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# Evaluation of Surface Hardness and Color Stability of Two Different Glass Ionomer Cements After Treatment with Calcium Chloride: An In-Vitro Study

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## ABSTRACT

**Background:** Glass ionomer cements (GICs) is one of the materials of choice for restoring carious teeth in children due to its good qualities. Some modifications were made on GIC to improve its properties such as calcium chloride ($CaCl_2$) solution application.

**Objective:** The purpose of this study was to evaluate the effect of 42.7 weight percent (wt%) $CaCl_2$ solution on two GIC restorations in relevance to color stability and surface hardness.

**Methods:** Forty GIC specimens were prepared for each of the two tests and grouped according to the type of GIC into Fuji II light cure (Fuji II) group (n=20) and Fuji IX group (n=20). Each group was further subdivided into two subgroups: control (n=10) and experimental (n=10). Control specimens were left to set without treatment for five minutes. Experimental specimens were left to set for five minutes and then each specimen was immersed in ten milliliters (ml) of 42.7 wt% $CaCl_2$ solution for 60 seconds.

**Results:** Fuji II group showed significant change in color after seven days while Fuji IX group showed no change in color at different time intervals. For surface hardness test, Fuji IX showed increase in surface hardness while Fuji II showed no change in surface hardness.

**Conclusion:** Treatment of GIC with $CaCl_2$ solution does not affect the color stability of Fuji IX GIC but it might affect that of Fuji II GIC. $CaCl_2$ application on GIC improved the surface hardness of Fuji IX GIC however surface hardness of Fuji II had not changed.

**Keywords:** Calcium chloride solution, Glass ionomer cements, Color Stability, Surface hardness.

## INTRODUCTION

GICs are widely used restorative materials in dentistry especially in pediatric dentistry. GIC has some advantageous properties, it has similar thermal expansion coefficient to that of dentin [1], chemical adhesive properties [2], and fluoride release property [3]. In spite of the advantageous properties of GIC, it has some drawbacks. GICs has early water sensitivity and has slow setting process, which affects and delay its final strength [4].

Some modifications were made on GIC composition to improve its properties such as laser system [5] and radiant heat transfer, or ultrasonic energy application on restoration surface [6]. Calcium Chloride application is one of the modifications that was made on GIC. It has been found that application of $CaCl_2$ on GIC improve its setting reaction, its physical and its mechanical properties [7].

Finally, there is considerable support in literature that favors using $CaCl_2$ solution for improving some mechanical properties but there were no studies done to test its effect on color stability. Accordingly, this study was formulated to test the effect of $CaCl_2$ solution on two types of GIC; a high viscous GIC (Fuji IX) and a resin modified GIC (Fuji II). Regarding surface hardness and color stability since there was no adequate research conducted in this sector upon reviewing literature.

The purpose of this study was to evaluate the effect of 42.7 weight percent (wt%) $CaCl_2$ solution on two GIC restorations in relevance to color stability and surface hardness.

## MATERIALS AND METHODS

Two types of GIC were used in this study: Fuji II LC capsule (GC Corp., Tokyo, Japan) and Fuji IX GP capsule (GC Corp., Tokyo, Japan). Calcium Chloride salt ($CaCl_2$; Calcium Chloride Anhydrous, Alpha Chemika, India, batch no.cc062) will be dissolved in deionized water at concentrations of 42.7 wt%. All materials were manipulated according to manufacturer instructions.

### Sample size:

A power analysis was designed to have adequate power to apply a two-sided statistical test of the null hypothesis that there is no difference would be found between tested groups. By adopting an alpha (α) level of 0.05, a beta (β) of 0.2 (i.e. power=80%), and an effect size (d) of 1.32 calculated based on the results of a previous study [8]. The total sample size was 80 samples: 40 for Fuji IX group and 40 for Fuji II LC group. The 40 disks for each type of GIC were further divided into 20 disks for each of the two tests. Each group was further subdivided into two subgroups: control subgroup (n=10) and experimental subgroup (n=10). Sample size calculation was performed using G*Power version 3.1.9.7

### Steps for disk shaped specimens' preparation for both groups:

For preparation of disk-shaped specimen measuring a specially designed custom-made split Teflon mold was fabricated with 7 mm diameter and 2



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mm thickness with an external metallic ring. The assembled split Teflon mold within the metallic ring were placed over a polyester strip (0.05 mm thickness) on a glass slab [8].

Each capsule was activated & was put into a rotating mixer for ten seconds. The mixed capsule was loaded onto the applicator and the material was dispensed slowly and directly into the mold. After filling the mold with GIC, the material was then covered by a polyester strip, then a glass slab was placed over it and pressed to provide a flat smooth surface and to remove excess material [8].

Then for Fuji IX group only, the specimens were left to set in the mold for five minutes under load of 500 gram, but for Fuji II LC group, each specimen was light cured with a halogen light-curing device according to manufacturer instructions for 20 seconds on each side of the specimen. After setting of both groups, the upper glass slab and polyester strip were removed. The metal ring was disassembled from the Teflon mold, then the GIC disks were removed from the split Teflon mold [8].

