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THE EFFECT OF THE TIME OF IRRADIATION USING LED UNIT ON WATER SORPTION OF TWO DIFFERENT GLASS IONOMER RESTORATIVES, IN-VITRO STUDY

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ABSTRACT

The aim of this study was to measure the effect of different irradiation times by Light Emitting Diode (LED) on Water sorption and solubility of two different glass ionomer restoratives.

Materials and methods: Two conventional GIC materials Equia Forte (EF) and Fuji IX (FIX) were used in this study. The specimens were divided into 3 experimental groups (n = 10). **In Group 1**, which is the control group of the study, where the specimens after mixing were left in the mold to set without any irradiation. **In Group 2**; the specimens were irradiated for 40 seconds at the top surface using a LED light curing unit using standard curing mode, after 2 minutes of termination of mixing. **In Group 3**; the specimens were irradiated for 40 seconds at the top surface using a LED light curing unit using standard curing mode after 4 minutes of termination of mixing. All specimens were subjected to a pH cycling model. Specimens were tested for water sorption and solubility % at baseline and after pH cycling.

Results: water sorption results in EF were statistically higher than Fuji IX, irrelative to pH modeling. However, the results differ in case of irradiation time, the control group >after 2 min >4 min. while solubility % irrelative to material effect, was higher in pH modeling than baseline, yet 4 min> control group >after 2 minutes irradiation time.

Conclusions: Water sorption is material dependent while solubility is pH dependent. Irradiation by LED could be safe option to improve both.

KEYWORDS: LED, Irradiation time, Water sorption, Solubility, Conventional GICs.

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INTRODUCTION

Due to their favorable characteristics, such as their anti-cariogenic activity, chemical attachment to dental tissues, and biocompatibility, GICs are typically recommended for use with dental restorations. However, compared to resin-containing restorative materials, they have weaker mechanical qualities and a higher solubility percent with induced water sorption.[1,2,3]

The solubility of restorative materials in water and their inability to be properly managed have a negative effect on survival rates. The mass loss of the restorative material is controlled by its solubility, which also hinders its mechanical behavior and interface failure.[4,5] Marginal contours get stained and breaks margins due to water sorption. The bioactivity of restorations is seriously affected by water solubility, which also accelerates the rate of deterioration.

These mechanical characteristics of a traditional GIC are influenced by the make-up of the material and the speed of the setting reaction. The literature contains a large number of studies analyzing the physical characteristics of high-viscosity GICs, but there are few studies analyzing water sorption and solubility. [6,7] It has been suggested to deliver heat energy by employing various forms of laser, ultrasonic energy, as well as dental light curing units on the surface of the GIC restorations, accelerating acid base setting reaction, in an effort to address the aforementioned drawbacks of conventional GICs.[8]

Compared to previous quartz-tungsten-halogen lamps used to light-cure dental materials, modern LED cure lights produce less heat. Modern cure lamps have been marketed as having a benefit due to their lesser heat emission. However, the benefit of exposing them to glass ionomers is negligible because the speed of curing is accelerated by heat rather than light. The most recent LED lights, on the other hand, have larger power densities and hence higher heat emissions. In terms of curing glass-ionomer cements, this might be more efficient.

Fluor-alumino silicate glass powder and poly-acrylic acid aqueous solution interact chemically in an acid-base manner to form glass ionomers cements.[9] In order to fix the GIC setting's initial problems, external energy sources were utilized. As a result, it may be desirable for the physical and mechanical properties of the restorative materials to accelerate and improve the setting response of GICs by applying thermal energy.[10,11] Different storage solutions, such as acetic acid, fuzy tea, or synthetic saliva, are used in vitro experiments to test the health of the oral cavity. Studies have indicated that these investigated solutions alter the sorption or solubility values of several restorative materials. [12]

Earlier research have mentioned, how LED illumination improved some GICs' ability to sorb water. This result was due to heat catalysis. However, it has not yet been investigated in the literature if this effect is affected by the irradiation period in connection to the setting response. Therefore, for a reliably estimate of the clinical performance for sensitive restorative materials, it is critical to understand how LED irradiation effect and/or erosive insult attack affect GIC physical properties. Therefore, the purpose of this research work was to examine glass ionomer solubility and water sorption before and after a pH cycling regimen.

