

The Effect of Using CaCl₂ Gel on Water Sorption and Solubility of Conventional Glass Ionomer (In-vitro Study)

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ABSTRACT

Background: Glass ionomer cements (GIC) offer unique properties and clinical uses, making them clinically attractive restorative materials. However, these materials still have limitations due to their lower mechanical and physical properties. Calcium chloride (CaCl₂) solution has been used as a method of improving water sorption and solubility properties of GIC. A new form of CaCl₂ was suggested by different authors to be more user friendly. Water sorption and solubility were measured at baseline and after the pH-cycling protocol. **Aim of the study:** to evaluate the effect of using CaCl₂ in solution and gel forms on water sorption and solubility of the GIC at baseline and after pH cycling. **Materials and Methods:** Thirty disc-shaped Fuji IX GIC specimens were created and divided into three groups as follows: (n=10); group 1 (control), group 2 (CaCl₂ solution), and group 3 (CaCl₂ in gel form). Specimens were subjected to pH cycling twice daily with 5% acetic acid (pH=3), rinsed, dried, and kept in distilled water. This cycle was performed 14 times. Water sorption and solubility were measured at baseline and after pH-cycling. **Results:** The control group showed the highest mean values at baseline and after pH-cycling. Insignificant differences between both CaCl₂ groups were seen. Significant increase in water sorption and solubility values was seen after pH-cycling in all groups compared to baseline. **Conclusions:** CaCl₂ in both forms decreased water sorption and solubility of GICs. However, CaCl₂ gel was more user-friendly. After pH cycling, all values of water sorption and solubility were diminished.

Keywords: Calcium chloride, Glass ionomer cement, pH-cycling, Solubility, Water sorption.

INTRODUCTION

Glass-ionomer cements (GICs) provide unique beneficial properties and clinical uses within the field of restorative dentistry, making them clinically attractive restorative

material.^{1,2} They offer different advantages: good tissue biocompatibility and anticariogenic effect by providing fluoride ion release and recharging ability.³⁻⁶

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However, these materials still have limitations; their lower mechanical and physical properties compared to resin composite materials hindered their use in stress-bearing areas.⁷

One of the most crucial factors determining the longevity of restorative materials is their resistance to disintegration or dissolution in the oral cavity.⁸ Solubility generates a loss of material mass. Which in turn negatively affects its mechanical properties. In addition, it produces tooth/restoration interface failure, increasing the risk of marginal microleakage and debonding. This will eventually lead to the development of dental caries and restoration failure.^{8,9} Whereas water sorption changes the mechanical properties through degradation and lamination.⁹

Conventional GICs present higher solubility and water sorption. This is mainly related to the nature of their composition and setting reaction.¹⁰⁻¹³ Aiming to overcome this main disadvantage, Dionysopoulos et al recommended the use of calcium chloride (CaCl₂) solution, as a chemo-catalytic technique to improve and accelerate the maturation process of GICs.^{14,16} It was proposed that this technique enhanced "cross-linking of the carboxylate groups of polyalkenoic acids with the Al³⁺ and Ca²⁺

ions released from glass particles is the main setting reaction of GICs".¹⁴⁻¹⁷

The above-mentioned method allows for reducing procedure time and faster achievement of initial mechanical strength. However, Calcium chloride used in the previous studies was in the form of a solution, which does not make it stable during handling.^{16,18} Thus, it was thought that using it in a gel form will allow its use in a more friendly way, which in turn will allow its clinical recommendation in different clinical situations.

Therefore, the aim of this study is to evaluate and compare the water sorption and solubility of conventional Fuji IX GIC using calcium chloride solution and gel forms at baseline and after pH cycling.

The null hypothesis to be tested: there will be no difference in the solubility and water sorption properties of conventional GI either used with no surface treatment or treated with calcium chloride solution and calcium chloride gel.

MATERIALS AND METHODS

Materials:

1-A Conventional GIC material (Fuji IX), A3.5 shade (GC Fuji IX GP®, GC, America).

2- Calcium chloride solution: A freshly made chemical solution using the following

formula (42.7 % CaCl_2 by weight),¹⁶ was used and prepared in the inorganic chemistry lab, Chemistry Department, Faculty of Science, Suez Canal University, Cairo, Egypt.

3- Calcium chloride gel form:

Carbopol 940 polymer was dissolved in 50 mL of distilled water. Gelatin powder was dissolved in another 50 mL of warm distilled water containing anhydrous CaCl_2 before being mixed with CaCl_2 for 2 hours. The completed gel was then stored in carefully sealed syringes in the refrigerator until utilized. (The gel was prepared in the inorganic chemistry lab, Chemistry Department, Faculty of Science, Suez Canal University, Cairo, Egypt).

