techniques and materials. This study investigated the effect of different light sources on microhardness and fluoride release in different GIC products.

The first null hypothesis was rejected. Regarding surface microhardness, EQ revealed statistically higher microhardness than FC and FGP in one month. These results align with the results of other studies [5,14]. Higher microhardness values yielded in high-viscosity GICs compared to conventional GICs may be attributed to higher cross-linking occurring during acid-base reactions [11,15]. Besides, differences in surface hardness between materials may be attributed to variations in composition. Powder/liquid ratios and molecular weight, viscosity, and concentration of polyalkenoic acid impact the properties of GICs [2,7].

The second null hypothesis was also rejected. The light sources revealed significant differences in microhardness on the 6th month, when obtained significantly less microhardness was obtained by GLC90 and WL60. However, no difference was obtained in the other periods. While several studies found that external energy sources increased microhardness [5,7,12], some revealed the opposite results [14]. Heat may accelerate the matrix-formation process of the cement, and thus the

setting reaction in the first stage will result in improved and higher surface hardness [16]. However, in the present study, although the light intensity in GLC (1400 mW/cm²) was higher than in others, long-term application negatively affected the microhardness. GICs are brittle materials and become more brittle as the curing reaction progresses. External energy may have reduced microhardness, causing the material to be highly brittle.

The current study examined the long-term change in microhardness with two different application periods (60-90 sec). The microhardness was observed from the first day to the sixth month. A significantly higher microhardness value was observed on the first day than in other periods. Acid-base reactions of GICs are completed within 24 hours, and it is necessary to wait for one week to gain sufficient physical properties [2,17]. In previous studies, contrary to ours, the microhardness values on the first day were lower than in other periods [5,10,14]. The curing process occurs after the gelation stage, and an increase in hardness occurs due to the continued formation of aluminum salt bridges. Storage strengthens the matrix of cement, thus leading to superior mechanical properties. However, long-term storage may cause a decrease in physical strength as a result of the erosion and plasticizing effect of water [3,8]. The long storage period of 6 months may have decreased the microhardness over time.

The third null hypothesis was accepted. A difference was not observed between light sources regarding F⁻ release in any period. However, some studies observed lower F⁻ release after radiant heat treatment [5]. Extra applied heat can reduce the bursting effect of the initial F⁻ release in the first hours after mixing. Although it was seen that there was a relative decrease in the results of this study, it did not yield statistical significance.

The fourth null hypothesis was rejected. In terms of F⁻ release, there was a significant difference between GIC materials. Consistent with previous studies [5], the highest F⁻ release was observed in EQ in all study groups, while the lowest value was observed in FG. Fluoride release involves complex reactions and can be affected by a variety of intrinsic and extrinsic factors, such as the type of glass particles and polyalkenoic acid, the natural fluoride content, the solubility and porosity of the material, the powder/liquid ratio used in the preparation of the material, the mixing method, and the type of storage medium [17,18]. The underlying reason for the higher fluoride release capacity of EQ may be attributed to its higher viscosity obtained by supplementing with nano-fillers.

While F⁻ release increased significantly until the first month and reached its highest value, it decreased from the first month to the sixth month. Fluoride release from restorative materials in short and long periods varies depending on factors such as integrated F⁻ content and natural structure, material components, and curing reactions [3]. The initial F⁻ release from the GIC is directly proportional to the concentration of F⁻ in the material. The high amount of F⁻ released during the first two days is called the "burst effect" phenomenon [8]. Fluoride release decreases rapidly and stabilizes after three to four weeks [19].

4. Conclusion

As a result of this in vitro study, it can be concluded that the lowest value for VHN was observed at month 6, no difference was observed in other periods, and the EQ material showed the highest values in terms of VHN on the first day and in the 1st month in terms of F⁻ release. External light that GIC materials are exposed to during their curing does not cause unacceptable long-term changes in the materials' physical and chemical properties. These outcomes should be supported by further in-vitro and in-vivo studies with larger sample sizes.

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