**For control specimens** (Without treatment with $CaCl_2$ solution)**:** Each specimen was immersed in ten ml deionized water individually in a plastic container for 24 hours for completion of GIC setting.

**For experimental specimens** ( Treatment with $CaCl_2$ solution)**:** Each specimen was immersed individually in ten ml of 42.7 wt % $CaCl_2$ solution for one minute then was rinsed with five ml de-ionized water. The specimens were then placed separately in plastic containers containing ten ml deionized water for 24 hours for the completion of setting of GIC [8].

***1) Color stability test:***

The disk shaped specimens were prepared as mentioned before for each type of GIC. The color of all specimen groups were measured using a spectrophotometer using CIE (Commission International de l'Éclairage) *L*a*b**( L* refers to the brightness, a* for redness to greenness, and b* for yellowness to blueness) for the time period of 24 hours, 7 days and 1 month for each specimen. The color change (ΔE) between time intervals was calculated using the equation: $\Delta E = [(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2]^{1/2}$ [9].

***2) Surface hardness test:***

The disk shaped specimens were prepared as mentioned before for each type of GIC. At 24 hours, the specimens were removed from the solution & dried before being measured for surface hardness test. Surface hardness was measured using Vickers hardness tester. A 200 gm load was applied to each specimen for 10 seconds and 5 indentations were made on the top only of each specimen, one in the centre and one in every quarter, the average of the readings was recorded & data were reported in Vicker Hardness Numbers (VHN) [8].

**Ethical approval:**

**This study does not contain any human participants or animals. This study took an exemption from the Scientific Research Ethics Committee, Ain Shams University, (81) 23-5-2018 with number (FDASU-Rec E051816). This work has been carried out in accordance with the code of Ethics of the World Medical Association (Declaration of Helsinki) for studies involving humans.**

***Statistical analysis***

Numerical data were presented as mean and standard deviation (SD). They were explored for normality by checking the data distribution, calculating the mean and median values and using Kolmogorov-Smirnov and Shapiro-Wilk tests. The repeated measures ANOVA followed by bonferroni post hoc test was used for intragroup comparisons. The significance level was set at $p \leq 0.05$. Statistical analysis was performed with IBM® SPSS® Statistics Version 26 for Windows.

## RESULTS

### I-Color stability test

Mean ± standard deviation (SD) values of color stability at different times were presented in table (1). As for Fuji II group, there was a statistical significant difference between values measured at different intervals (p=**0.047**). The highest value was measured after 7 days (2.424±0.517) followed by value measured after 24 hours (1.69± 0.315) while the lowest value was measured after 30 days (1.558±0.265). Post hoc pairwise comparisons showed that the value measured after 7 days tends to be significantly higher than values at other intervals (p<0.05). As regards Fuji IX group, there was no statistical significant difference between values measured at different intervals. The highest value was measured after 7 days (1.54±0.311) followed by value measured after 30 days (1.703±0.324) while the lowest value was measured after 24 hours (1.54±0.311). Post hoc pairwise comparisons showed no statistical significant difference between any of the measured intervals (Table 1).

**Table (1):** Mean ± standard deviation (SD) values of color change at different time intervals

| Difference points | Color change (mean±SD) | | | p-value |
|---|---|---|---|---|
| | **24 hours** | **7 days** | **30 days** | |
| **Fuji II** | 1.69± $0.315^B$ | 2.424± $0.517^A$ | 1.558± $0.265^B$ | **0.047** |
| **Fuji IX** | 1.54± $0.311^A$ | 1.85± $0.336^A$ | 1.703± $0.324^A$ | **0.1189ns** |

Means with different superscript letters within the same horizontal row are statistically significantly different (p<0.05), ns; non-significant (p>0.05).

**II- Surface hardn ess test**

Mean ± standard deviation (SD) values of surface hardness in different subgroups were presented in table (2). As for Fuji II group, there was no statistical significant difference between both subgroups (p=0.061). Concerning Fuji IX group, the experimental subgroup (21.38±1.52) had a statistically significant higher value than the control subgroup (19.02±1.61) (p=0.003).

**Table (2):** Mean ± standard deviation (SD) values of surface hardness in different groups

| Subgroup | Surface hardness (mean±SD) | |
|---|---|---|
| | Fuji II | Fuji IX |
| **Control** | $39.50\pm1.20^{A}$ | $19.02\pm1.61^{B}$ |
| **Experimental** | $38.28\pm1.50^{A}$ | $21.38\pm1.52^{A}$ |
| **p-value** | **0.061ns** | **0.003*** |

Means with different superscript uppercase letters within the same vertical row for each group are statistically significantly different, ns; non-significant (p>0.05)*; significant (p<0.05).

## DISCUSSION

Dental caries is a chronic disease affecting large population. Different filling materials have been introduced in dentistry for restoring the carious teeth such as composite resin filling and GIC. Composite resin has good esthetic appearance and mechanical properties, but it is technique-sensitive especially in uncooperative children, and in cases where moisture control is critical [10].

