# VOLUMETRIC DIMENSIONAL CHANGES

# OF LUTING CEMENTS

by

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# TABLE OF CONTENTS

| | |
|---|---|
| Introduction…………………………………………………………………………… | 01 |
| Review of Literature………………………………………………………………….. | 05 |
| Methods and Materials……………………………………………………………….. | 14 |
| Results…………………………………………………………………………………… | 19 |
| Figures and Tables……………………………………………………………………. | 22 |
| Discussion……………………………………………………………………………. | 45 |
| Summary and Conclusions…………………………………………………………… | 51 |
| References……………………………………………………………………………. | 53 |
| Appendix………………………………………………………………………………… | 59 |
| Abstract……………………………………………………………………………….<br>Curriculum Vitae | 69 |

# LIST OF ILLUSTRATIONS

| | | |
|---|---|---|
| TABLE I | Manufacturer’s instructions | 23 |
| TABLE II | Summary Statistics for Volumetric Dimensional Changes | 24 |
| TABLE III | Summary Statistics for $Ca^{+2}$ Concentration | 26 |
| TABLE IV | Summary Statistics for $OH^-$ Concentration | 26 |
| FIGURE I | Mean Luting Agent Volumetric Dimensional Changes - Stored in Water at $22^{o}$ C | 27 |
| FIGURE II | Mean Luting Agent Volumetric Dimensional Changes - Stored in Water at $37^{o}$ C | 28 |
| FIGURE III | Mean Luting Agent Volumetric Dimensional Changes - Stored in Silicone oil at $37^{o}$ C | 29 |
| FIGURE IV | Mean Luting Agent Volumetric Dimensional Changes - Stored in Silicone oil at $22^{o}$ C | 30 |
| FIGURE V | Mean Ceramir Volumetric Dimensional Changes - Stored in Water | 31 |
| FIGURE VI | Mean Ceramir Volumetric Dimensional Changes - Stored in Silicone oil | 31 |
| FIGURE VII | Mean Fuji plus Volumetric Dimensional Changes - Stored in Water | 32 |
| FIGURE VIII | Mean Fuji plus Volumetric Dimensional Changes - Stored in Silicone oil | 32 |
| FIGURE IX | Mean Volumetric Dimensional Changes of Rely X Unicem Stored in Water | 33 |
| FIGURE X | Mean volumetric Dimensional Changes of Rely X Unicem Stored in Silicone Oil | 33 |
| FIGURE XI | Mean Volumetric Dimensional Changes of Rely X Ultimate Stored in Water | 34 |
| FIGURE XII | Mean Volumetric Dimensional Changes of Rely X Ultimate Stored in Silicone Oil | 34 |
| FIGURE XIII | $Ca^{2+}$ ions release during the storage time | 35 |
| FIGURE XIV | $OH^-$ ions release during the Storage time | 36 |
| FIGURE XV | Ceramir C&B (CM) (Doxa Dental AB, Uppsala, Sweden), | 37 |
| FIGURE XVI | Resin-modified GIC (Fuji Plus (FP), GC America, Illinois, USA) | 38 |
| FIGURE XVII | Resin luting agent (Rely X Ultimate (RUL), 3M ESPE, CA, USA 3M ESPE, Irvine CA, USA | 39 |
| FIGURE XVIII | Resin luting agents (Rely X Unicem (RU), 3M ESPE, | 40 |

| | | |
|---|---|---|
| | Irvine CA, USA) | |
| FIGURE XIX | Silicone Fluid (100 Cps Silicone Oli – Polydimethylsilxane) Factur II,AZ USA | 41 |
| FIGURE XXI | Cylindrical specimens (7 $^+$ 0.1 mm in diameter and 2 $^+$ 0.1 mm in height) | 42 |
| FIGURE XXII | Calcium analysis by atomic absorption spectroscopy (Perkin Elmer Analyst 200) | 43 |
| FIGURE XXIII | A 0.00001- g balance (Model AG285, Mettler Toledo) with an apparatus for weighing specimens in air and in fluid | 44 |

# INTRODUCTION

Long-term success of fixed restorations depends on many factors. The luting agent is a crucial factor in the outcome of cemented fixed restorations. Dental luting agents can be categorized as water-based, oil-based, and resin-based. The ideal luting agent should provide reasonable bond strength between dissimilar materials; demonstrate favorable compressive and tensile strengths; and have sufficient fracture toughness, adequate film thickness, and sufficient working time.[1] The film thickness of the luting agent determines the adaptation of the restoration, and according to the ISO specification, the film thickness requirement for a water-based luting agent should be below 25 µm.[1] A sufficient period of time must be available to allow the complete seating of the restoration; this time lapse is specified by the ISO for water-based luting agents as between 2.5 min to 8 min at body temperature.[1]

Another attribute of luting agents is solubility in water and oral fluids. Generally, water-based luting agents are considered more soluble than resin- or oil-based luting agents.[2] Also, the dimensional stability is a crucial consideration related to the setting contraction, coefficient of thermal expansion, water sorption, and solubility of luting agents. Dimensional changes of the luting agent are a concern for clinicians. The luting agent’s shrinkage associated with setting may introduce gaps and channels along the tooth/luting agent or restoration/luting agent interface. These channels may be large enough to permit micro-organisms to pass along the spaces, influencing the mechanical properties, and contributing to loss of marginal integrity and resulting in restoration failure.[3] Moreover, materials that absorb water can expand markedly in a wet environment like the oral cavity. Hygroscopic expansion associated with water sorption may be beneficial in relieving some of the internal setting stresses and increasing the longevity of the adhesive union to the surrounding tooth. This phenomenon has been reported by Momoi and McCabe.[4] They suggest that during the early stages, hygroscopic expansion may close any gaps and relieve interface stresses that have been generated. A hygroscopic expansion that approximates the value of setting shrinkage is of concern. An excessive expansion may cause an outward force against cavity walls and cause cracking and fracture of the tooth structure in specific clinical situations, such as the cementing of an inlay or a post. Also, using a luting agent that significantly expands underneath a brittle material such as a ceramic could cause failure of the restoration.

The dimensional stability during and after setting of one luting agent in particular is the focus of the present study. Ceramir C&B (CM) is water-based cement approved for marketing in the US in its powder-liquid, hand-mixed version, and most recently in a capsule-delivery system, Composed of calcium aluminate and glass ionomer, Ceramir, originally named XeraCem (Doxa Dental AB, Uppsala, Sweden), is indicated for permanent cementation of cast restorations, all-zirconia or all-alumina crowns, and prefabricated metal and cast dowel and cores.

The dimensional stability of Ceramir is not fully understood and needs to be further investigated. The aim of this study is to evaluate the volumetric dimensional changes of this new calcium aluminate-glass ionomer luting agent.

## HYPOTHESES

### Null Hypotheses

1. Water storage will not impact the dimensional stability of calcium aluminate-glass ionomer cement.
2. Calcium aluminate-glass ionomer cement will not have detectable $Ca^{+2}$ and $OH^-$ release concentrations.

### Alternative Hypotheses

1. The water storage will result in an increase in the dimensions of the new cement.
2. Calcium aluminate-glass ionomer cement will have detectable $Ca^{+2}$ and $OH^-$ release concentrations.

# REVIEW OF LITERATURE

Dimensional stability is relevant to the appropriate function of a luting agent.[6] Sindel et al. evaluated the crack formation of IPS-Impress all-ceramic crowns dependent on different core materials and luting agents. After 12 months' storage, it was found that resin-modified glass ionomer and compomer used for cores or luting will produce failures of IPS-Impress all-ceramic crowns due to hygroscopic expansion of the material, and that the time of cracking depends on the amount and type of the material used.

Over the past century, a variety of luting agents have been used in dentistry including zinc-phosphate cement, polycarboxylate cement, glass-ionomer cement, resin-modified glass ionomer cement, and resin luting agents. Each has its advantages and disadvantages. The selection criteria are dependent upon each product's properties and the published data. Handling properties, biocompatibility, insolubility, and degradation resistance are examples of properties that influence the clinical selection. Also, the clinician's preference might play a major role in selection.

## ZINC PHOSPHATE LUTING AGENT

Zinc phosphate cement has been used for a long time. The cement strength depends on the powder- to-liquid ratio. Compressive and tensile strengths of properly mixed zinc phosphate are adequate to resist masticatory stresss.[2] The set cement is extremely stiff and exhibits a high modulus of elasticity, which permits the cement to resist elastic deformation in regions of high masticatory stresses or in long-span prostheses.[2] Lack of chemical adhesion and solubility in oral fluids are disadvantages of the zinc-phosphate cements.

## POLYCARBOXYLATE LUTING AGENT

Polycarboxylate cements have lower compressive and higher tensile strengths than zinc phosphate. Polycarboxylate cement is hydrophilic and capable of wetting dentinal surfaces. It exhibits chemical adhesion to tooth structure through the interaction of free carboxylic acid groups with calcium. Polycarboxylate cement has a pseudoplastic behavior; however, it exhibits an early increase in film thickness that may impact seating of the restoration.[2]

## GLASS-IONOMER LUTING AGENT

In 1972 Wilson and Kent introduced conventional glass-ionomer cement (GIC) for dental uses.[6] GIC was derived from silicate cement and polycarboxylate cement. As a luting agent, clinical success of glass-ionomer cement has been well documented.[7] Fluoride release and bonding to tooth structure are reasons for the popularity of the GIC. It has been reported that the compressive strength of GIC continues to increase over time. Glass-ionomer cement is sensitive to water.[8] The success of GIC depends on early protection from both hydration and dehydration. It is weakened by early exposure to moisture, while desiccation produces shrinkage cracks in the recently set cement.[9] Hersek et al. reported that GIC aged in water is mechanically weaker.[11] Keyf et al. showed that glass-ionomer luting agent has low solubility with significant differences between water sorption and water solubility. Some glass-ionomer cements show negative values for solubility and thereby imply the uptake of water into the cement structure.[12]

## RESIN-MODIFIED GLASS-IONOMER LUTING AGENT

In 1988 resin-modified glass-ionomer cements (RM-GIC) were introduced to overcome some of the glass-ionomer cements' problems, such as sensitivity to humidity and early weak mechanical strength. RM-GICs were formed by the replacement of the polyacid with a modified polyacid grafted with unsaturated groups, and the incorporation of polymerizable hydrophilic resins.[13] Hydrophilic resin, such as hydroxyethyl methacrylate (HEMA), is added as a co-solvent. It also polymerizes or copolymerizes with the modified polyacid.[14] RM-GICs show some advantages over the conventional GIC. They particularly allow a longer working time as they are photo-chemically initiated, reducing the early sensitivity to moisture and dehydration associated with the early stage of the acid-base setting reaction in the conventional GIC. Moreover, RM-GICs show rapid hardening of their surface. The inclusion of resin in the glass-ionomers leads to an increase in flexural and tensile strength of the cement. It is not clear if the inclusion of resin into the cements increases the surface microhardness and resistance to compression.[14,15] Ellakuria et al. compared the microhardness of resin-modified versus conventional glass-ionomer cements after one year of water storage. The resin-modified glass-ionomer cements showed a significantly lower hardness than the conventional GIC. They attributed their findings to the interposition of the HEMA matrix preventing the complete formation of the poly-salt matrix, inhibiting the acid-base reaction.[17] On the other hand, the decrease in microhardness may be due to the hypothetical separation of the phases described in the microstructure of these materials.[13,19] A third explanation for the reduction in microhardness could be the high proportion of functional hydrophilic groups contained within the matrix absorbing a large quantity of water and thus producing a plasticizing effect.[20,21] Bourke et al. stated that RM-GIC reaches its maximum hardness at one day after which no significant increase is detected.[22] A luting agent should have sufficient mechanical properties to resist functional forces over the lifetime of the restoration. Glass-ionomer was found to be significantly harder than the resin modified glass-ionomer. These results are possibly related to the presence of a solid silicate phase around the non-reacting glasses responsible for the hardening.[23,24]

Cattani-Lorinte et al. tested the effect of water on the physical properties of the resin-modified glass-ionomer cements, and stated that the RM-GICs are very sensitive to water sorption. Samples left in contact with water showed lower flexural strength, lower elastic modulus, and a softer surface than dry samples.[25] Watts et al. studied the effect of different environmental conditions on different resin-modified glass ionomer. Their results showed that RM-GIC exhibited pronounced expansion in water. In silicone oil it expanded slightly at 23°C and shrank by 3.6 percent at 37°C.[26] Attin et al. evaluated the volumetric changes of conventional glass ionomer, hybrid composite, and resin-modified glass ionomer after 14 days and 28 days of water storage. The result showed that conventional glass-ionomer cement showed a marked volumetric loss due to water storage. The volume of the hybrid composite was nearly constant. The other materials expanded as a function of the duration of the water immersion.[27]

### Resin-Based Luting Agents

Resin-based luting material has gained in popularity due to the benefit of the acid-etch technique for attaching resin to tooth substrate and the possibility of attaching the resin to the prosthetic material if properly prepared. In addition, low solubility has been reported with the resin luting agents.

The resin-based luting materials are suitable for use with all indirect restorative materials. They have shown the best mechanical properties of all luting cements. One drawback is that they require more complicated clinical procedures, such as different bonding systems for the dentin, enamel, and for the restoration.[28]

Self-adhesive resin cement is based on the use of polymerizable acidic monomers, which will etch both enamel and dentin simultaneously. Commercially available self-etching adhesive products contain monomers that can be divided into three main groups according to their function: 1) self-etching adhesive monomers; 2) cross-linking monomers, and 3) additional monofunctional co-monomers.[29] Phosphorus-containing monomers are capable of etching both enamel and dentin. They were first used in dental adhesives of the second generation; the first commercially introduced compound being the glycerol dimethacrylate ester of phosphoric acid (GDMP).[30] These compounds are well known in the literature for their hydrolytic instability.[31] Applying monomers containing a more hydrolytically stable bond between the polymerizable group and the strong acidic phosphate group helps to reduce the hydrolytic instability of methacrylate phosphates. Anbar et al. carried out the first evaluation of polymerizable phosphates for dental adhesives.[32,33] They showed that vinylphosphonic acid (VPA) and 4-vinylbenzylphosphonic acid (VBPA) or corresponding copolymers can improve the adhesion of restorative composites on etched enamel. Other acrylic ether phosphoric acids (AEPA) were also introduced with improved hydrolytic stability and reactivity in the free-radical polymerization. All self-adhesive luting agents tested in this study contained phosphorous monomers.[34]

Fillers in the luting agents are divided into two groups. One group is silanated to bond with the polymerizable monomer that improves the mechanical properties of the luting agent. The other group is alkaline in nature, acting as a buffering agent to raise the pH after conditioning and facilitating proper penetration. reducing the hydrophilicity, and thus the viscosity, and increasing the wettability.[35]

The self-etch luting agent should have a degree of hydrophilicity to improve its wettability and penetration into conditioned enamel/dentin; but, it becomes more hydrophobic during the setting reaction.