The null hypotheses for this study for both tested parameter, water sorption and solubility were the following:

1. Water sorption and solubility of the glass ionomer cements would not be influenced by thermal irradiation using LED.
2. Timing of LED irradiation would not influence percentage of water sorption and solubility.
3. There is no difference between both tested materials regarding water sorption and solubility.
4. pH cycling would not affect both water sorption and solubility.

MATERIALS AND METHODS

Sample size calculation:

As the main outcome, sorption% is used in this power analysis for a 2 x 2 x 3 factorial design. Glass ionomer type is the first variable (there are two levels), followed by pH cycling (there are two levels), and curing time (3 levels). The effect sizes (f) were 0.32, 0.4, and 0.38 for the three parameters, respectively, based on the findings of a pilot research that was done on three specimens in each subgroup. The minimum predicted sample size for each subgroup was 10 specimens using alpha () levels of 5% and beta () levels of 20%, i.e., power = 80%. Using IBM ® SPSS ® Sample Power ® Release 3.0.1, the sample size computation was carried out.

Two different traditional GICs were tested in this study (Equia Forte– EF and Fuji IX). Materials commercial name, type, manufacturer's name and composition were shown in Table (1).

Sample grouping:

The samples of each material (Fuji IX or Equia Forte) were subdivided into 3 subgroups according to the time of irradiation exposure (none, after 2 minutes, after 4 minutes) using light emitting diode curing unit (cordless LED B-woodpecker-1000mw/cm^2).

Preparation of Specimens:

60 disc-shaped specimens of the tested groups were prepared using cylindrical Teflon molds. Capsules of Fuji IX and Equia Forte glass ionomer were mixed by an Amalgamator (IMIS-M3, Macao, China) for 10 seconds respectively, according to manufacturer's instructions. To ensure proper packing of the material without sticking, the moulds were carefully positioned on glass slabs before being filled with the triturated mixture. 13 In the case of the control groups, a polyester strip was placed on top of the mould after the mixed materials had been injected, and a second glass microscope slide was used to give a consistent surface finishing and to remove the excess material. After setting, the specimen was taken out of the mold, and the excess material that had protruded around the mold's edge was carefully removed using a surgical blade. Each specimen's surface was examined to make sure there were no air bubbles or cracks.

Experimental Groups:

The specimens were divided after initial setting into one control and two other groups (n = 10).

a. In Group1, the study's control group, the specimens were mixed and then left in the mould to set without any further care.

TABLE (1) Materials commercial name, type, manufacturer's name and composition:

Materials name	Type	Manufacturer	Composition
Fuji IX GP fast	Conventional Bulk-fill glass ionomer cement	GC, Tokyo, Japan	**Powder:** 60-70% fluoro alumino silicate glass, 30–40% polyacrylic-acid, poly carboxylic acid, 40% distilled water. **Liquid:** 50 % distilled water, 40 % poly-acrylic acid, and 10% poly-basic carboxylic acid. Powder/liquid ratio: 3.6/ 1.0.
Equia Forte	Glass Hybrid filling	GC, Tokyo, Japan	**Powder:** 70-90 % strontium fluoro-alumino-silicate glass, 30 % poly-acrylic acid **Liquid:** 40% aqueous polyacrylic acid

b. In Group 2, the specimens were exposed to an LED light curing unit in the conventional curing mode for 40 seconds, two minutes after the top of the mould had been filled.

c. In Group 3, the specimens will be exposed to an LED light curing unit in the standard curing mode for 40 seconds four minutes after the top of the mould has been filled.

All Group (1), (2), and (3) samples were subjected to erosive medium twice daily for 10 minutes for a total of 14 days. A Multi-parameter Benchtop Meter inoLab® Multi 9630 IDS ionometer (WTW; Weilheim in) was used to determine the pH of the erosive fluid.