Considering the advantages of GIC it may be advantageous for restoring carious teeth especially in children. GICs are widely used in restorative dentistry due to its adhesive property and anticariogenic effect owing to its fluoride release [11, 3].

$CaCl_2$ solution application on GIC during initial setting is a recent technique to enhance some properties of GIC through improving its acid base reaction [8]. $CaCl_2$ is a highly soluble salt in water. In the current study, we evaluated the effects of 42.7 wt% $CaCl_2$ solution on surface hardness & color stability of two types of GICs. This concentration (42.7 wt% $CaCl_2$) was selected upon a previous study conducted by **Shiozawa *et al.*** [12] who stated that this concentration is the saturated concentration of $CaCl_2$ salt.

Two types of GIC were used in this study Fuji IX ( conventional GIC) & Fuji II ( resin modified GIC), which are commonly used restorative materials, to test the effect of $CaCl_2$ solution on their mechanical & physical properties They are used due to their fluoride release property, biocompatibility, self-adhesive property [13, 3].

Color stability is an important property for aesthetic restorative materials. Failure or success of any restoration depends on color match & color stability of aesthetic restorations. Spectrophotometer was selected to measure the color change as it is accurate & reliable technique in measuring color stability of dental aesthetic materials. The CIE lab system was chosen because it is well suited to measure small color differences [9].

Regarding the effect of $CaCl_2$ solution on color stability of GIC, there was no abundant data in literature. Fuji IX showed non-significant change in color after treatment with $CaCl_2$ solution at different time intervals (24 hours, 7 days & 30 days), which revealed that $CaCl_2$ solution did not affect the color stability of Fuji IX. This result might be due to the effect of $CaCl_2$ solution, which improved the acid-base reaction & accordingly, prevented the water sorption of GIC due to the decrease in the porosity during the setting of GIC. This improvement in the acid-base reaction can be explained by the progression of polycarboxylate network formation as a result of absorption of Calcium ions, which create a chemical bond with the remaining unreacted carboxylic acid in GIC matrix. This reaction strengthen the polyacid salt matrix and so improves properties of GIC [12].

On the other hand, Fuji II showed significant change in color after 7 days. This result may be due to presence of HEMA (Hydroxyethyl methacrylate) in resin modified GICs (RMGIC) which increases water sorption and so increases the potential for staining [14].

This result might also be explained by the fact that RMGIC sets by a dual reaction: polymerization reaction & acid-base reaction. In RMGIC the acid-base reaction is slower and is retarded, which may affect or delay the effect of $CaCl_2$ solution on acid-base reaction [15, 16]. The delayed and slower acid base reaction happens in RMGIC as a result of the decreased amount of water in RMGIC where the water is required to initiate the acid base reaction while higher amount of monomer is included in RMGIC, which is required for photopolymerization. The delayed and slower acid base reaction also happens in RMGIC due to decreased polyacid levels in RMGIC [17], and it might have delayed the effect of $CaCl_2$ solution which might in turn have affected the color stability of RMGIC. Additionally, photo polymerization of resin during polymerization in RMGIC might also have affected the color stability in RMGIC [14].

Surface hardness is one of the mechanical properties, which is used to predict the wear resistance of GIC & determine its deformation degree [18]. It is accepted as an important valuable parameter for comparing material such as GIC with tooth structure. Vicker hardness test method was used in the current study due to its ability to determine hardness of small areas & brittle materials such as GIC restorative materials. Disk shaped design specimens were selected in this study owing to their suitability in design for the desired tests [19].

As for the results of surface hardness of GIC after treatment with $CaCl_2$ solution, Fuji IX GIC showed significant increase in surface hardness. This might be explained by the progression of polycarboxylate network formation as a result of absorption of Calcium ions, which create a chemical bond with the remained unreacted carboxylic acid in GIC matrix. This reaction strengthen the polyacid salt matrix & so increase surface hardness of GIC [12]. This result comes in agreement with **Dionysopoulos *et al.*** [8] **and Shiozawa *et al.*** [12] who reported that the surface hardness of conventional GIC has increased after treatment with $CaCl_2$ solution.

On the other hand Fuji II showed insignificant increase in the surface hardness after treatment with $CaCl_2$ solution. This result may be due to the delayed acid-base reaction, which delays the effect of $CaCl_2$ solution on its acid-base reaction [15, 16]. Another explanation might be due to the less permeability & porosity of RMGIC in comparison with conventional GIC and thus it absorbs less calcium ions, hence it did not affect its surface hardness [21].

## CONCLUSION

Based on the findings of this study, it was concluded that treatment of GIC with $CaCl_2$ solution does not affect the color stability of Fuji IX but it might affect color stability of RMGIC. Also, it has been shown to improve the surface hardness of Fuji IX however the surface hardness of Fuji II had not changed.

## RECOMMENDATION

This study was conducted in vitro; considering the limited information of laboratory tests, which is not necessarily similar to clinical condition. We recommend using $CaCl_2$ solution as a mouthrinse or as a gel or foam to interact more with restoration surface in vivo for enhancing its physical and mechanical properties.

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