Dimensional changes of resin composite during and after setting are a source of concern for the clinician. Martin et al. evaluated the hygroscopic expansion of composite restoratives. The results of the study showed that all the materials tested expanded with mean dimensional changes after water immersion with values ranging from 0.15 percent to 2.39 percent. The nature of the resin matrix plays a significant role in the hygroscopic expansion. The hydrophilicity of the resin matrix will determine the ability of the water molecule to diffuse into the matrix.[36] Sideridou studied the volumetric dimensional changes of light-polymerizing composite resin after sorption of water or ethanol. Their result showed that the amount of the expansion was dependent on the monomer used in preparing the composite resin matrix.[37] Wei et al. studied the dimensional changes of self-adhesive composite and their result showed that due to the presence of a hydrophilic monomer, self-adhesive composite was the least dimensionally stable.[38]

## CALCIUM ALUMINATE CEMENT

Calcium aluminate cement is a material with applications for the medical and dental fields. Its application in dentistry may include use as a posterior restorative material; as a luting cement, or as an endodontic sealer. It is a ceramic powder mainly composed of calcium aluminate that reacts with water in an acid-base reaction. Fillers like $Zro_2$, Sio, or Feo are added to improve the properties. It is biocompatible. However, this cement's long-term performance has limitations when subjected to loading because of the porous nature of the material. Sunnegårdh et al. showed that the flexural strength of calcium-aluminate cement was significantly lower compared with the resin composite tested in the study and that the storage time has a significant effect on the flexural strength.[39] Furthermore, it has been shown that the material has significantly higher bulk levels of porosities than both composite resins and amalgam.[40] Also, calcium aluminate cement has lower dimensional stability over time than composite resin, which was reported by Berglunda.[41] The results of the study showed that the dimensional change of calcium-aluminate cement in distilled water and in 100-percent humidity between 24 hours and 360 days was 5 times to 10 times higher than that of the composite resins tested. The conclusion was that a material that continues to absorb water and continues to react is questionable for use clinically.[41] Low flexural strength, high porosity concentration, and low dimensional stability are possible explanations of the shortcoming of the materials. Van Dijken and Sunnegårdh-Grönberg have shown in a clinical study that the cumulative failure frequency of calcium aluminate cement was 43 percent compared with 3 percent for the composite resin.[42]

## CALCIUM ALUMINATE-GLASS IONOMER CEMENT

Water-based cement called Ceramir is approved for marketing in the US in a powder-liquid, hand-mixed version, and most recently, in a capsule-delivery system. Composed of calcium aluminate and glass ionomer, Ceramir C&B (CM), originally named XeraCem (Doxa Dental AB, Uppsala, Sweden), is a luting agent indicated for permanent cementation of cast restorations, all-zirconia or all-alumina crowns, and prefabricated metal and cast dowel and cores.

The manufacturer claims that the cement has demonstrated favorable biocompatibility properties when tested *in vitro* and *in vivo* and has been shown to be bioactive.[43] Its basic pH and the production of excess $Ca^{2+}$ ions are factors that contribute to its bioactivity. Furthermore, they claim that it has less microleakage compared with other cements.[44] However, the dimensional stability of the material during and after setting is not fully understood and needs to be further investigated.

## PURPOSE OF THE STUDY

The aim of this study was to evaluate the volumetric dimensional changes of Ceramir, a calcium aluminate-glass ionomer luting agent.

# MATERIALS AND METHODS

A calcium aluminate glass ionomer luting agent (Ceramir C&B, CM), originally named XeraCem (Doxa Dental AB, Uppsala, Sweden), was investigated in this study(Figure XV). Resin-modified GIC (Fuji Plus; FP, GC America, Illinois) and resin luting agents (Rely X Ultimate; RA, and Rely X Unicem; RU, 3M ESPE, Irvine, CA) were used as control groups (Figure XVI-XVIII). The tested materials were manipulated according to the manufacturer's instructions (Table I). A total of 20 specimens were made for each material, 5 for each group. Cylindrical specimens were prepared using Teflon molds with internal dimensions of 7 mm ± 0.1 mm diameter and 2.0 ± 0.1 mm depth (Figure XXI). Each mold was placed on top of a glass slide covered with a Mylar sheet. Then, the mold was slightly overfilled with the materials to be tested. After filling, the mold was covered with a second Mylar sheet, and a glass slide was used to remove excess material. The slide was loaded with a 1-kg force device. For the dual-cured cements a visible light-curing unit (Demetron Optilux, Model No.VCL 401, Demetron Research Corp.) with an output of 790 mW/cm$^2$ and a 13-mm tip was used to initiate the polymerization. The specimen was exposed to the light for 40 s from both sides of the mold. Each specimen was finished by holding the periphery against 1000-grit abrasive paper. Each specimen was washed with distilled water, wiped with tissues, and then blown with a dust-off blower to ensure all debris was removed. Five specimens were randomly assigned to the four test conditions. These conditions were silicone oil (polydimethylsiloxane, Figure 16) at 23 °C and 37 °C and distilled water at 23 °C and 37 °C. Fluids were stored at 23 °C and 37 °C for 24 h before their use. After 24 h and throughout the study, the fluid temperatures were monitored with a digital thermometer (Omega, Model 871).

A 0.01-mg resolution balance (AG 285, Mettler, Toledo) was used to measure the weight of each specimen at various time intervals up to 30 days (Figure XXII). Each specimen was weighed at 10 min from the start of fabrication in air and in its immersion fluid (silicone oil or distilled water). This value was the original weight of each material. The time intervals measured were 7 days, 14 days, 21 days, and 30 days from the start of fabrication. For weight in air, each specimen was removed from the test tube and was dried by blotting with absorbent tissue to remove the excess liquid. The specimen was placed on the balance for 30 s and the displayed weight was noted. The same specimen was removed from the pan and placed in the holder immersed in its immersion fluid. This procedure was done with extreme care to ensure that no air bubbles adhered to the solid. Separate brushes for each fluid were used to remove air bubbles. The specimen was kept on the apparatus for 30 s and the displayed weight was noted. The specimen was then placed back in its test tube. This procedure was repeated three times at each time period.

Archimedes' principle (buoyancy of a material in fluid) was used to measure volumetric dimensional changes. By Archimedes' principle and using equations previously developed:[26, 45]

Volume displaced [volume of object] = mass up thrust/density of immersion fluid

$$V = \Delta M/ \rho= (M_a-M_w)/ \rho \qquad (1)$$

$M_a$ = the mass in air

$M_w$= the apparent mass, immersed in the fluid.

The volumetric change $\Delta V$ can be defined as follows:

$\Delta V (\%) = [\{V_e-V_o\}/V_o\}x100 \;\; =[\{V_e-V_o\}- 1] \text{ x } 100$ (2)

$V_e$ = the changed volume

$V_o$ = the original volume

Substituting Eq. (1) in Eq. (2),

$\Delta V = [\{(M_{a,t}-M_{w,t})/ \rho_t] \text{ X } [\rho / (M_a-M_w)\}^{-1}]x100$

$M_{ast}$ = the mass in air at time (t)

$M_{w,t}$ = the apparent mass, immersed in fluid at time (t)

$P_t$ = density of liquid at time (t) temperature

ρ = density of liquid at original time temperature

ION RELEASE TESTS

Chemical analysis of the storage medium solution (distilled water) of the new cement was performed. Forty solutions (n = 5; Five samples from each storage temperature at different time intervals, which included 7 days, 14 days, 21 days and 30 days) were analyzed using atomic absorption spectroscopy to determine the [$Ca^+$]. For each storage period, a 50-µl aliquot of each sample was transferred to a 7-ml vial. Then 1 ml of lanthanum chloride ($LaCl_3$) was added to the vial with 3.95 ml of DI water. Each sample was analyzed with atomic absorption spectroscopy (a Perkin Elmer Analyst 200) (Figure XXI). $Ca^+$ concentration was determined by comparison with a standard curve. The pH values were measured to determine the $OH^-$ concentration in the solutions.

By knowing the pH value of the solutions at the different time intervals, $OH^-$ concentration was calculated through the $pOH^-$ value. Baseline pH was established by measuring the DI water's pH before sample immersion:

At room temperature:

$pH + pOH = 14$ (1)

$[OH^-] = 10^{-pOH}$ (2)

## STATISTICAL METHODS

Summary statistics (mean, standard deviation, standard error, range) were calculated for each of the 16 treatment combinations (4 materials × 2 temperatures × 2 fluids). Linear mixed effects models were used to evaluate the effects of material, storage temperature, storage fluid, and time on volumetric dimensional change, Ca+ release, and OH- release. The models included all interactions among the factors and a random effect to account for within-sample correlations. Pair-wise comparisons between the treatment combinations were performed using Fisher's Protected Least Significant Differences to control the overall significance level at 5 percent.

## SAMPLE SIZE JUSTIFICATION

Based on a previous study the within-group standard deviation of the volumetric dimensional change was estimated to be 0.25. With a sample size of 5 samples per treatment combination, the study had an 80-percent power to detect differences of 0.5 between two treatment combinations, assuming two-sided tests conducted at a 5-percent significance level.

# RESULTS

## VOLUMETRIC DIMENSIONAL CHANGES

Linear mixed-effect model for repeated measurements was used to test materials (Ceramir, Fuji, RA, and RU); storage media (oil and water); temperature (22°C and 37°C); time (1 week, 2 weeks, 3 weeks, and 4 weeks); two-way interactions, and three way interactions. The result shows that time, material, and media had significant impact on volume changes. Two-way interactions time*media, material*media; time*temperature, and media*temperature had significant impact on volume changes. In addition, three-way interactions time*material*media, time*media*temperature, and material*media*temperature had significant impact on volume changes. The mean percentage volumetric dimensional changes and other statistical data of the four luting agents in the study are shown in Table II. Figure I through Figure XII show the mean percentage volumetric changes for all four luting agents for each of the environmental conditions. For the luting agents stored in distilled water at both temperatures, Ceramir exhibited more expansion than the other luting agents. In silicone oil at both temperatures, Fuji Plus showed the most shrinkage among the tested luting agents.

## $Ca^{+2}$ RELEASE

The mean release concentration is shown in Table IV. Figure XIII illustrates the scatter plot and prediction means of [$Ca^{2+}$] for each temperature. From week 1 to week 3, as the time increased, the [$Ca^{2+}$] increased more rapidly at 37°C than at 22°C.

However, after week 3, they had a similar rate of increase. From week 1 to week 7, the $[Ca^{+2}]$ was always higher at 37°C than at 22°C.

## $OH^-$ RELEASE

The mean $OH^-$ concentration is presented in Table V. Figure XIV illustrates the scatter plot and prediction means of $[OH^-]$ for each temperature. From week 1 to week 3, as the time increased, the $[OH^-]$ increased more rapidly at 22°C than at 37°C. However, after week 3, the increase of $[OH^-]$ was similar for both 22°C and 37°C.

# FIGURES AND TABLES

Table I

Manufacturer's instructions

| Luting Agents | Instructions |
|---|---|
| Fuji Plus<br>(GC America, Illinois, USA) | 1. The powder to liquid ratio was 2.0g / 1.0g. (1 level large scoop of powder to 3 drops of liquid.<br>2. The powder and liquid were dispensed onto a mixing pad. Using a plastic spatula, all the powder was added to the liquid and mixed rapidly for 20 seconds.<br>3. Isolation will be maintained until set is verified (Approx. 4 minutes). |
| Ceramir C&B (CM),<br>(Doxa Dental AB, Uppsala, Sweden) | 1. The aluminium pouch was opened and the capsule was removed.<br>2. The capsule was placed into the activator holder with the cap upward and the application tip pointing in a line away from the activator body.<br>3. The activating lever was pressed all the way down and hold for 3 seconds. This is important to ensure that all liquid in the sachet is squeezed into the mixing chamber.<br>4. Immediately the capsule was inserted in a mixer of high frequency oscillating mixers (4,000 to 5,000 rpm),and was mixed for 8 to 10 seconds<br>5. Immediately was inserted into the applicator using the grooves to hold it in place. |
| Rely X Unicem (RU)<br>(3M ESPE Seefeld, Germany) | 1. The 3M™ESPE™RelyX™Unicem Self-Adhesive Universal Resin Cement capsule was mixed in a high-frequency mixing unit (e.g. Capmix™) for 15 sec or in the Rotomix™capsule-mixing unit for 10 sec (see also the section on "Times").<br>2. Application: the capsule was inserted in the Aplicap Applier after mixing and the nozzle was opened as far as possible. Isolation will be maintained.<br>3. Light curing: 20 sec for each surface. |
| Rely X Ultimate (RA)<br>(3M ESPE Seefeld, Germany) | 1. Apply and evenly distribute a thin layer of cement to the bonding surface of the indirect restoration.<br>2. Setting time 3–5 min.<br>3. Light cure for 40 seconds or allowed to self-cure for 10 minutes from start of mix. |

Table II

Summary Statistics for Volumetric Dimensional Changes

| Week | Material | Media | Temperature | N | Mean | Standard Deviation | Standard Error | Minimum | Maximum |
|---|---|---|---|---|---|---|---|---|---|
| Week 1 | Ceramir | Oil | 22C | 5 | -0.56 | 0.35 | 0.16 | -1.04 | -0.21 |
| Week 1 | Ceramir | Oil | 37C | 5 | -2.14 | 2.16 | 0.97 | -4.52 | 0.21 |
| Week 1 | Ceramir | Water | 22C | 5 | 17.52 | 5.81 | 2.60 | 7.99 | 23.30 |
| Week 1 | Ceramir | Water | 37C | 5 | 19.69 | 3.38 | 1.51 | 14.64 | 23.96 |
| Week 1 | Fuji | Oil | 22C | 5 | -1.91 | 1.06 | 0.48 | -3.74 | -1.20 |
| Week 1 | Fuji | Oil | 37C | 5 | -5.71 | 1.32 | 0.59 | -7.54 | -3.99 |
| Week 1 | Fuji | Water | 22C | 5 | 7.62 | 2.77 | 1.24 | 5.53 | 12.40 |
| Week 1 | Fuji | Water | 37C | 5 | 9.73 | 0.68 | 0.30 | 8.84 | 10.58 |
| Week 1 | RelayUL | Oil | 22C | 5 | -0.76 | 0.44 | 0.20 | -1.38 | -0.17 |
| Week 1 | RelayUL | Oil | 37C | 5 | -2.08 | 0.46 | 0.21 | -2.57 | -1.60 |
| Week 1 | RelayUL | Water | 22C | 5 | 0.50 | 0.91 | 0.41 | -0.61 | 1.87 |
| Week 1 | RelayUL | Water | 37C | 5 | 1.58 | 1.02 | 0.46 | 0.60 | 3.30 |
| Week 1 | RelayUN | Oil | 22C | 5 | -1.03 | 1.12 | 0.50 | -2.92 | -0.00 |
| Week 1 | RelayUN | Oil | 37C | 5 | -1.97 | 0.43 | 0.19 | -2.52 | -1.55 |
| Week 1 | RelayUN | Water | 22C | 5 | 0.64 | 0.39 | 0.17 | 0.11 | 1.19 |
| Week 1 | RelayUN | Water | 37C | 5 | 1.23 | 0.42 | 0.19 | 0.55 | 1.66 |
| Week 2 | Ceramir | Oil | 22C | 5 | -0.57 | 0.49 | 0.22 | -1.24 | -0.00 |
| Week 2 | Ceramir | Oil | 37C | 5 | -1.83 | 3.14 | 1.41 | -6.28 | 1.21 |
| Week 2 | Ceramir | Water | 22C | 5 | 15.64 | 4.33 | 1.94 | 8.08 | 18.69 |
| Week 2 | Ceramir | Water | 37C | 5 | 19.83 | 3.29 | 1.47 | 14.34 | 22.51 |
| Week 2 | Fuji | Oil | 22C | 5 | -2.13 | 1.62 | 0.72 | -4.99 | -1.08 |
| Week 2 | Fuji | Oil | 37C | 5 | -6.00 | 0.32 | 0.14 | -6.31 | -5.45 |
| Week 2 | Fuji | Water | 22C | 5 | 6.99 | 0.60 | 0.27 | 6.42 | 7.63 |
| Week 2 | Fuji | Water | 37C | 5 | 10.29 | 1.62 | 0.73 | 8.07 | 12.06 |
| Week 2 | RelayUL | Oil | 22C | 5 | -0.55 | 0.43 | 0.19 | -1.21 | -0.17 |
| Week 2 | RelayUL | Oil | 37C | 5 | -2.00 | 0.32 | 0.14 | -2.34 | -1.66 |
| Week 2 | RelayUL | Water | 22C | 5 | 1.06 | 0.73 | 0.33 | 0.11 | 1.82 |
| Week 2 | RelayUL | Water | 37C | 5 | 0.79 | 0.71 | 0.32 | 0.10 | 1.71 |
| Week 2 | RelayUN | Oil | 22C | 5 | -0.72 | 0.56 | 0.25 | -1.55 | -0.00 |
| Week 2 | RelayUN | Oil | 37C | 5 | -2.59 | 0.45 | 0.20 | -3.28 | -2.24 |
| Week 2 | RelayUN | Water | 22C | 5 | 0.30 | 0.35 | 0.16 | -0.06 | 0.70 |
| Week 2 | RelayUN | Water | 37C | 5 | 1.44 | 0.41 | 0.18 | 0.89 | 1.88 |
| Week 3 | Ceramir | Oil | 22C | 5 | -0.62 | 0.56 | 0.25 | -1.45 | -0.00 |
| Week 3 | Ceramir | Oil | 37C | 5 | -2.34 | 3.16 | 1.41 | -6.88 | 0.66 |
| Week 3 | Ceramir | Water | 22C | 5 | 14.86 | 3.80 | 1.70 | 8.54 | 18.61 |
| Week 3 | Ceramir | Water | 37C | 5 | 20.20 | 3.65 | 1.63 | 14.03 | 23.23 |
| Week 3 | Fuji | Oil | 22C | 5 | -2.71 | 2.15 | 0.96 | -6.54 | -1.44 |
| Week 3 | Fuji | Oil | 37C | 5 | -6.46 | 0.63 | 0.28 | -7.27 | -5.75 |
| Week 3 | Fuji | Water | 22C | 5 | 5.94 | 1.19 | 0.53 | 4.15 | 7.46 |
| Week 3 | Fuji | Water | 37C | 5 | 10.36 | 0.41 | 0.19 | 9.89 | 10.92 |
| Week 3 | RelayUL | Oil | 22C | 5 | -0.83 | 0.42 | 0.19 | -1.56 | -0.52 |
| Week 3 | RelayUL | Oil | 37C | 5 | -3.21 | 1.10 | 0.49 | -4.41 | -2.32 |
| Week 3 | RelayUL | Water | 22C | 5 | 0.81 | 0.37 | 0.17 | 0.28 | 1.29 |
| Week 3 | RelayUL | Water | 37C | 5 | 1.97 | 1.02 | 0.46 | 0.39 | 3.25 |