Specimen Preparation:

Thirty discs were prepared using a split Teflon mold with a central hole of 6 mm diameter and 2mm thickness.¹⁹ GC Fuji IX GP®, Capsules were triturated using an amalgamator¹ for 10 s, as recommended by the manufacturer, with high speed. The material was loaded and immediately injected into the mold using an applicator.² A polyester strip 0.05 mm thick³ was placed onto a glass slab before mixing the cement for

the mold to be placed over it. A second polyester strip was used on top of the mold with another glass slab to maintain a standardized surface, finishing with excess cement removal. After setting, specimens were removed from the mold, and a surgical blade was used carefully to remove excess material around the edge of the mold. Examination of the specimen surface was done using an optical microscope ($\times 10$ magnification)⁴ to confirm that no air bubbles or cracks were contained.¹⁶

Specimens Grouping and Treatment:

The thirty prepared specimens were distributed randomly into three groups after mixing. Group 1 (control): specimens were left to set in the mold without any treatment. Group 2: Calcium Chloride (CaCl_2) solution, 42.7 weight % solution was applied on the top surface of the specimens for 2 minutes, then rinsed with 5 ml of deionized water. Group 3: CaCl_2 in gel was applied to the specimens' surface and left for 2 minutes then rinsed with 5ml deionized water. Subsequently, specimens were stored in plastic containers containing deionized water at 37 ± 1 °C for 24 h to complete the greatest part of GIC setting reaction.¹⁶

¹ IMIX-M3, Macao, China.

² 3M ESPE, USA.

³ TOR-VM Celluloid Transparent strip.

⁴ 10X Clinic Stereo Optical Microscope Low Magnification Microscopes A22.1204.

Evaluation of Water Sorption and Solubility:

The specimens were weighed using an electronic balance with four digits precision,⁵ with accuracy up to 0.01 mg. The specimens' initial mass is reported as dry weight or original weight (W1). The specimens were then immersed in 10 mL of distilled water at 37°C ± 1°C in separate containers in an incubator. After 24 hours, samples were withdrawn from distilled water, blot-dried with filter paper, then waved in air for 15 seconds to eliminate any visible moisture. The final weight was taken 1 minute after being removed from the water (W2). The samples were transferred into a glass desiccator containing dehydrated silica gel,⁶ maintained at 37°C ± 1°C and stored for another 24 hrs. The specimens were weighed until constant weights were attained (W3).

Water Sorption and Solubility calculation:^{16,20}

The volume of specimens was calculated using the following equation:

$$V = \pi \times r^2 \times h$$

Where V is volume of the specimen, $\pi = 3.14$, r is the radius, and h is the thickness of the specimen. The volume was equal =

56.52 mm².

Water Sorption (WSP) was calculated using the following equation:

$$WSP = (w_2 - w_3)/V$$

Water solubility (WSL) was calculated using the following equation:

$$WSL = (w_1 - w_3)/V$$

Where, w1= original weight. w2= weight gain, w3= weight after desiccation, V= volume of specimen. Units were converted to µg/mm³ for statistical analysis purposes.

pH cycling Protocol:

The specimens were exposed to erosive media twice a day for 10 min. for 14 days.²¹ The pH of the erosive media was determined using pH Meter.⁷ Acidity measurements were performed once and then rinsed. The mean pH of Acetic acid was 3.0. After each erosive pH cycle, the samples were washed and stored in distilled water at 37°C until the next erosive pH cycle.¹⁹

Statistical Analysis:

Data presented as mean and standard deviation (SD). Data was explored for normality using Kolmogorov-Smirnov tests. Since the data was not normally distributed, the Kruskal Wallis test was used to compare

⁵ Sartorius, Biopharmaceutical and Laboratories, Ger.

⁶ Fischer Scientific, Leicester, UK.

⁷ Waterproof pH Meter and Temperature Tester Adwa (AD11).

between tested groups followed by a pairwise comparison with the Steel-Dwass test. For comparison between baseline and pH cycle, Wilcoxon signed rank test was used. The significance level was set at $P \leq 0.05$.

Statistical analysis was performed with IBM SPSS Statistics for Windows, Version 26.0. Armonk, NY: IBM Corp.

RESULTS (Table 1 & 2)

There were statistically significant differences between the tested groups. When compared to the other two examined groups, the control group had the highest mean values. For water sorption values, at baseline, a significant difference resulted between all tested groups at $p < 0.001$. The control group showed the highest significant water sorption values compared to Calcium Chloride (CaCl₂) solution, Calcium Chloride (CaCl₂) gel. CaCl₂ solution and CaCl₂ gel showed an insignificant difference in water sorption values. After pH Cycle, a significant decrease in water sorption values for the control group

only at $p=0.005$. Insignificant change in CaCl₂ gel and CaCl₂ solution after pH cycle.

For water solubility values, at the Baseline, the control group showed the highest water solubility values with significant differences from all other groups. An insignificant difference resulted between CaCl₂ solution and CaCl₂ gel. After the pH Cycling, the control group showed the highest significant water solubility values compared to all other groups. Also, a significant increase in water solubility values resulted for the control group and CaCl₂ solution only.

DISCUSSION

GICs have the beneficial feature and the ability to change their biological and physical properties easily by adjusting the powder-to-liquid ratio or by being treated with different methods.^{7,16,17,20} Regarding water sorption and solubility, they are critical features to consider when assessing bonding materials since these factors are related to the

Table 1: Descriptive statistics of water sorption values ($\mu\text{g}/\text{mm}^3$) for different tested groups and statistical comparison between these groups.