Evaluation of Solubility and Water Sorption:

The solubility and water sorption were determined by 4digit precision electronic balance (Sartorius Biopharmaceutical and Laboratories, Germany) recording the initial weight **(W1)** of samples by using a precision weighing scale. Directly after weighing, samples were then stored in individual vials containing 10 ml of (distilled water), room temperature for 1 day. Specimens were removed from their vials and immediately weighed **(W2).**

After that, the samples were dehydrated at 37°C for full 24 hours, dehydrated and then weighed again (**W3).** The above-mentioned procedure was repeated after pH cycling (total of 14 days) for same samples simulating the condition of erosive wear for specific time period. [15-16]

The water sorption potential was determined from the difference between the initial and the wet weighing **(W2 -W1).** The loss of material (SL) was obtained from the difference between the initial and final drying weight of each sample **(W1 -W3)**. The percentage of mass change or increase in weight of the specimen (WS %) is the apparent value for water sorption by the specimen, and the SL% represents the amount of material lost in the media during pH cycling.[17] The values of water sorption (WS %) and SL%, for each sample tested, were calculated using the following equations:

$$WS\% = ([W2 - W1]/W1 \times 100)$$

$$SL\% = ([W1 - W3]/W1) \times 100$$

Statistical Analysis

The difference between baseline and after pH cycling solubility and water sorption readings were calculated, tabulated and statistically analyzed for each of the control and test groups. Numerical data were explored for normality by checking the distribution of data and using tests of normality (Kolmogorov-Smirnov and Shapiro-Wilk tests). Data showed normal (parametric) distribution. Data were presented as mean and standard deviation (SD) values. Three-way ANOVA test was used to study the effect of GIC type, irradiation, pH cycling, and their interactions on mean sorption and solubility %. Bonferroni's post-hoc test was used for pair-wise comparisons when ANOVA test is significant. The significance level was set at $P \leq 0.05$. Statistical analysis was performed with IBM SPSS Statistics for Windows, Version 23.0. Armonk, NY:IBM Corp.

RESULTS

Water sorption results:

The results showed that GIC type (regardless of irradiation and pH cycling) had a statistically significant effect on mean sorption %. While in pH cycling (regardless of GIC type and irradiation) had no statistically significant effect on mean sorption %. Irradiation (regardless of GIC type and pH cycling) had a statistically significant effect on mean sorption %. The interaction between variables had a statistically significant effect on mean sorption % indicating that variables are dependent upon each other**, (Tables2,3).**

TABLE (2) Three-way ANOVA results for the effect of different variables on mean sorption %

Source of variation	Type III Sum of Squares	df	Mean Square	*F*-value	*P*-value	Effect size *(Partial eta squared)*
GIC type	11.634	1	11.634	96.226	<0.001*	0.667
pH cycling	0.133	1	0.133	1.096	0.300	0.022
Irradiation	9.356	2	4.678	38.694	<0.001*	0.617
GIC type x Curing x pH cycling interaction	2.357	2	1.179	9.748	<0.001*	0.289

df: degrees of freedom = (n-1), *: Significant at P ≤ 0.05

TABLE (3) The mean, standard deviation (SD) values and results of three-way ANOVA test for main effects of different variables on sorption %

Variable		Mean	SD	*P*-value	Effect size *(Partial eta squared)*
GIC type	**Fuji**	1.62	0.73	<0.001*	0.667
	Equia	2.5	1.04		
pH cycling	**Before**	2.01	0.82	0.300	0.022
	After	2.11	1.16		
Irradiation	**No curing**	2.58 ^{A}	1.04	<0.001*	0.617
	2 minutes	1.96 ^{B}	0.88		
	4 minutes	1.63 ^{C}	0.85		

: Significant at P ≤ 0.05, Different superscripts in the same column indicate statistically significant changes by curing

Solubility results:

The results showed that GIC type (regardless of irradiation and pH cycling) had no statistically significant effect on mean solubility %. pH cycling (regardless of GIC type and irradiation) had a statistically significant effect on mean solubility %. irradiation (regardless of GIC type and pH cycling) had a statistically significant effect on mean solubility %. The interaction between variables had a statistically significant effect on mean solubility % indicating that variables are dependent upon each other (Tables 4,5).

TABLE (4). Three-way ANOVA results for the effect of different variables on mean solubility %

Source of variation	Type III Sum of Squares	df	Mean Square	*F*-value	*P*-value	Effect size *(Partial eta squared)*
GIC type	0.139	1	0.139	1.383	0.245	0.028
pH cycling	2.356	1	2.356	23.415	<0.001*	0.328
Irradiation	15.270	2	7.635	75.874	<0.001*	0.760
GIC type x irradiation x pH cycling interaction	28.161	2	14.080	139.927	<0.001*	0.854

df: degrees of freedom = (n-1), *: Significant at P ≤ 0.05

TABLE (5) The mean, standard deviation (SD) values and results of three-way ANOVA test for main effects of different variables on solubility %