| Week | Material | Media | Temperature | N | Mean | Standard Deviation | Standard Error | Minimum | Maximum |
|---|---|---|---|---|---|---|---|---|---|
| Week 3 | RelayUN | Oil | 22C | 5 | -0.79 | 0.69 | 0.31 | -1.72 | 0.18 |
| Week 3 | RelayUN | Oil | 37C | 5 | -2.97 | 0.92 | 0.41 | -4.13 | -1.66 |
| Week 3 | RelayUN | Water | 22C | 5 | 0.24 | 0.76 | 0.34 | -0.57 | 1.22 |
| Week 3 | RelayUN | Water | 37C | 5 | 1.13 | 0.39 | 0.17 | 0.61 | 1.53 |
| Week 4 | Ceramir | Oil | 22C | 5 | -0.64 | 0.57 | 0.26 | -1.45 | -0.00 |
| Week 4 | Ceramir | Oil | 37C | 5 | -2.48 | 3.14 | 1.40 | -6.82 | 0.64 |
| Week 4 | Ceramir | Water | 22C | 5 | 17.28 | 4.62 | 2.07 | 9.92 | 22.60 |
| Week 4 | Ceramir | Water | 37C | 5 | 20.52 | 3.46 | 1.55 | 14.56 | 22.99 |
| Week 4 | Fuji | Oil | 22C | 5 | -2.83 | 2.32 | 1.04 | -6.95 | -1.44 |
| Week 4 | Fuji | Oil | 37C | 5 | -6.45 | 0.54 | 0.24 | -6.91 | -5.75 |
| Week 4 | Fuji | Water | 22C | 5 | 6.97 | 0.58 | 0.26 | 5.99 | 7.46 |
| Week 4 | Fuji | Water | 37C | 5 | 10.45 | 1.07 | 0.48 | 8.54 | 10.98 |
| Week 4 | RelayUL | Oil | 22C | 5 | -0.80 | 0.40 | 0.18 | -1.38 | -0.35 |
| Week 4 | RelayUL | Oil | 37C | 5 | -2.82 | 0.98 | 0.44 | -3.86 | -1.94 |
| Week 4 | RelayUL | Water | 22C | 5 | 0.99 | 0.42 | 0.19 | 0.57 | 1.64 |
| Week 4 | RelayUL | Water | 37C | 5 | 1.30 | 0.72 | 0.32 | 0.11 | 1.94 |
| Week 4 | RelayUN | Oil | 22C | 5 | -0.76 | 0.84 | 0.38 | -1.72 | 0.53 |
| Week 4 | RelayUN | Oil | 37C | 5 | -2.84 | 0.44 | 0.20 | -3.45 | -2.41 |
| Week 4 | RelayUN | Water | 22C | 5 | 0.77 | 0.42 | 0.19 | 0.40 | 1.45 |
| Week 4 | RelayUN | Water | 37C | 5 | 1.13 | 0.35 | 0.16 | 0.55 | 1.44 |

# Table III

| Week | Temp | N | Mean | Standard Deviation | Standard Error | Minimum | Maximum |
|---|---|---|---|---|---|---|---|
| Week 1 | 22C | 5 | 0.89 | 0.20 | 0.09 | 0.64 | 1.12 |
| | 37C | 5 | 1.53 | 0.30 | 0.14 | 1.23 | 2.03 |
| Week 2 | 22C | 5 | 1.73 | 0.32 | 0.14 | 1.39 | 2.18 |
| | 37C | 5 | 2.93 | 0.48 | 0.22 | 2.28 | 3.62 |
| Week 3 | 22C | 5 | 2.52 | 0.49 | 0.22 | 1.99 | 3.05 |
| | 37C | 5 | 4.21 | 0.58 | 0.26 | 3.49 | 5.05 |
| Week 7 | 22C | 5 | 5.79 | 0.43 | 0.19 | 5.44 | 6.41 |
| | 37C | 5 | 7.36 | 0.60 | 0.27 | 6.53 | 8.19 |

# Table IV

| Week | Temp | N | Mean | Standard Deviation | Standard Error | Minimum | Maximum |
|---|---|---|---|---|---|---|---|
| Week 1 | 22C | 5 | 4.3057E-9 | 1.0976E-9 | 4.909E-10 | 2.5119E-9 | 5.0119E-9 |
| | 37C | 5 | 4.2296E-9 | 7.884E-10 | 3.526E-10 | 3.1623E-9 | 5.0119E-9 |
| Week 2 | 22C | 5 | 9.4243E-9 | 2.0102E-9 | 8.99E-10 | 6.493E-9 | 1.1321E-8 |
| | 37C | 5 | 7.5557E-9 | 1.0373E-9 | 4.639E-10 | 6.3246E-9 | 8.9929E-9 |
| Week 3 | 22C | 5 | 1.6714E-8 | 2.3551E-9 | 1.0532E-9 | 1.4436E-8 | 1.9265E-8 |
| | 37C | 5 | 1.1209E-8 | 1.3964E-9 | 6.245E-10 | 9.4868E-9 | 1.2974E-8 |
| Week 7 | 22C | 5 | 2.3415E-8 | 2.7796E-9 | 1.2431E-9 | 1.9284E-8 | 2.7026E-8 |
| | 37C | 5 | 1.98E-8 | 3.2819E-9 | 1.4677E-9 | 1.5318E-8 | 2.2974E-8 |