	Water Sorption Values										p-value
	Baseline					pH Cycle					
	Mean	SD	Median	Min	Max	Mean	SD	Median	Min	Max	
Control	83.2	4.0	83.2 ^a	76.1	90.2	88.9	11.8	92.0 ^a	65.5	100.8	0.005*
CaCl ₂ _Gel	58.8	12.4	61.9 ^b	38.9	72.5	65.6	13.6	63.7 ^b	44.2	86.7	0.256 NS
CaCl ₂ _Sol	60.7	17.2	61.9 ^b	35.4	88.5	66.5	11.5	61.9 ^b	53.1	90.2	0.436 NS
p-value	<0.0001*					<0.0001*					

*Different letters within each column indicate significant difference. NS= Non-significant, *: Significant at $P \leq 0.05$.*

cement durability and the longevity of restorative materials.⁷ They may result in degradation of the cement, causing restoration debonding, recurrent caries, pulpal inflammation, post-operative hypersensitivity and periodontal disease also may occur.²²⁻²⁴

depends on its hydrophilicity and porosity”¹⁴⁻¹⁷.

A new formulation of calcium chloride was suggested in previous studies in order to improve its stability during handling. Therefore, a gel form of calcium chloride was used in this present study with (42.7 weight

Table 2: Descriptive statistics of water solubility values (µg/mm³) for different tested groups and statistical comparison between these groups.

	Water Solubility Values										p-value
	Baseline					pH Cycle					
	Mean	SD	Median	Min	Max	Mean	SD	Median	Min	Max	
Control	30.0	6.8	30.1 ^a	17.7	42.5	55.4	11.0	60.2 ^a	40.7	72.5	0.001*
CaCl ₂ _Gel	14.0	12.1	13.3 ^{bc}	-3.5	33.6	23.0	10.4	24.8 ^b	8.8	37.2	0.083 ^{NS}
CaCl ₂ _Sol	11.0	9.3	7.1 ^c	0.0	28.3	26.5	14.8	24.8 ^b	3.5	51.3	0.002*
p-value	<0.0001*					<0.0001*					

*Different letters within each column indicate significant difference. NS= Non-significant, *: Significant at P ≤ 0.05.*

The use of chemocatalyzing techniques was introduced in previous studies to improve the physical and mechanical characteristics of glass ionomer cement material using calcium chloride solution and showed positive results.^{14,16,17} This was explained “through the cross-linking of the carboxylate groups of polyalkenoic acids with the Al₃₊ and Ca₂₊ ions released from glass particles is the main setting reaction of GICs. Aqueous solutions of divalent metal salts, such as CaCl₂, may inhibit GIC water uptake due to the decreased porosity of the material surface attributed to an enhanced acid–base reaction. It has been postulated that the amount of water sorption of a GIC

%) concentration and was used for 2 minutes). As this treatment using calcium chloride in a gel form to be more user friendly has never been properly investigated before.

Therefore, the aim of this study is to evaluate and compare the water sorption and solubility of conventional Fuji IX GIC using calcium chloride solution and calcium chloride in gel form at baseline and after pH cycling.

The results of the recent study showed that there was no statistically significant difference between both calcium chloride solution and gel form in water sorption and solubility values, but they were significantly lower values than the control group. This

could be attributed to the equal concentration of used forms; however, easier handling was achieved with Calcium Chloride gel form. Despite the fact that the effect of the calcium chloride gel has never been discussed before, it was recommended by Dionysopoulos et al.¹⁶

After pH cycling, all treatments showed high solubility values. Also, there was a significant increase in water solubility values in the control group and CaCl₂ solution only. The significant increase in solubility values resulting from calcium chloride solution compared to the gel form might denote the relative compatibility between the surface tension of the gel and the surface energy of the GIC. This, in turn, allowed greater adherence between both substrates which allowed the substantive existence of Ca cations that augmented the chemo catalytic effect to a level that allowed its sustainability after pH cycling. The results were in agreement with Labban et al (2021),²⁵ which explained that there is no relation between water sorption and solubility.

The current study also found that pH cycling had a significant negative effect on the water sorption and solubility of all test and control groups. All groups showed an increase in water sorption and solubility values of GIC. However, the control group h-

ad higher values compared to the other groups.

As a result, the null hypothesis was rejected since the results revealed differences in applying CaCl₂ solution and CaCl₂ gel at baseline, as unfavorable impacts were seen on the water sorption and solubility properties of the three groups after the pH cycling protocol.

Despite the fact that the two forms of calcium chloride might enhance the water sorption and solubility properties of conventional GIC, this enhancement might break down under different oral conditions. More research and further studies should be carried out to elaborate on the significant effect of using Calcium Chloride gel in the clinical setting and to improve the physical properties of GIC as it is still considered one of the best-performing restorative materials.

CONCLUSION

1. Chemo catalysis using Calcium Chloride in both solution and gel forms showed positive results.

2. The use of Calcium Chloride in gel form is a user-friendly method that may interact more with the surface of the restoration in clinical practice.

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CONFLICTS OF INTEREST

The authors claim that they have no conflicts of interest.

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