Variable		**Mean**	**SD**	***P*-value**	**Effect size *(Partial eta squared)***
GIC type	**Fuji**	1.77	1.16	0.245	0.028
	Equia	1.67	1.49		
pH cycling	**Before**	1.52	1.54	<0.001*	0.328
	After	1.92	1.06		
Irradiation	**No irradiation**	1.53 [B]	0.88	<0.001*	0.760
	2 minutes	1.23 [C]	0.64		
	4 minutes	2.41 [A]	1.86		

: Significant at $P \leq 0.05$, Different superscripts in the same column indicate statistically significant changes by curing

DISCUSSION

The present study examined the effect of radiant heat timing, and pH cycling on the water sorption and solubility behavior of two conventional GICs. These restorative materials always suffered from deteriorated mechanical properties that limited it usage to certain clinical situations as (IRM, Cements or ART) as well as limited life expectancy for the entailed restorations. [18]

On the other hand, other trials to improve the clinical performance of GIC were considered during material application and setting in order to enhance the setting reaction aiming at accelerating the maturation of the material to minimize the critical early phase of setting.

Radiant heat treatments in GIC restorations was introduced as an idea to minimize the time where GIC is highly sensitive to water contamination through enhancement of setting reaction thus the need for surface coating could be eliminated. Therefore, heat treatments may have a positive effect on life expectancy of GIC restorations. These improvements may be attributed to thermal energy transfer via the LED units which causes increased ion mobility at the early phase of setting, increased flowability and consequently, increased reactivity of the released ions of the glasses and the polyacrylic acid, accelerating the setting reaction. [15-16]

Solubility and water uptake are two important properties in the evaluation of GIC and are directly related to the success of restorations. Water sorption tests usually evaluate the total increase in weight of a specimen resulting from diffusion of the water and other small molecules .[19]

For water sorption, all of the tested groups showed water gain at the end of the immersion period in the current study. Regarding water sorption findings, irradiation for 40 seconds at 2 minutes from mixing as well as 4 minutes reduced significantly the water sorption of the tested materials, however the irradiation after 4 minutes was more effective. Therefore, both first and second null hypotheses could be rejected. Also, comparing both tested GIC materials, Equia Forte showed more water sorption compared to Fuji IX dictating rejection the third null hypothesis. On the other hand, although pH cycling slightly increased water sorption, yet the increase was not statistically significant, thus the fourth null hypothesis could be accepted.

Water sorption through polymers could be explained based on two theories; first, water diffusion through microvoids regardless of the polarity of the molecules within (free volumetric theory). Second is the water diffusion derived by the hydrophilicity of the existing groups in order to bind thereafter (interaction theory).

In the early phase of setting reaction of GICs, the diffused water allows the transportation of calcium and aluminum ions to start the reaction with the polyalkenoic acid. However, over time, excessive water sorption may end up in disintegration and solubility of the material, deteriorating its physical properties. In the present study, the water sorption and solubility were measured after both immersion times of the specimens for 24 hrs. in neutral pH, then after 14 days in a pH cycling regimen, since the highest water gain values usually occurred in the first 7 days of most materials.[10-12-19]

Concerning the effect of irradiation on water sorption, thermal heat energy delivered by LED unit to the surfaces of the GIC specimens leads to the thermocatalytic increase in ion mobility during the early phase of setting as previously described, leading to the acceleration of the setting reaction. This finding agrees with previous studies [7-11-16]

Regarding the time of irradiation, it was found that irradiation after 4 minutes from the start of setting produced better water sorption results. No previous studies in the literature tackled this point, however, further studies are needed to study the effect of irradiation time on the surface and structural as well as the chemical characteristics of GICs.

In the current study, EQUIA Forte showed significantly higher water sorption compared to Fuji IX. The variability in water absorption values of different GICs could be attributed to different compositions, filler particle size and distribution, method of mixing as well as the powder/liquid proportioning. Moreover, Equia Forte Fil was introduced to the market as a bulk fil, fluoride releasing, glass hybrid restorative system to be used in stress bearing areas in conjunction with its corresponding light cure resin coat whereas, Fuji IX did not require the use of a protective resin coating. This might explain the difference in the sorption values between both materials where, during the maturation phase of EQUIA Forte, the resin coat could positively fill the microvoids in the surface of the GIC specimens, thus reducing the active sites for water sorption. In the current study, no coat was used to study the direct effect of irradiation on the GIC. [20]