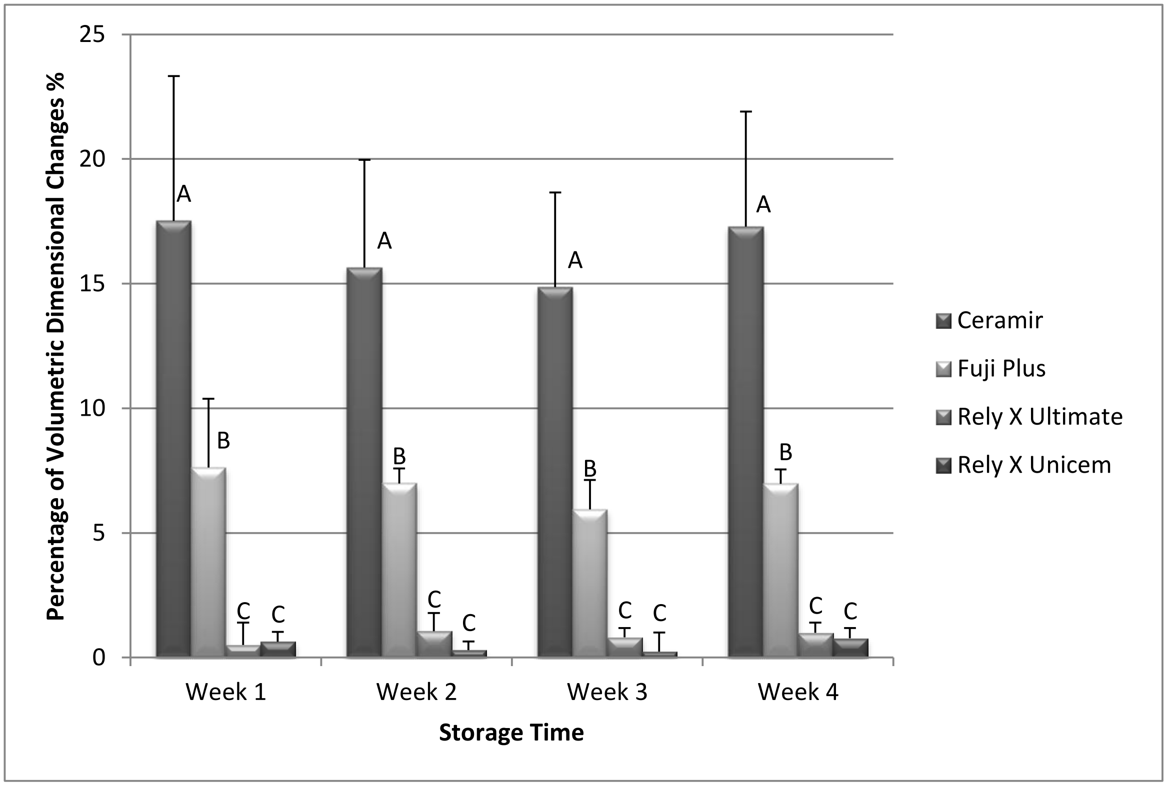

**Figure I: Mean Luting Agent Volumetric Dimensional Changes - Stored in Water at 22º C**

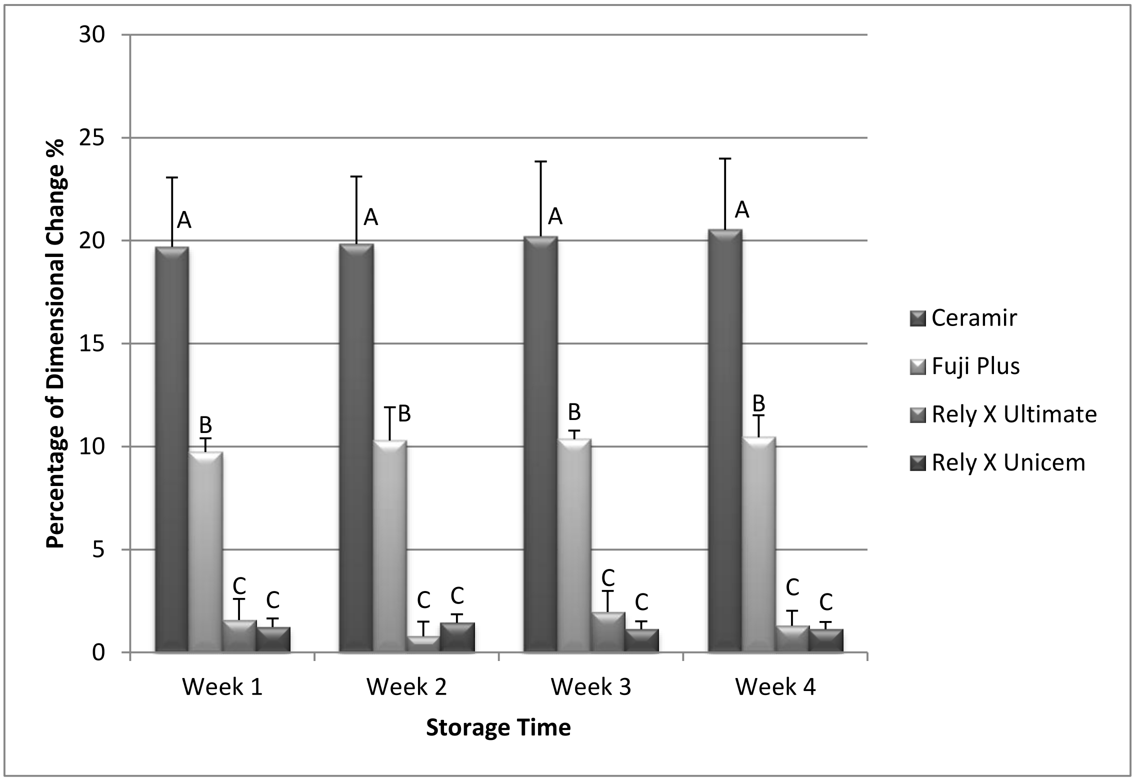

**Figure II: Mean Luting Agent Volumetric Dimensional Changes - Stored in Water at 37º C**

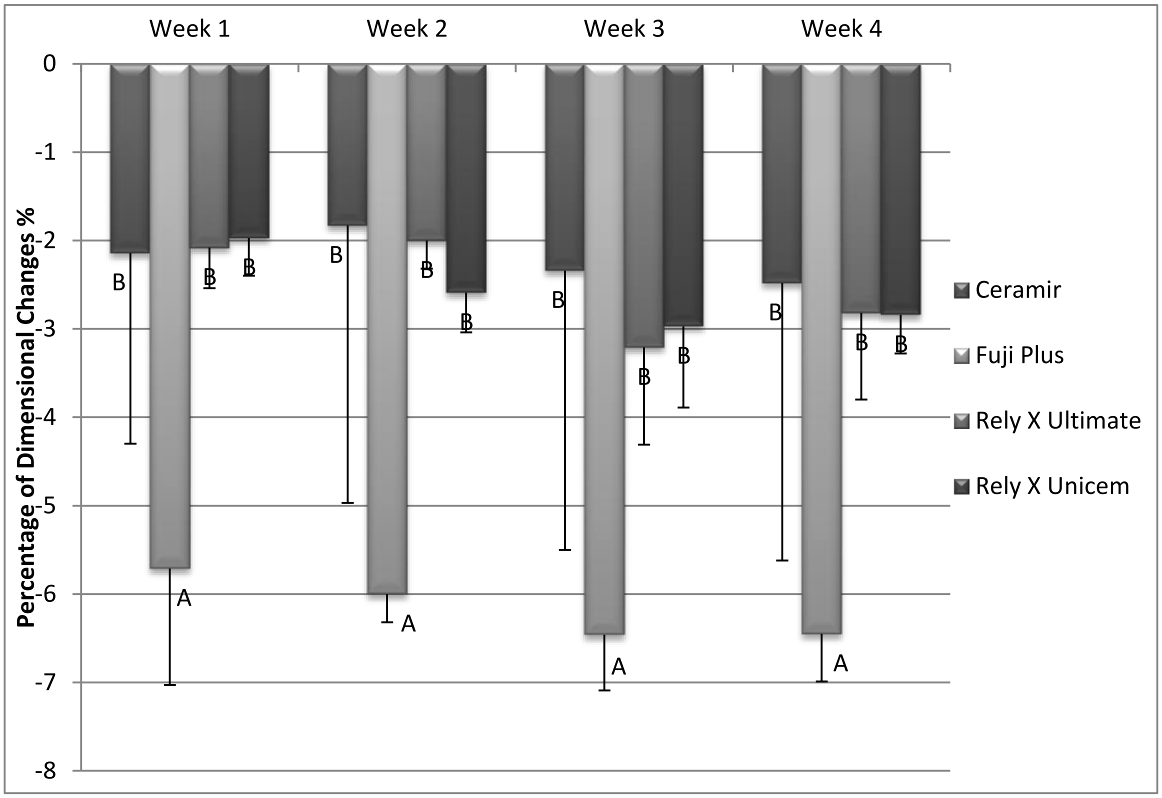

**Figure III: Mean Luting Agent Volumetric Dimensional Changes - Stored in Silicone oil at 37º C**

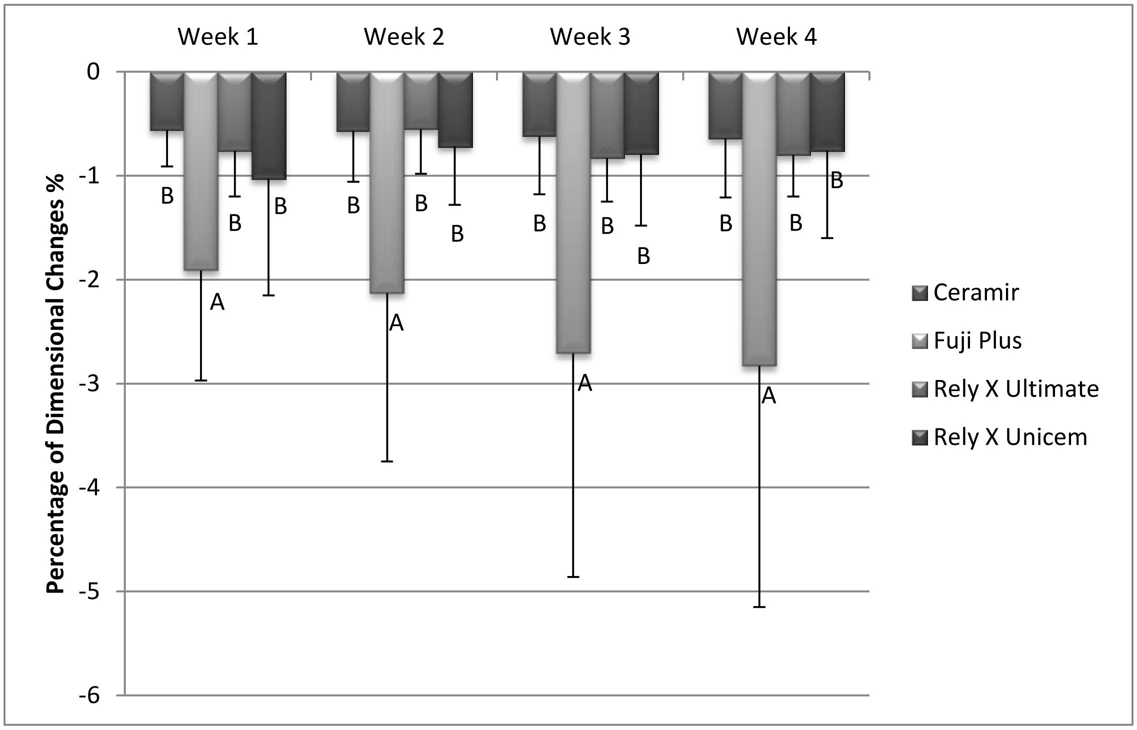

**Figure IV: Mean Luting Agent Volumetric Dimensional Changes - Stored in Silicone oil at 22º C**

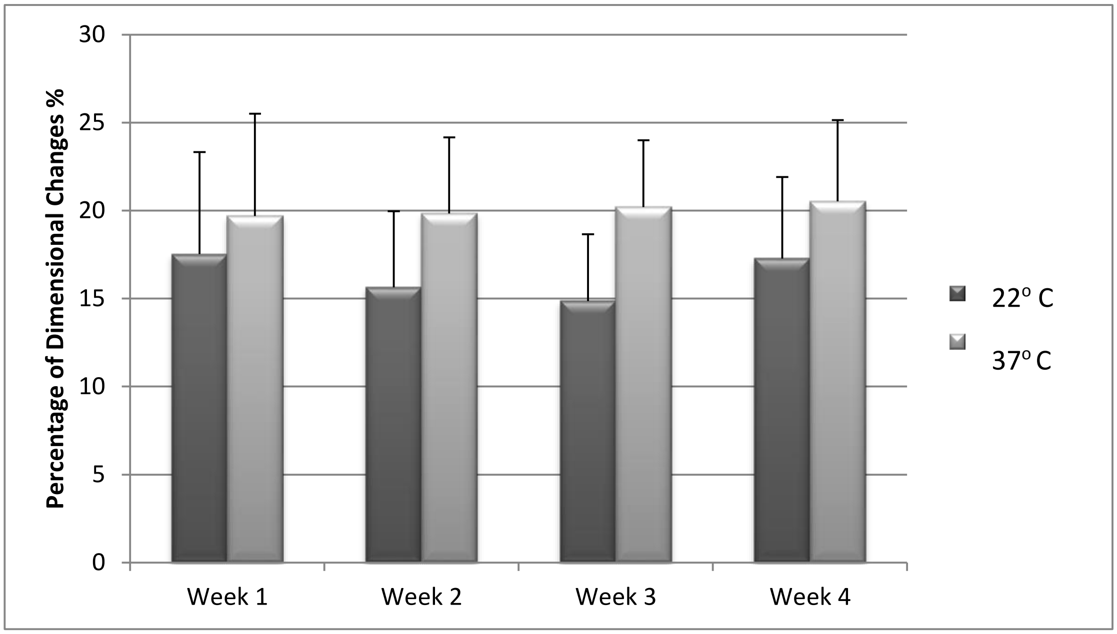

**Figure V: Mean Ceramir Volumetric Dimensional Changes - Stored in Water**

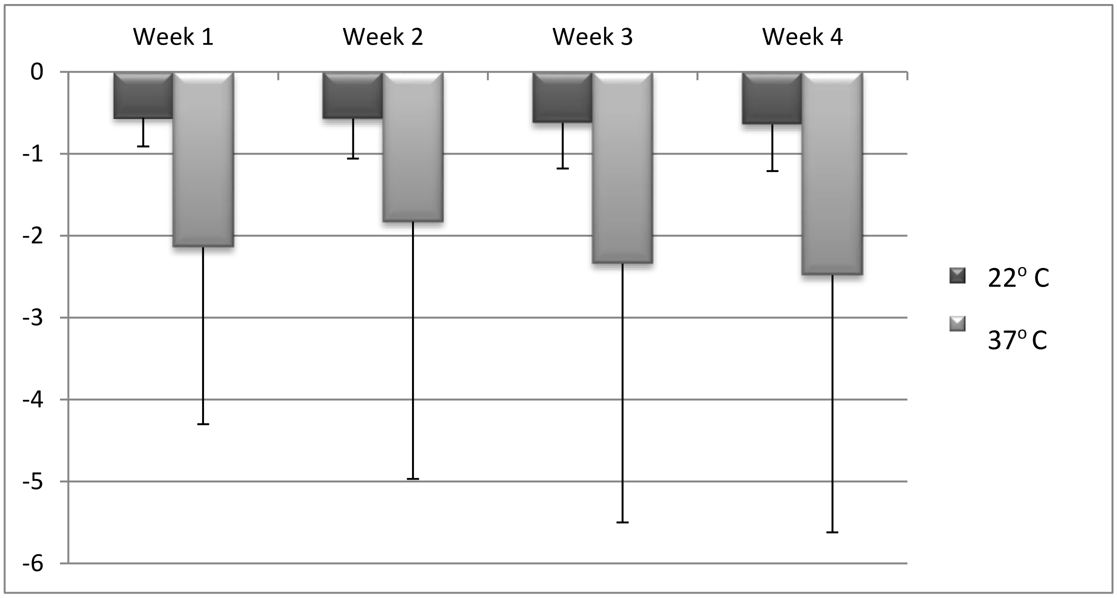

**Figure VI: Mean Ceramir Volumetric Dimensional Changes - Stored in Silicone oil**

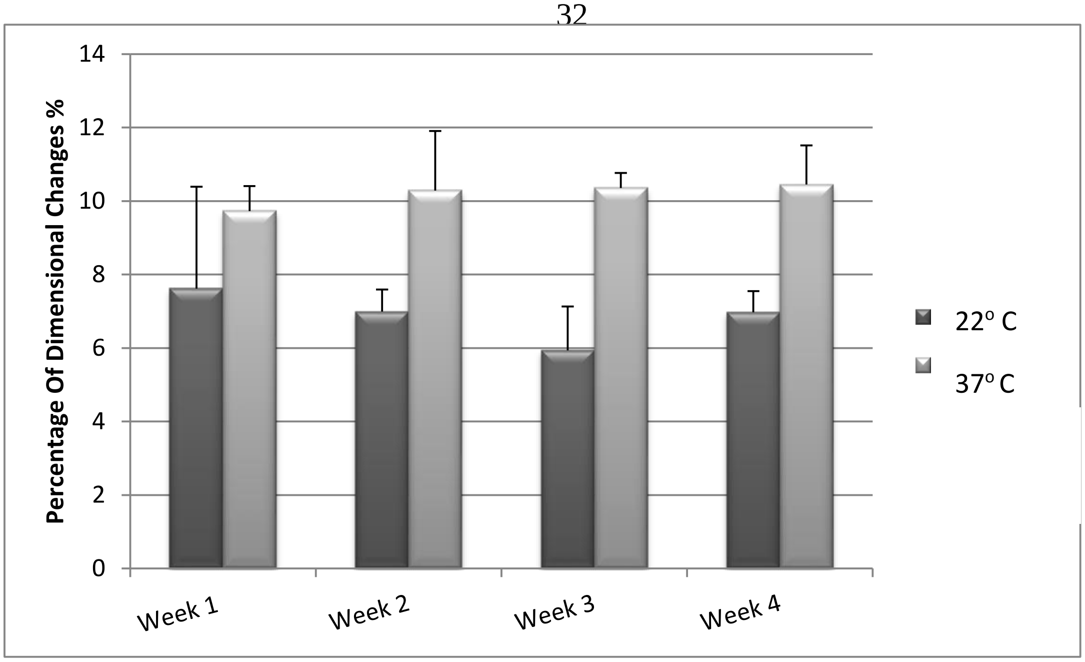

Figure VII: Mean Fuji plus Volumetric Dimensional Changes - Stored in Water

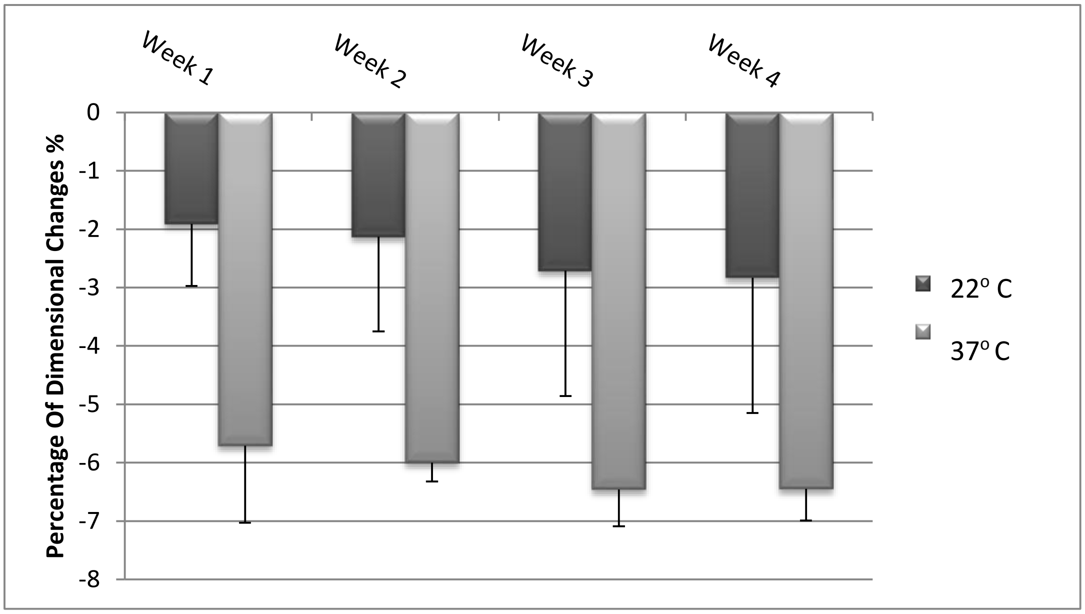

Figure VIII: Mean Fuji plus Volumetric Dimensional Changes - Stored in Silicone oil

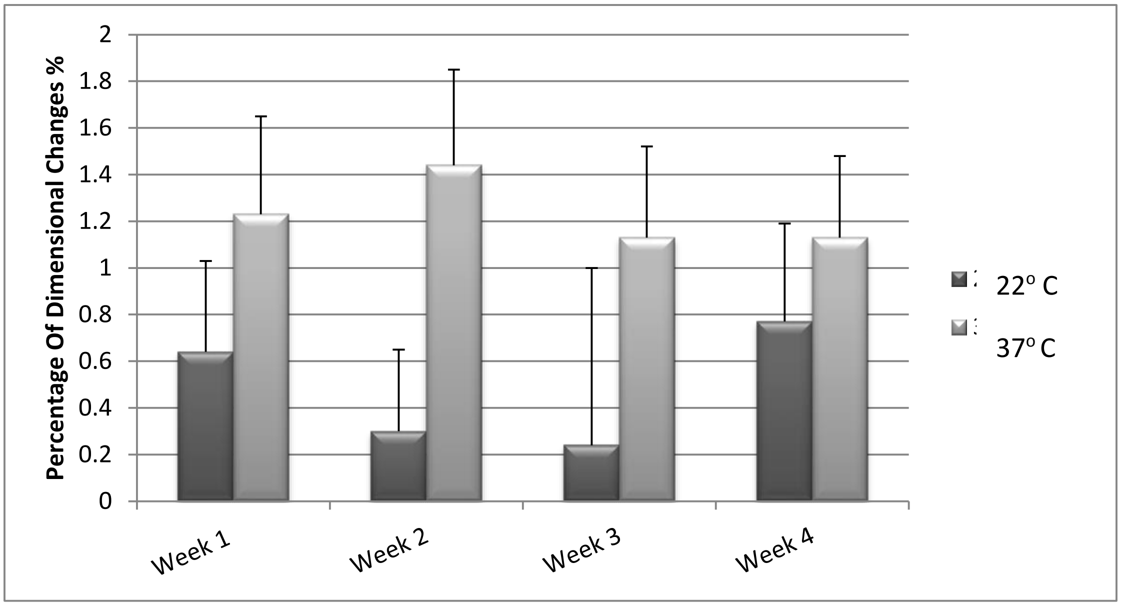

Figure IX: Mean Volumetric Dimensional Changes of Rely X Unicem Stored in Water

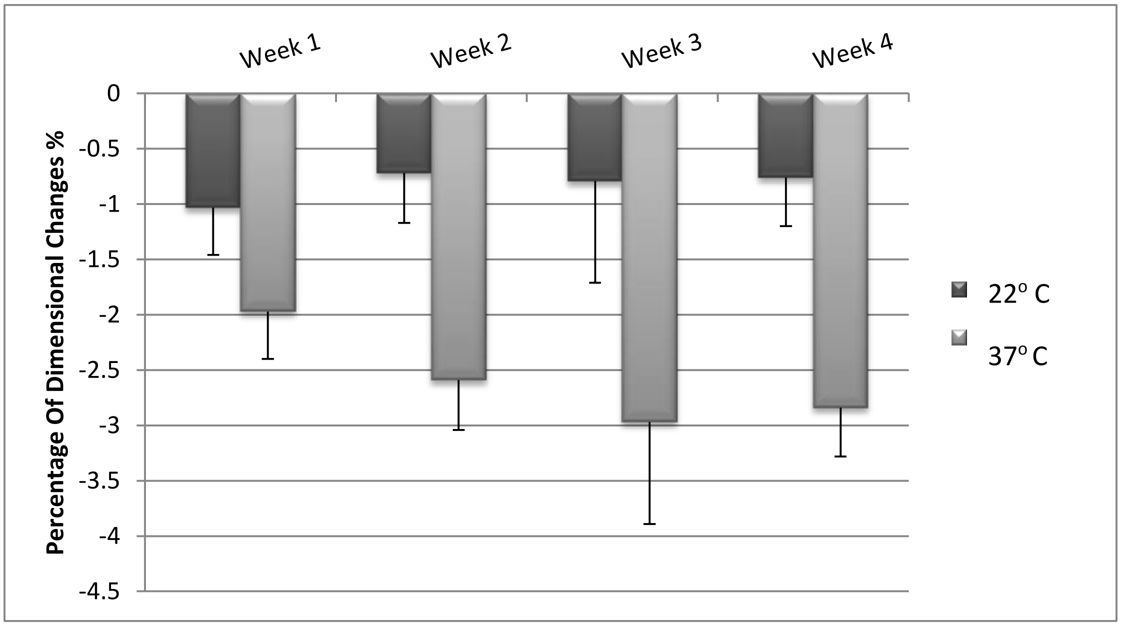

Figure X: Mean volumetric Dimensional Changes of Rely X Unicem Stored in Silicone Oil