Moreover, In one previous study crack lines were also observed in many specimens of the different groups, and SEM for Equia Forte showed more and larger surface cracks compared to other tested materials which might be partly responsible for the higher water sorption values. [15-16]

In the current study the effect of pH cycling on water sorption was statistically insignificant. A previous study by Lima et al in 2018 reported on lack of influence of the storage medium pH on the water sorption of 5 different GICs used for ART.[21] Culina et al 2022, confirmed the influence of pH changes on water sorption (14) In the literature, the great variety of results is due to methodological and composition differences of the test storage solutions.[22]

For solubility, all of the tested groups showed weight loss at the end of the immersion period in the current study. Regarding solubility findings, irradiation for 40 seconds at 2 as well as 4 minutes from mixing affected significantly the water sorption of the tested materials, however the irradiation after 4 minutes increased solubility, whereas irradiation at 2 minutes reduced it. Therefore, it is considered that both first and second null hypotheses could be rejected. On the contrary, comparing both tested GIC materials, both Equia Forte and Fuji IX showed almost similar solubility pattern, thus the third null hypothesis could be accepted.[23] On the other hand, pH cycling increased solubility and the increase was statistically significant, thus the fourth null hypothesis could be rejected.

Solubility is the ability of a substance to dissolve in another substance, expressed as the concentration of saturated solution of a solvent in a dissolvent.[24] For water solubility, all of the tested groups showed positive mean values. Positive solubility values could be attributed to loss of these materials,. In the current study, irradiation of GICs by LED curing is supposed to accelerate the acid-base reaction, decreasing the amount of water uptake therefore, on dehydration the loosely trapped water could be easily vaporized, leading to mass loss of the tested materials.[28]

The positive effect of irradiation on solubility of both tested materials could be explained on the acceleration of the material maturation as previously mentioned however the detrimental effect of irradiation at 4 minutes opposite to water sorption could be explained on basis of previous findings that showed surface cracks on the heat treated GIC specimens, these cracks could have increased the surface area available for ion elution from the material.[29] Also, it may increase the surface area available for water loss during the dehydration period for solubility assessment. This finding would postulate that irradiation during the first 2 minutes from the setting reaction might be safer concerning the solubility of GIC compared to after 4 minutes where heating the early formed matrix might have created surface cracks as heating might cause dehydration of material hindering the formation of carboxylic bonds as shown in previous studies.[16]

Concerning the solubility of the tested GIC material, both materials showed similar results opposite to the water sorption findings corroborating the results of additional studies. Lima et al 2018, reported no relationship considering the type of cements, when evaluating the water sorption and the solubility properties.[21] However, previous studies showed differences in the solubility values among conventional GICs. The differences may be related to the different compositions of the materials. The reasons for differences between the results likely reflect changes in the chemistry of the particles as well as the setting times of the tested materials. [29]

Concerning the increased solubility after pH cycling confirming the results of previous studies.[30] No relation could be established between water-sorption behavior and water solubility, neither they indicate influence over each other. This finding is in contradiction to the water sorption which was not affected by the pH cycling. [8,14,] The increased mass loss detected at the end of the storage period might be attributed to different explanations. First, this loss could be considered as acidic erosion of the material which was proved to increase by increasing the acidity of the storage medium (5% acetic acid). Second, the increased storage time during pH cycling that extended for 14 days compared to a 24 hr storage before pH cycling, where elongating the time of storage resulted in higher solubility values in previous studies.[18,21]

The results of the current study demonstrated that irradiation of conventional GIC using LED curing unit improved the water sorption and solubility of the tested materials and increased its resistance to water sorption, however, solubility did not follow the same pattern. Further studies on the effect of the tested variables on the surface, structural and chemical changes of restoratives can be beneficial in understanding the cause-effect relationship of the tested properties,

CONCLUSIONS

Under the limitations of the current study, the following conclusions could be derived:

1. Irradiation of conventional GIC material during setting could improve the water sorption and solubility
2. The timing of irradiation affected the outcome regarding the tested properties, where the irradiation during the early setting (2 minutes) could be a safer option
3. Water sorption is material dependent rather than pH dependent
4. Solubility is pH dependent rather than material dependent

Clinical significance

One curing cycle using LED might be a simple method that can improve the physical properties of conventional GICs suggesting an improved longevity of restorations.

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