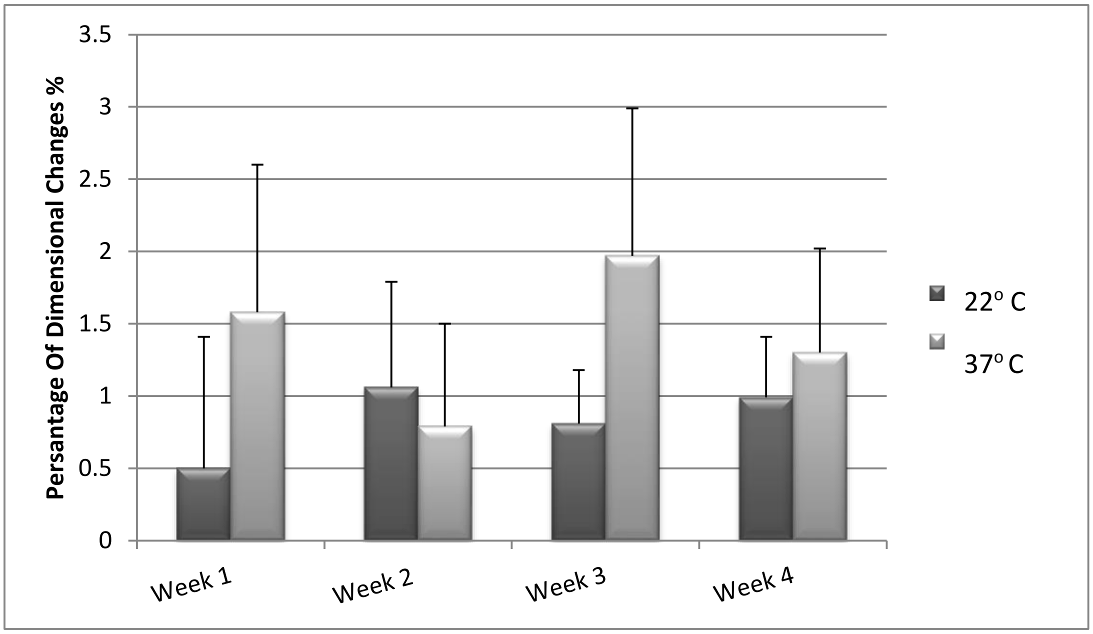

**Figure XI: Mean Volumetric Dimensional Changes of Rely X Ultimate Stored in Water**

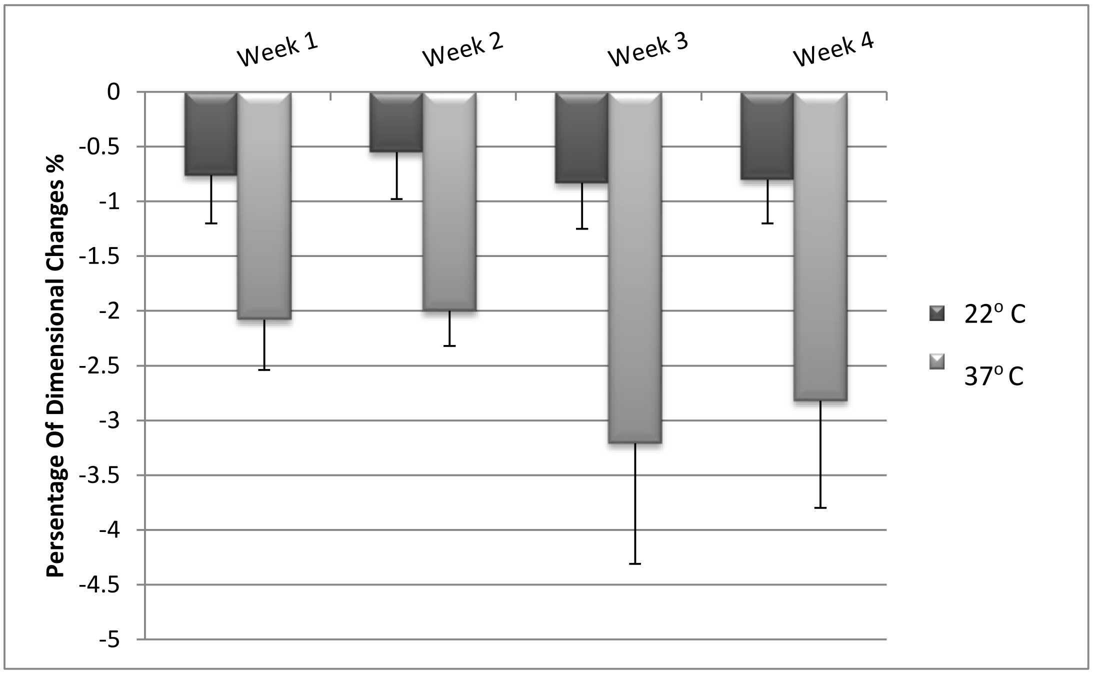

**Figure XII: Mean Volumetric Dimensional Changes of Rely X Ultimate Stored in Silicone Oil**

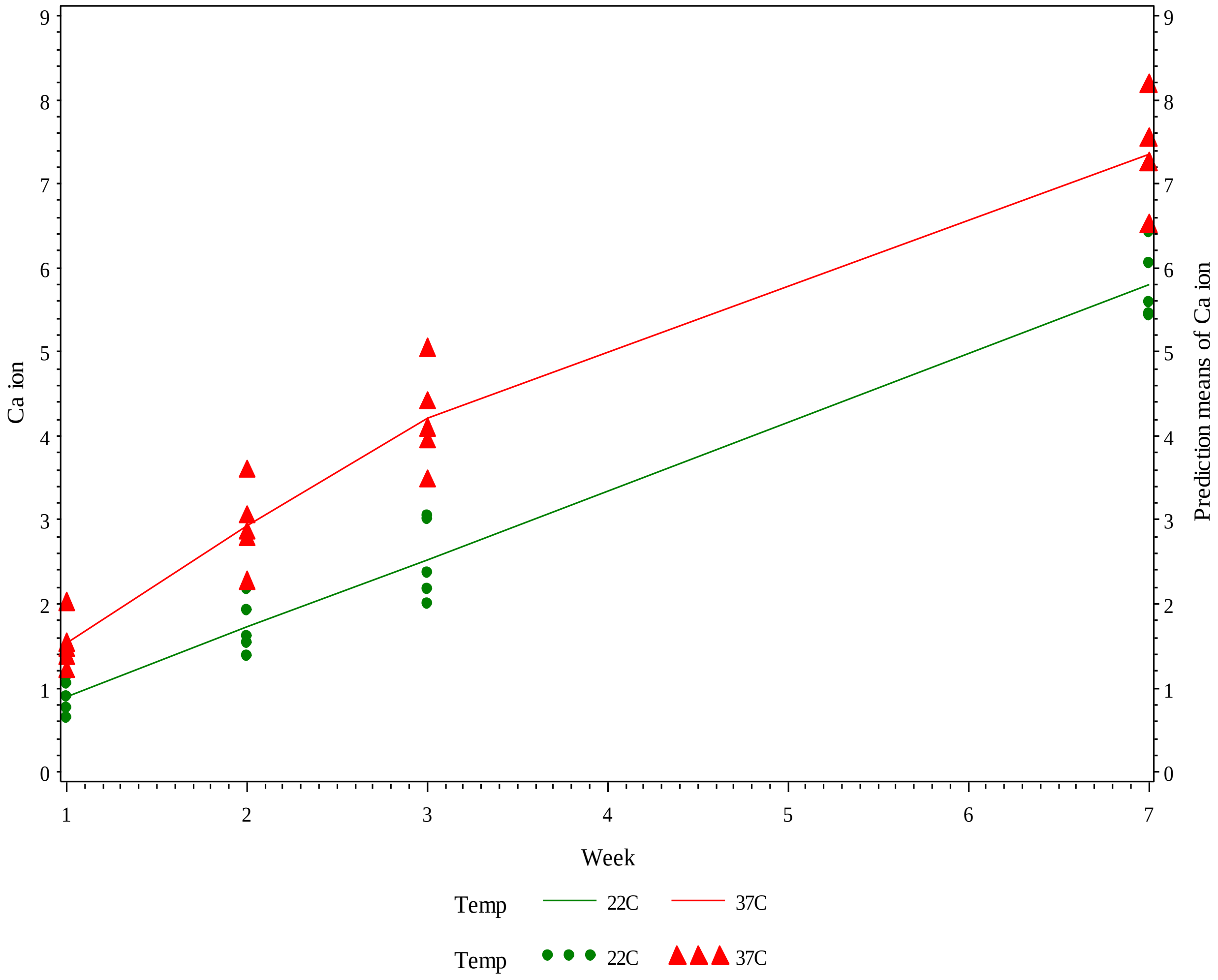

**Figure XIII: $Ca^{2+}$ ions release during the storage time**

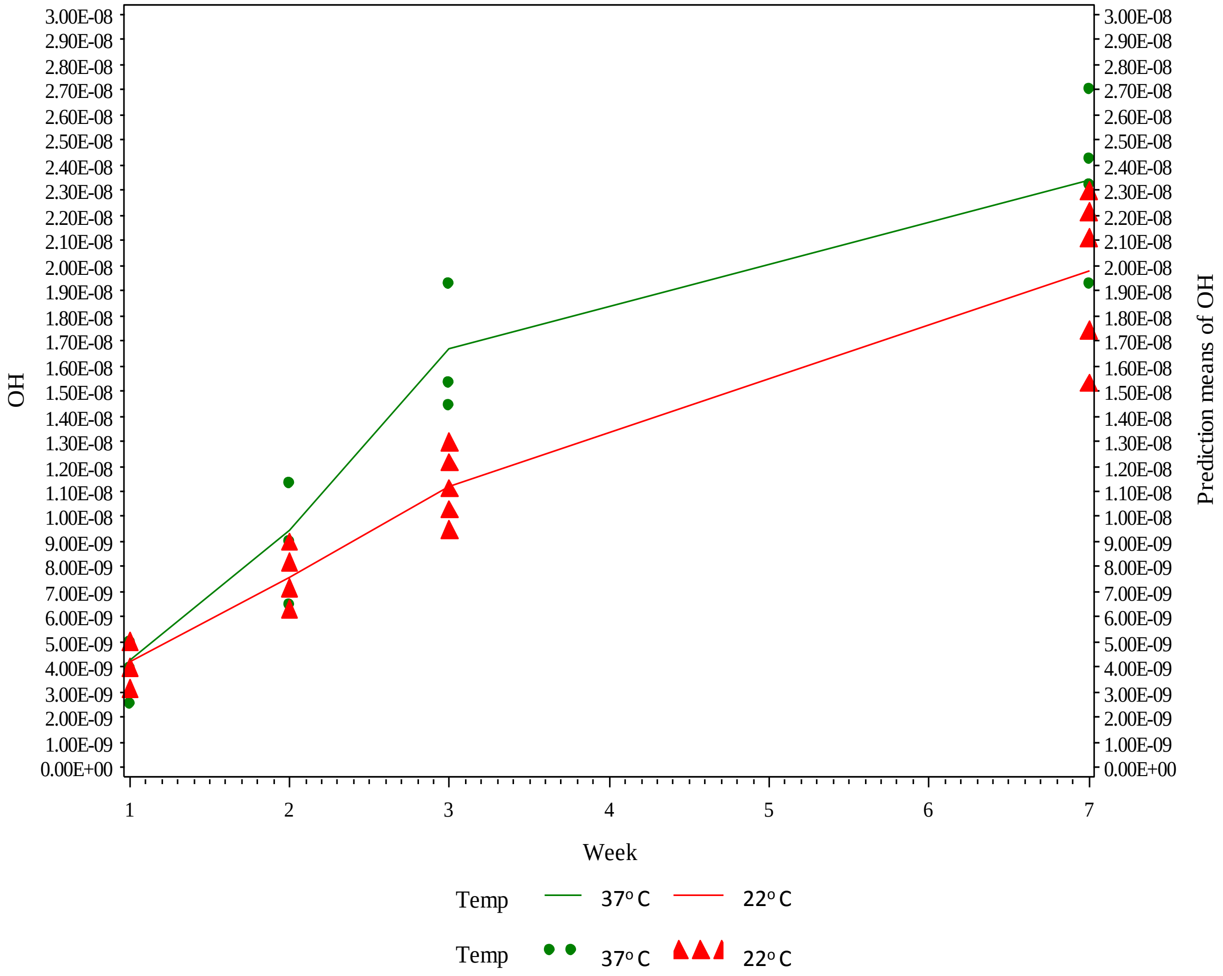

**Figure XIV: OH$^-$ ions release during the Storage time**

**Figure**

**Figure XV: Ceramir C&B (CM) (Doxa Dental AB, Uppsala, Sweden),**

**Figure XVI: Resin-modified GIC (Fuji Plus (FP), GC America, Illinois, USA)**

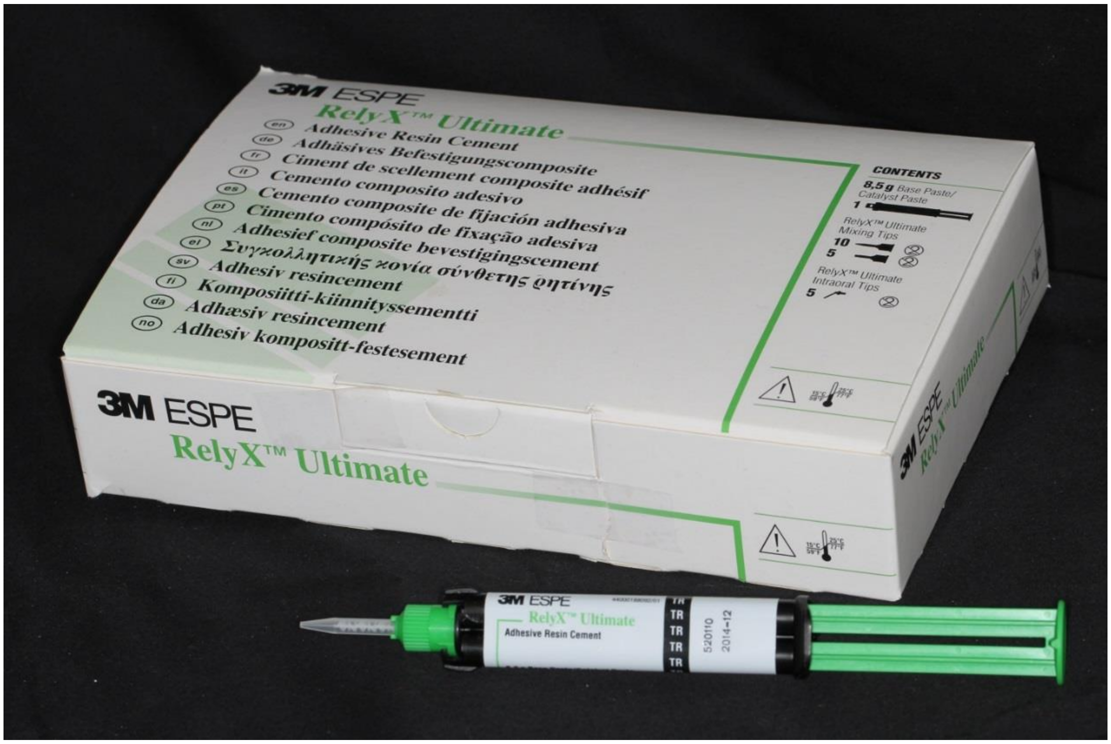

**Figure XVII: Resin luting agent (Rely X Ultimate (RUL), 3M ESPE, CA, USA 3M ESPE, Irvine CA, USA**

**Figure XVIII: Resin luting agents (Rely X Unicem (RU), 3M ESPE, Irvine CA, USA)**

**Figure XIX: Silicone Fluid (100 Cps Silicone Oli –Polydimethylsilxane) Factur II,AZ USA**

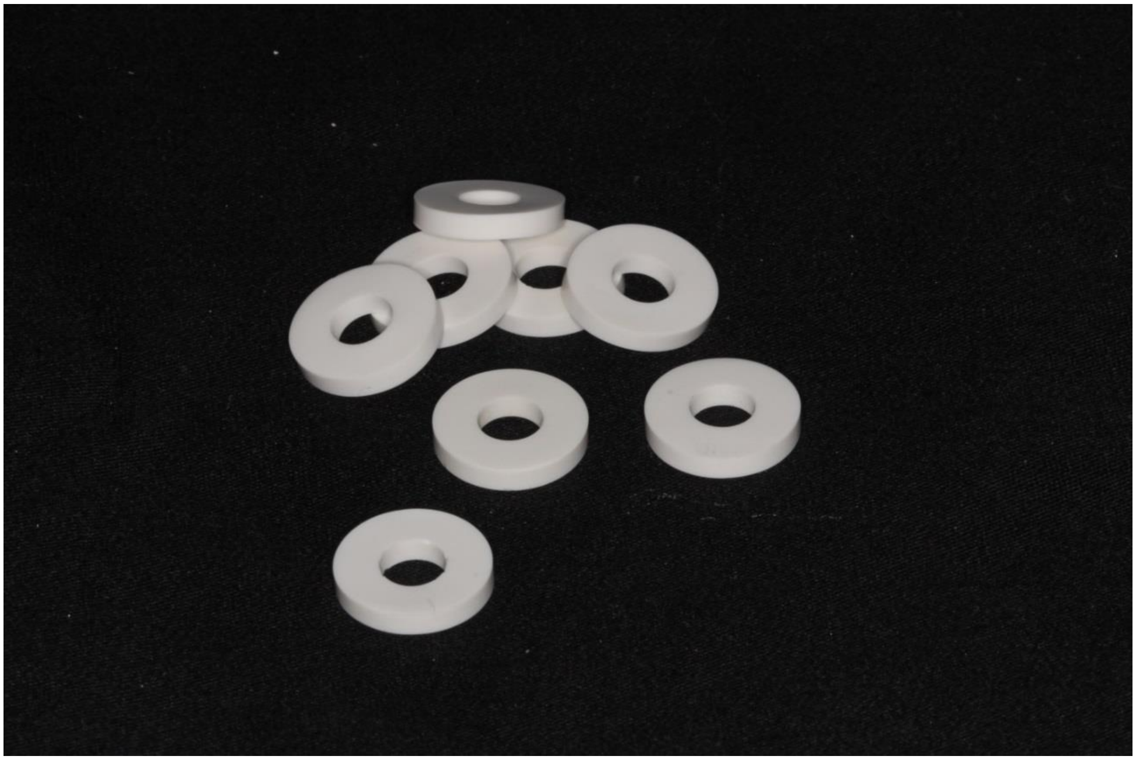

**Figure XX: Cylindrical specimens (7 $^{+}$ 0.1 mm in diameter and 2 $^{+}$ 0.1 mm in height)**

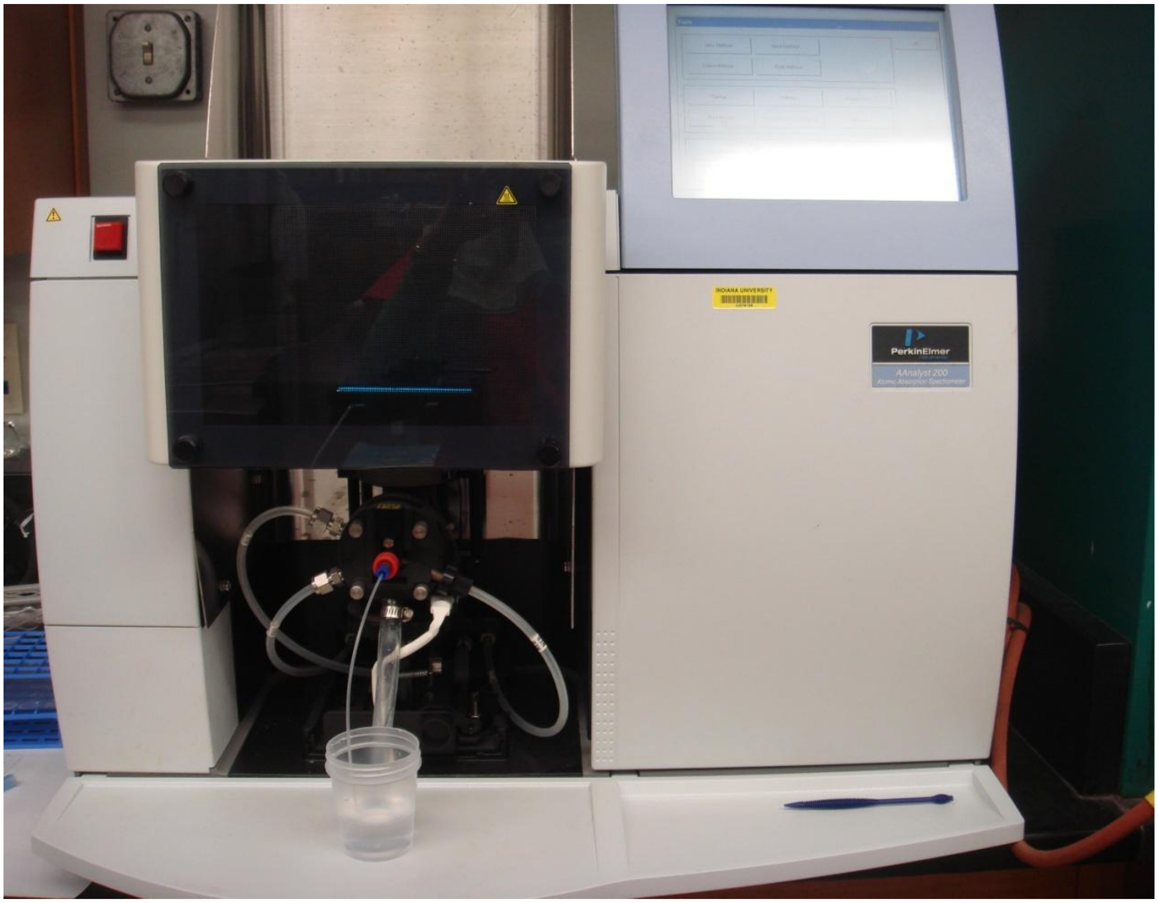

**Figure XXI: Calcium analysis by atomic absorption spectroscopy (Perkin Elmer Analyst 200).**

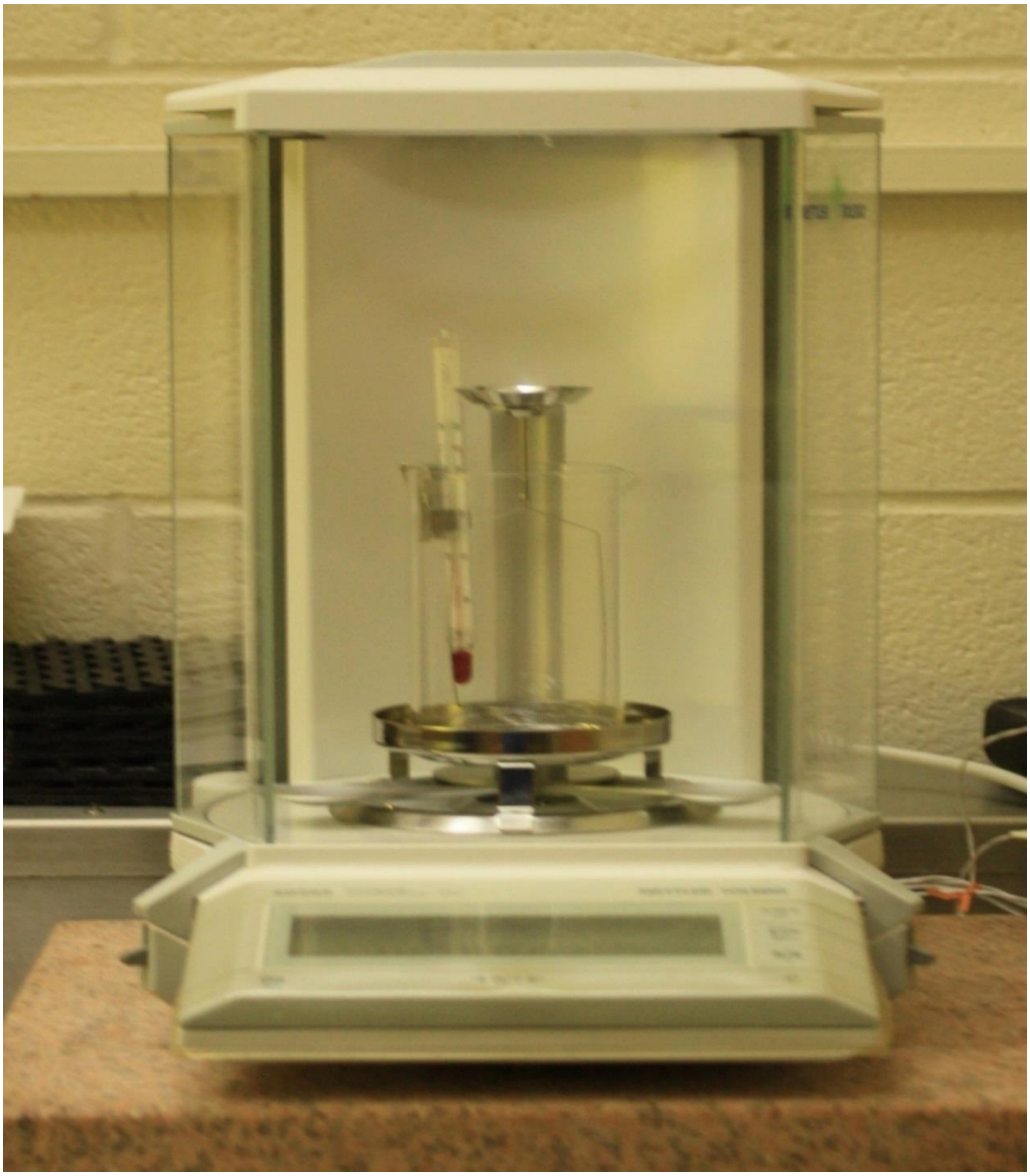

**Figure XXII: A 0.00001- g balance (Model AG285, Mettler Toledo) with an apparatus for weighing specimens in air and in fluid**

# DISCUSSION

Various techniques have been developed to measure the dimensional changes of restorative materials. These include water and mercury dilatometer and transducer methods, which record total shrinkage and are useful for evaluation of deformation of materials during curing.[27,46] Polymerization shrinkage of light-curing dental materials has been measured by using linometers,[46-47] dilatometers,[47] laser inferometric methods,[48] and video-imaging.[49] Buoyancy methods based on Archimedes' principle is well understood and has been used to measure dimensional changes of dental materials.[27,49,50] From previous studies, this method has proven to be a reliable method to study and compare the dimensional change of the same material when immersed in silicone oil and distilled water. Given a body immersed in a fluid, the principle states that the volume of the displaced fluid is equal to the mass up-thrust of the body divided by the density of the fluid. Using Archimedes' principle, the volume change in a luting agent can be computed from the mass up-thrust after immersion in a fluid. Using the value for mass up-thrust, the density before and after the immersion can be computed. This study required precision of the fluid density data, which are temperature-dependent. In particular, this study required fluid density data as a function of temperature with the resolution of at least 0.1°C for the two fluids.

To study the effect of water on the dimensional changes, both distilled water and silicone oil were used. Silicone oil was used to isolate the dimensional changes associated with the setting reaction. The two temperatures 22$^{o}$C and 37$^{o}$C were selected to mimic the clinical situation and laboratory.

To justify the clinical relevance of this study, a typical elapsed time of 10 minutes before immersion of the specimen in the test fluids was used. By the end of this time, the majority of the setting shrinkage would have already occurred.

In silicone oil, it was assumed that the volume loss in the specimens after immersion in the silicone oil was due to ongoing setting contraction, because it was less likely for ions or molecules to move into or out of any material into the silicone oil. In distilled water, it was assumed that the volume gained in the specimen after immersion was due to the effect of water, water sorption and solubility. This study does not account for the solubility of the tested materials. It is possible that the solubility of the materials is significant, in which case the volume gained by water sorption could be offset by the volume loss due to solubility. Further study should take into account the solubility of these materials.

Ceramir immersed in silicone oil exhibited volume loss (0.64 percent at 22$^{o}$C and 2.48 percent at 37$^{o}$C), which is related to the setting reaction. Also, the luting agent showed the highest level of dimensional change in water at both temperatures (17.28 percent and 20.52 percent). After reviewing the results, the null hypothesis was rejected, which proposed that water storage would not significantly impact the dimensional stability of calcium aluminate glass ionomer cement.

Moreover, making a sample for the Ceramir free of the porosity was a challenge. This observation is in agreement with an *in-vitro* study by Geirsson et al. that calcium aluminate and glass ionomer restorative materials contain a higher level of porosity than either amalgam or composite.[40] From the studies of fracture analysis and the correlation between failures and flaws,[51,52] it can be assumed that the presence of the high volume of porosities will affect the mechanical strength of the material. It appears obvious that the samples prepared in the laboratory setting will have less porosity compared with the clinical situations. The RM-GIC exhibited pronounced expansion in water by 6.97 percent at 22° C and 10.45 percent at 37°C. In silicone oil it exhibited the highest shrinkage at 22°C and 37°C by 2.83 percent and 6.45 percent, respectively. The behavior was generally in agreement with results from Watts et al. with volumetric expansions in water ranging between 5.1 percent and 6.8 percent observed on this product in those studies.[26] Moreover, Attin et al. concluded in their study that water storage of resin-modified glass ionomer cement resulted in significant volumetric changes.[27] Most RM-GICs contain, in addition to the essential components of conventional GICs, photocurable resins (usually 2-hydroxyethyl methacrylate) which replace part of the water. This resin is a known synthetic hydrogel[53] and has been shown to absorb water and consequently is associated with the expansion of these resin-ionomers. The characteristic sudden expansion, attaining a maximum in one day and remaining nearly constant, may be attributed to the amount of this hydrophilic resin being in the range 4.5 percent to 6.0 percent.[54] The volumetric shrinkage noted agreed closely with that reported in some previous studies.[55,56]

The increase in dimension may be beneficial in relieving some of the internal polymerization shrinkage stresses and increasing the longevity of the adhesive union to the surrounding tooth. As Momoi and McCabe suggested, hygroscopic expansion may close any gaps and relieve interface stresses that have been generated by setting shrinkage.[19] On the other hand, if the  hygroscopic expansion approximates or exceeds the setting shrinkage, it might be a concern for some clinical situations, such as in luting a cast post and core or inlay restorations. Significant expansion may cause an outward force against cavity walls causing cracking and fracture of the tooth. Furthermore, significant expansion might lead to the failure of full coverage restorations fabricated from ceramic. Sindel et al. concluded that resin modified glass ionomer and compomer used as a core or luting agent produces failure of all ceramic crowns because of the associated hygroscopic expansion.[5]

Finally, the self-adhesive resin showed non-significant dimensional changes compared with the conventional resin-based luting materials. This was not expected due to the chemistry of such materials; they are expected to be somewhat more hydrophilic, improving wettability and penetration into the tooth structures. They become less hydrophilic and more hydrophobic during the polymerization reaction. But, compared with the conventional resin-based luting materials, they experience inferior hydrolytic stability.

Covering the resin matrix composition of the self-adhesive luting materials, UDMA polymers show significantly more water uptake than polymers based on non-hydroxylated Bis-GMA analogues. HEMA polymers also have a more hydrophilic portion that leads to more water sorption compared with the BisGMA polymers. It was found that water can induce stresses between the different phases in the studied resin luting agent.[57]

## $Ca^+$ and $OH^-$ RELEASE

Chemical analysis of the storage medium solution (distilled water) of the CM cement was performed using atomic absorption spectroscopy to determine the $Ca^+$ ions released. In general, the ion release increased significantly with increasing immersion time and as the temperature increased.

The pH values were measured to determine the $OH^-$ concentration in the solutions. The results show that the pH value increased with time and also as the temperature increased. These indicate that the solutions become more basic as time elapses. The results show the total amount of both ions is significant at the end of storage time. The findings lead to rejection of the null hypothesis that calcium aluminate glass ionomer cement will not have noticeable $Ca^{+2}$ and $OH^-$ release.

# SUMMARY AND CONCLUSIONS

This *in-vitro* study was conducted to evaluate the volumetric dimensional changes of four luting agents for four periods of time using Archimedes' principle to measure the change. The application of Archimedes' principle is well understood and it has been proposed as a method for determination of the volumetric dimensional changes of restorative material.

The results indicate that the temperature and the time had significant impact on volumetric dimensional changes and also on the ions released. Luting agents stored in water at both 22°C and 37°C had an increase in volume. Ceramir showed the highest level of dimensional changes (17.28 percent and 20.52 percent) and both of the resin luting agents expanded the least without a significant difference between them. However, luting agents stored in silicon oil exhibit shrinkage at both temperatures. Resin modified glass ionomer exhibited the highest shrinkage at 22°C and 37°C by 2.83 percent and 6.45 percent, respectively.

For all luting agents, most of the volumetric dimensional changes happened during the first week of storage. The clinical implications of the results in the present study are uncertain. Ceramir luting agent expands significantly in water and continues to release ions. The luting agent released significant $Ca^{2+}$ ions that were significantly increased with time and at higher temperature. Further study should take into account the solubility of these materials and confirm whether the assumption made in this study is valid. Furthermore, long-term effects of water storage on the success of the restoration need to be determined in future work.

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# APPENDIX

## TABLE V
## ANOVA

| Effect | Num DF | Den DF | F Value | P Value |
|---|---|---|---|---|
| Week | 3 | 64 | 4.35 | 0.0075 |
| Material | 3 | 64 | 114.46 | <.0001 |
| Week*Material | 9 | 64 | 1.48 | 0.1736 |
| Media | 1 | 64 | 598.83 | <.0001 |
| Week*Media | 3 | 64 | 7.38 | 0.0003 |
| Material*Media | 3 | 64 | 115.67 | <.0001 |
| Temp | 1 | 64 | 0.03 | 0.8625 |
| Week*Temp | 3 | 64 | 2.89 | 0.0420 |
| Material*Temp | 3 | 64 | 1.99 | 0.1241 |
| Media*Temp | 1 | 64 | 31.47 | <.0001 |
| Week*Material*Media | 9 | 64 | 2.57 | 0.0136 |
| Week*Media*Temp | 3 | 64 | 6.02 | 0.0011 |
| Material*Media*Temp | 3 | 64 | 2.86 | 0.0438 |

## Table VI
## Comparisons for material

| Result | Control for | Difference | Standard Error | P value |
|---|---|---|---|---|
| Ceramir & Fuji n.s. | 22C,Oil | 1.66 | 1.08 | 0.1277 |
| Ceramir > Fuji | 37C,Oil | 4.09 | 1.08 | 0.0003 |
| Ceramir > Fuji | 22C,Water | 9.14 | 1.08 | <.0001 |
| Ceramir > Fuji | 37C,Water | 10.16 | 1.08 | <.0001 |
| Ceramir > RelayUL | 22C,Water | 14.89 | 1.08 | <.0001 |
| Ceramir > RelayUL | 37C,Water | 19.24 | 1.08 | <.0001 |
| Ceramir & RelayUL n.s. | 22C,Oil | 0.04 | 1.08 | 0.9703 |
| Ceramir & RelayUL n.s. | 37C,Oil | 0.43 | 1.08 | 0.6948 |
| Ceramir > RelayUL | Week1,Water | 17.56 | 0.92 | <.0001 |
| Ceramir > RelayUL | Week2,Water | 16.81 | 0.77 | <.0001 |
| Ceramir > RelayUL | Week3,Water | 16.14 | 0.79 | <.0001 |
| Ceramir > RelayUL | Week4,Water | 17.75 | 0.82 | <.0001 |
| Ceramir > RelayUL | Water | 17.07 | 0.77 | <.0001 |
| Ceramir & RelayUL n.s. | Oil | 0.23 | 0.77 | 0.7638 |
| Ceramir & RelayUL n.s. | Week1,Oil | 0.07 | 0.92 | 0.9397 |
| Ceramir & RelayUL n.s. | Week2,Oil | 0.07 | 0.77 | 0.9231 |
| Ceramir & RelayUL n.s. | Week3,Oil | 0.54 | 0.79 | 0.4935 |
| Ceramir & RelayUL n.s. | Week4,Oil | 0.24 | 0.82 | 0.7661 |
| Ceramir & RelayUN n.s. | 22C,Oil | 0.05 | 1.08 | 0.9620 |
| Ceramir & RelayUN n.s. | 37C,Oil | 0.57 | 1.08 | 0.5975 |
| Ceramir > RelayUN | 22C,Water | 15.50 | 1.08 | <.0001 |

| Result | Control for | Difference | Standard Error | P value |
|---|---|---|---|---|
| Ceramir > RelayUN | 37C,Water | 19.16 | 1.08 | <.0001 |
| Ceramir > RelayUN | Week1,Water | 17.67 | 0.92 | <.0001 |
| Ceramir > RelayUN | Week2,Water | 16.86 | 0.77 | <.0001 |
| Ceramir > RelayUN | Week3,Water | 16.85 | 0.79 | <.0001 |
| Ceramir > RelayUN | Week4,Water | 17.94 | 0.82 | <.0001 |
| Ceramir > RelayUN | Water | 17.33 | 0.77 | <.0001 |
| Ceramir & RelayUN n.s. | Oil | 0.31 | 0.77 | 0.6872 |
| Ceramir & RelayUN n.s. | Week1,Oil | 0.15 | 0.92 | 0.8678 |
| Ceramir & RelayUN n.s. | Week2,Oil | 0.46 | 0.77 | 0.5547 |
| Ceramir & RelayUN n.s. | Week3,Oil | 0.40 | 0.79 | 0.6098 |
| Ceramir & RelayUN n.s. | Week4,Oil | 0.23 | 0.82 | 0.7769 |
| Fuji > RelayUL | Water | 7.42 | 0.77 | <.0001 |
| Fuji < RelayUL | Week1,Oil | -2.39 | 0.92 | 0.0115 |
| Fuji < RelayUL | Week2,Oil | -2.79 | 0.77 | 0.0006 |
| Fuji < RelayUL | Week3,Oil | -2.57 | 0.79 | 0.0018 |
| Fuji < RelayUL | Week4,Oil | -2.83 | 0.82 | 0.0010 |
| Fuji > RelayUL | Week1,Water | 7.63 | 0.92 | <.0001 |
| Fuji > RelayUL | Week2,Water | 7.72 | 0.77 | <.0001 |
| Fuji > RelayUL | Week3,Water | 6.76 | 0.79 | <.0001 |
| Fuji > RelayUL | Week4,Water | 7.56 | 0.82 | <.0001 |
| Fuji & RelayUL n.s. | 22C,Oil | -1.62 | 1.08 | 0.1370 |
| Fuji < RelayUL | 37C,Oil | -3.66 | 1.08 | 0.0012 |
| Fuji > RelayUL | 22C,Water | 5.75 | 1.08 | <.0001 |
| Fuji > RelayUL | 37C,Water | 9.09 | 1.08 | <.0001 |
| Fuji > RelayUN | Water | 7.68 | 0.77 | <.0001 |
| Fuji < RelayUN | Week1,Oil | -2.30 | 0.92 | 0.0145 |
| Fuji < RelayUN | Week2,Oil | -2.41 | 0.77 | 0.0027 |
| Fuji < RelayUN | Week3,Oil | -2.70 | 0.79 | 0.0010 |
| Fuji < RelayUN | Week4,Oil | -2.84 | 0.82 | 0.0009 |
| Fuji > RelayUN | Week1,Water | 7.74 | 0.92 | <.0001 |
| Fuji > RelayUN | Week2,Water | 7.77 | 0.77 | <.0001 |
| Fuji > RelayUN | Week3,Water | 7.47 | 0.79 | <.0001 |
| Fuji > RelayUN | Week4,Water | 7.76 | 0.82 | <.0001 |
| Fuji & RelayUN n.s. | 22C,Oil | -1.61 | 1.08 | 0.1398 |
| Fuji < RelayUN | 37C,Oil | -3.52 | 1.08 | 0.0018 |
| Fuji > RelayUN | 22C,Water | 6.36 | 1.08 | <.0001 |
| Fuji > RelayUN | 37C,Water | 9.01 | 1.08 | <.0001 |
| RelayUL & RelayUN n.s. | 22C,Oil | 0.01 | 1.08 | 0.9916 |
| RelayUL & RelayUN n.s. | 37C,Oil | 0.15 | 1.08 | 0.8918 |
| RelayUL & RelayUN n.s. | 22C,Water | 0.61 | 1.08 | 0.5723 |
| RelayUL & RelayUN n.s. | 37C,Water | -0.08 | 1.08 | 0.9386 |
| RelayUL & RelayUN n.s. | Week1 | 0.10 | 0.65 | 0.8829 |
| RelayUL & RelayUN n.s. | Week2 | 0.22 | 0.55 | 0.6940 |
| RelayUL & RelayUN n.s. | Week3 | 0.28 | 0.56 | 0.6110 |

| Result | Control for | Difference | Standard Error | P value |
|---|---|---|---|---|
| RelayUL & RelayUN n.s. | Week4 | 0.09 | 0.58 | 0.8747 |
| RelayUL & RelayUN n.s. | Oil | 0.08 | 0.77 | 0.9184 |
| RelayUL & RelayUN n.s. | Water | 0.26 | 0.77 | 0.7328 |
| RelayUL & RelayUN n.s. | 22C | 0.31 | 0.76 | 0.6841 |
| RelayUL & RelayUN n.s. | 37C | 0.03 | 0.76 | 0.9667 |
| RelayUL & RelayUN n.s. | Week1,Oil | 0.08 | 0.92 | 0.9277 |
| RelayUL & RelayUN n.s. | Week1,Water | 0.11 | 0.92 | 0.9064 |
| RelayUL & RelayUN n.s. | Week2,Oil | 0.38 | 0.77 | 0.6209 |
| RelayUL & RelayUN n.s. | Week2,Water | 0.05 | 0.77 | 0.9509 |
| RelayUL & RelayUN n.s. | Week3,Oil | -0.14 | 0.79 | 0.8610 |
| RelayUL & RelayUN n.s. | Week3,Water | 0.71 | 0.79 | 0.3722 |
| RelayUL & RelayUN n.s. | Week4,Oil | -0.01 | 0.82 | 0.9887 |
| RelayUL & RelayUN n.s. | Week4,Water | 0.20 | 0.82 | 0.8125 |

Table VII
Comparisons for week

| Result | Control for | Difference | Standard Error | P value |
|---|---|---|---|---|
| Week1 & Week2 n.s. | 37C | 0.05 | 0.19 | 0.7941 |
| Week1 & Week2 n.s. | Fuji | 0.15 | 0.27 | 0.5848 |
| Week1 & Week2 n.s. | RelayUL | -0.01 | 0.27 | 0.9667 |
| Week1 & Week2 n.s. | RelayUN | 0.11 | 0.27 | 0.6897 |
| Week1 & Week2 n.s. | Oil | 0.03 | 0.19 | 0.8742 |
| Week1 & Week2 n.s. | Ceramir,Oil | -0.15 | 0.38 | 0.6965 |
| Week1 & Week2 n.s. | Fuji,Oil | 0.26 | 0.38 | 0.4979 |
| Week1 & Week2 n.s. | Fuji,Water | 0.04 | 0.38 | 0.9246 |
| Week1 & Week2 n.s. | RelayUL,Oil | -0.14 | 0.38 | 0.7064 |
| Week1 & Week2 n.s. | RelayUL,Water | 0.12 | 0.38 | 0.7507 |
| Week1 & Week2 n.s. | RelayUN,Oil | 0.16 | 0.38 | 0.6858 |
| Week1 & Week2 n.s. | RelayUN,Water | 0.06 | 0.38 | 0.8729 |
| Week1 > Week2 | Ceramir,Water | 0.87 | 0.38 | 0.0259 |
| Week1 & Week2 n.s. | 22C,Oil | -0.07 | 0.27 | 0.7986 |
| Week1 & Week2 n.s. | 37C,Oil | 0.13 | 0.27 | 0.6321 |
| Week1 & Week2 n.s. | 37C,Water | -0.03 | 0.27 | 0.9124 |
| Week1 > Week2 | 22C,Water | 0.58 | 0.27 | 0.0371 |
| Week1 > Week3 | 37C,Oil | 0.77 | 0.31 | 0.0153 |
| Week1 > Week3 | 22C,Water | 1.11 | 0.31 | 0.0007 |
| Week1 & Week3 n.s. | 22C,Oil | 0.17 | 0.31 | 0.5801 |
| Week1 & Week3 n.s. | 37C,Water | -0.36 | 0.31 | 0.2552 |
| Week1 > Week3 | Ceramir,Water | 1.07 | 0.44 | 0.0171 |
| Week1 & Week3 n.s. | Ceramir,Oil | 0.13 | 0.44 | 0.7696 |
| Week1 & Week3 n.s. | Fuji,Oil | 0.78 | 0.44 | 0.0799 |
| Week1 & Week3 n.s. | Fuji,Water | 0.53 | 0.44 | 0.2346 |
| Week1 & Week3 n.s. | RelayUL,Oil | 0.60 | 0.44 | 0.1752 |

| Result | Control for | Difference | Standard Error | P value |
|---|---|---|---|---|
| Week1 & Week3 n.s. | RelayUL,Water | -0.35 | 0.44 | 0.4316 |
| Week1 & Week3 n.s. | RelayUN,Oil | 0.38 | 0.44 | 0.3905 |
| Week1 & Week3 n.s. | RelayUN,Water | 0.25 | 0.44 | 0.5681 |
| Week1 & Week3 n.s. | RelayUL | 0.13 | 0.31 | 0.6834 |
| Week1 & Week3 n.s. | RelayUN | 0.32 | 0.31 | 0.3129 |
| Week1 & Week4 n.s. | 22C,Oil | 0.19 | 0.29 | 0.5151 |
| Week1 & Week4 n.s. | 22C,Water | 0.07 | 0.29 | 0.8185 |
| Week1 & Week4 n.s. | 37C,Water | -0.29 | 0.29 | 0.3216 |
| Week1 & Week4 n.s. | 22C | 0.13 | 0.21 | 0.5337 |
| Week1 & Week4 n.s. | Ceramir | -0.04 | 0.29 | 0.8931 |
| Week1 & Week4 n.s. | RelayUL | 0.14 | 0.29 | 0.6293 |
| Week1 & Week4 n.s. | RelayUN | 0.14 | 0.29 | 0.6392 |
| Week1 & Week4 n.s. | Water | -0.11 | 0.21 | 0.5887 |
| Week1 & Week4 n.s. | Ceramir,Oil | 0.21 | 0.41 | 0.6089 |
| Week1 & Week4 n.s. | Ceramir,Water | -0.29 | 0.41 | 0.4834 |
| Week1 & Week4 n.s. | Fuji,Water | -0.04 | 0.41 | 0.9315 |
| Week1 & Week4 n.s. | RelayUL,Oil | 0.39 | 0.41 | 0.3511 |
| Week1 & Week4 n.s. | RelayUL,Water | -0.10 | 0.41 | 0.8008 |
| Week1 & Week4 n.s. | RelayUN,Oil | 0.29 | 0.41 | 0.4813 |
| Week1 & Week4 n.s. | RelayUN,Water | -0.02 | 0.41 | 0.9665 |
| Week1 > Week4 | Fuji,Oil | 0.83 | 0.41 | 0.0481 |
| Week1 > Week4 | 37C,Oil | 0.67 | 0.29 | 0.0248 |
| Week2 & Week3 n.s. | Ceramir | 0.24 | 0.19 | 0.2053 |
| Week2 & Week3 n.s. | RelayUN | 0.21 | 0.19 | 0.2741 |
| Week2 & Week3 n.s. | Ceramir,Oil | 0.28 | 0.27 | 0.2972 |
| Week2 & Week3 n.s. | Ceramir,Water | 0.20 | 0.27 | 0.4508 |
| Week2 & Week3 n.s. | Fuji,Oil | 0.52 | 0.27 | 0.0545 |
| Week2 & Week3 n.s. | Fuji,Water | 0.49 | 0.27 | 0.0694 |
| Week2 & Week3 n.s. | RelayUL,Water | -0.47 | 0.27 | 0.0818 |
| Week2 & Week3 n.s. | RelayUN,Oil | 0.22 | 0.27 | 0.4023 |
| Week2 & Week3 n.s. | RelayUN,Water | 0.19 | 0.27 | 0.4761 |
| Week2 > Week3 | RelayUL,Oil | 0.75 | 0.27 | 0.0065 |
| Week2 & Week3 n.s. | 22C,Oil | 0.24 | 0.19 | 0.2022 |
| Week2 & Week3 n.s. | 37C,Water | -0.33 | 0.19 | 0.0869 |
| Week2 > Week3 | 37C,Oil | 0.64 | 0.19 | 0.0011 |
| Week2 > Week3 | 22C,Water | 0.53 | 0.19 | 0.0061 |
| Week2 > Week4 | 37C,Oil | 0.54 | 0.17 | 0.0022 |
| Week2 & Week4 n.s. | 22C,Oil | 0.26 | 0.17 | 0.1296 |
| Week2 & Week4 n.s. | 37C,Water | -0.26 | 0.17 | 0.1276 |
| Week2 < Week4 | 22C,Water | -0.51 | 0.17 | 0.0039 |
| Week2 > Week4 | Fuji,Oil | 0.57 | 0.24 | 0.0202 |
| Week2 > Week4 | RelayUL,Oil | 0.53 | 0.24 | 0.0299 |
| Week2 & Week4 n.s. | Ceramir,Oil | 0.36 | 0.24 | 0.1360 |
| Week2 & Week4 n.s. | Fuji,Water | -0.07 | 0.24 | 0.7652 |

| Result | Control for | Difference | Standard Error | P value |
|---|---|---|---|---|
| Week2 & Week4 n.s. | RelayUL,Water | -0.23 | 0.24 | 0.3482 |
| Week2 & Week4 n.s. | RelayUN,Oil | 0.14 | 0.24 | 0.5698 |
| Week2 & Week4 n.s. | RelayUN,Water | -0.08 | 0.24 | 0.7433 |
| Week2 < Week4 | Ceramir,Water | -1.16 | 0.24 | <.0001 |
| Week2 & Week4 n.s. | RelayUN | 0.03 | 0.17 | 0.8644 |
| Week3 & Week4 n.s. | 22C,Oil | 0.02 | 0.17 | 0.9141 |
| Week3 & Week4 n.s. | 37C,Oil | -0.10 | 0.17 | 0.5589 |
| Week3 & Week4 n.s. | 37C,Water | 0.06 | 0.17 | 0.7100 |
| Week3 < Week4 | 22C,Water | -1.04 | 0.17 | <.0001 |
| Week3 & Week4 n.s. | Ceramir,Oil | 0.08 | 0.24 | 0.7335 |
| Week3 & Week4 n.s. | Fuji,Oil | 0.05 | 0.24 | 0.8334 |
| Week3 & Week4 n.s. | RelayUL,Oil | -0.21 | 0.24 | 0.3847 |
| Week3 & Week4 n.s. | RelayUL,Water | 0.24 | 0.24 | 0.3235 |
| Week3 & Week4 n.s. | RelayUN,Oil | -0.09 | 0.24 | 0.7233 |
| Week3 & Week4 n.s. | RelayUN,Water | -0.27 | 0.24 | 0.2736 |
| Week3 < Week4 | Ceramir,Water | -1.36 | 0.24 | <.0001 |
| Week3 < Week4 | Fuji,Water | -0.56 | 0.24 | 0.0244 |
| Week3 & Week4 n.s. | RelayUL | 0.01 | 0.17 | 0.9328 |
| Week3 & Week4 n.s. | RelayUN | -0.18 | 0.17 | 0.3058 |
| Week3 & Week4 n.s. | Oil | -0.04 | 0.12 | 0.7358 |
| Week3 & Week4 n.s. | 37C | -0.02 | 0.12 | 0.8802 |

Table VIII
Comparisons for storage media

| Result | Control for | Difference | Standard Error | P value |
|---|---|---|---|---|
| Oil < Water | Ceramir | -19.59 | 0.77 | <.0001 |
| Oil < Water | Fuji | -12.82 | 0.77 | <.0001 |
| Oil < Water | 37C | -11.60 | 0.55 | <.0001 |
| Oil < Water | Week1,Ceramir | -19.96 | 0.92 | <.0001 |
| Oil < Water | Week1,Fuji | -12.48 | 0.92 | <.0001 |
| Oil < Water | Week1,RelayUL | -2.46 | 0.92 | 0.0092 |
| Oil < Water | Week1,RelayUN | -2.44 | 0.92 | 0.0099 |
| Oil < Water | Week2,Ceramir | -18.94 | 0.77 | <.0001 |
| Oil < Water | Week2,Fuji | -12.71 | 0.77 | <.0001 |
| Oil < Water | Week2,RelayUL | -2.20 | 0.77 | 0.0059 |

| Result | Control for | Difference | Standard Error | P value |
|---|---|---|---|---|
| Oil < Water | Week2,RelayUN | -2.53 | 0.77 | 0.0017 |
| Oil < Water | Week3,Ceramir | -19.01 | 0.79 | <.0001 |
| Oil < Water | Week3,Fuji | -12.74 | 0.79 | <.0001 |
| Oil < Water | Week3,RelayUL | -3.41 | 0.79 | <.0001 |
| Oil < Water | Week3,RelayUN | -2.57 | 0.79 | 0.0018 |
| Oil < Water | Week4,Ceramir | -20.46 | 0.82 | <.0001 |
| Oil < Water | Week4,Fuji | -13.35 | 0.82 | <.0001 |
| Oil < Water | Week4,RelayUL | -2.96 | 0.82 | 0.0006 |
| Oil < Water | Week4,RelayUN | -2.75 | 0.82 | 0.0014 |
| Oil < Water | Week1,22C | -7.64 | 0.65 | <.0001 |
| Oil < Water | Week1,37C | -11.03 | 0.65 | <.0001 |
| Oil < Water | Week2,22C | -6.99 | 0.55 | <.0001 |
| Oil < Water | Week2,37C | -11.19 | 0.55 | <.0001 |
| Oil < Water | Week3,22C | -6.70 | 0.56 | <.0001 |
| Oil < Water | Week3,37C | -12.16 | 0.56 | <.0001 |
| Oil < Water | Week4,22C | -7.76 | 0.58 | <.0001 |
| Oil < Water | Week4,37C | -12.00 | 0.58 | <.0001 |
| Oil & Water n.s. | RelayUL,22C | -1.87 | 1.08 | 0.0892 |
| Oil & Water n.s. | RelayUN,22C | -1.27 | 1.08 | 0.2461 |
| Oil < Water | Ceramir,22C | -16.72 | 1.08 | <.0001 |
| Oil < Water | Ceramir,37C | -22.47 | 1.08 | <.0001 |
| Oil < Water | Fuji,22C | -9.24 | 1.08 | <.0001 |
| Oil < Water | Fuji,37C | -16.40 | 1.08 | <.0001 |
| Oil < Water | RelayUL,37C | -3.65 | 1.08 | 0.0013 |
| Oil < Water | RelayUN,37C | -3.88 | 1.08 | 0.0007 |

Table IX
Comparisons for temperature

| Result | Control for | Difference | Standard Error | P value |
|---|---|---|---|---|
| 22C & 37C n.s. | RelayUL | 0.89 | 0.76 | 0.2436 |
| 22C & 37C n.s. | RelayUN | 0.61 | 0.76 | 0.4222 |
| 22C < 37C | Week1,Water | -1.49 | 0.65 | 0.0249 |
| 22C < 37C | Week2,Water | -2.09 | 0.55 | 0.0003 |
| 22C < 37C | Week3,Water | -2.95 | 0.56 | <.0001 |
| 22C < 37C | Week4,Water | -1.85 | 0.58 | 0.0022 |
| 22C > 37C | Week1,Oil | 1.91 | 0.65 | 0.0045 |

| Result | Control for | Difference | Standard Error | P value |
|---|---|---|---|---|
| 22C > 37C | Week2,Oil | 2.11 | 0.55 | 0.0003 |
| 22C > 37C | Week3,Oil | 2.51 | 0.56 | <.0001 |
| 22C > 37C | Week4,Oil | 2.39 | 0.58 | 0.0001 |
| 22C & 37C n.s. | Ceramir,Oil | 1.40 | 1.07 | 0.1972 |
| 22C & 37C n.s. | RelayUL,Oil | 1.78 | 1.07 | 0.1014 |
| 22C & 37C n.s. | RelayUL,Water | 0.00 | 1.07 | 0.9982 |
| 22C & 37C n.s. | RelayUN,Oil | 1.92 | 1.07 | 0.0784 |
| 22C & 37C n.s. | RelayUN,Water | -0.69 | 1.07 | 0.5204 |
| 22C < 37C | Ceramir,Water | -4.35 | 1.07 | 0.0001 |
| 22C < 37C | Fuji,Water | -3.34 | 1.07 | 0.0028 |
| 22C > 37C | Fuji,Oil | 3.82 | 1.07 | 0.0007 |

# ABSTRACT

# VOLUMETRIC DIMENSIONAL CHANGES

# OF LUTING CEMENTS

by

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The luting agent is a crucial factor in the outcome of cemented fixed restorations. A new water-based cement, Ceramir C&B (CM), approved to be marketed in the US, is composed of calcium aluminate and glass ionomer. CM is a luting agent indicated for permanent cementation of cast restorations, all-zirconia or all-alumina crowns, and prefabricated metal and cast dowel and cores. The manufacturer claims

that the cement has demonstrated favorable biocompatibility properties when tested *in vitro* and *in vivo* and has proven to be bioactive.

The objective of this study was to evaluate volumetric dimensional changes and the amount of $Ca^{2+}$ released by the new luting agent. Twenty specimens of each material, namely calcium aluminate glass ionomer, resin-modified glass ionomer, and two resin luting agents, were fabricated and weighed. The 20 specimens for all materials were divided into four groups (five samples in each group) based on storage conditions: silicone oil at 22°C and 37°C and distilled water at 22°C and 37°C. Using the manufacturers' instructions for each material, cylindrical specimens were prepared with dimensions of $7 ^{\pm} 0.1$ mm in diameter and $2 ^{\pm} 0.1$ mm in height. A 0.01-mg resolution balance was used to determine volumetric dimensional change using an Archimedean equation. Measurements were made 30 minutes after mixing, and at the time intervals of 7 days, 14 days, 21 days, and 30 days, and after total dehydration of the specimen. Chemical analyses of the solutions were performed using atomic absorption spectroscopy to determine the $Ca^{+2}$ ion concentration. Moreover, the pH values were measured to determine the $OH^{–}$ concentration in the solutions.

The results showed that CM had the most expansion among the tested luting agents in distilled water at 22°C and 37°C, and significantly increased at higher temperature. In silicone oil, resin-modified glass ionomer shrank the most and also shrank more with the high temperature. The result of the ion concentration analysis indicated that $Ca^{+2}$ and $OH^{-}$ ion release increased with increasing time and also significantly with temperature rise. In conclusion, calcium aluminate-glass ionomer exhibited the most significant dimensional change when stored in water storage. The solubility of the tested luting agents should be evaluated in the future because they were not evaluated in this study. Furthermore, to evaluate the clinical effect of the dimensional changes, the impact on the gap formation at tooth-crown margins should be determined in future work.

# CURRICULUM VITAE

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PROFESSIONAL ORGANIZATIONS

American Academy of Implant Dentistry
American College of Prosthodontists (ACP)
American Academy of Maxillofacial Prosthetics (AAMP)
John F. Johnston Society (JFJ)
The Saudi Commission for Health Specialties
Saudi Dental Society (SDS)