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Effect of Salivary pH on Water Absorption and Solubility of Enhanced Resin- Modified Glass Ionomer

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Cover Page Footnote

We would like to thank GC Asia for providing the materials Fuji II LC.

ORIGINAL ARTICLE

Effect of Salivary pH on Water Absorption and Solubility of Enhanced Resin-Modified Glass Ionomer

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ABSTRACT

The influence of immersion period and liquid pH on water absorption capacity and solubility of the resin-modified glass ionomer cement (RMGIC) raises the question of whether the critical pH of hydroxyapatite and fluoroapatite influences the water absorption capacity and solubility of enhanced resin-modified glass ionomer (ERMGI). **Objective:** This study was designed to investigate the effects of immersion periods and various pH levels of artificial saliva on the water absorption and solubility of RMGIC and ERMGI. **Methods:** Fifty-four disc-shaped specimens (15 mm x 1 mm) of enhanced RMGI (ACTIVA™ BioACTIVE-RESTORATIVE, Pulpdent, Watertown, MA, USA) and 54 disc-shaped specimens (15 mm x 1 mm) of RMGIC (Fuji II LC Capsules, GC Corp, Tokyo, Japan) were prepared. Both materials were divided into 9 groups based on artificial saliva pH (pH 7, pH 5.5, and pH 4.5) and immersion time (1, 7, and 14 days). Water absorption and solubility were measured based on ISO 4049: 2009. Data were analyzed statistically using one-way ANOVA. **Results:** The results showed significant differences in water absorption and solubility value between RMGIC and enhanced RMGI for all the groups. Both materials showed increased water absorption and solubility when immersed in artificial saliva that exhibited a lower pH level and under higher immersion period. **Conclusion:** Lower pH levels and longer immersion time influence the water absorption and solubility of enhanced RMGI and RMGIC.

Key words: glass ionomer, pH, solubility, water absorption

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INTRODUCTION

The glass ionomer cement (GIC) was introduced in the 1970s. This material can bind to the tooth structure, can release fluorine, and exhibits good biocompatibility.¹ However, disadvantages of conventional GIC include poor physical and mechanical traits, easy reaction ability with water in the initial hardening period, short processing time, and long hardening time.^{2,3} To overcome these, a new type of GIC material has been developed, which is the resin-modified glass ionomer cement (RMGIC). This RMGIC has an additional resin component that enables a longer working time and faster hardening time owing to the use of a light-curing unit. Moreover, this material retains the advantage of GIC in terms of fluorine ions released and close bonding with tooth surface.^{1,2}

The RMGIC has undergone refinement, and one of the revised versions is the ACTIVA™ BioACTIVE-

RESTORATIVE (Pulpdent Corp., Watertown, MA, USA) restorative material, called the enhanced RMGIC (ERMGI). The modification in this material is characterized by the presence of a bioactive resin matrix, a shock-absorbing rubberized resin component, and a reactive ionomer glass filler that can mimic the physical and chemical properties of teeth.^{4,5} Previous studies have stated that ERMGI has better compressive and tensile strength than other RMGIC materials.^{4,6} Moreover, the bioactive resin matrix of ERMGI is believed to increase the release of fluorine ions, calcium ions, and phosphate ions that play an important role in teeth remineralizing.⁵

Besides physical/mechanical properties, the ideal restorative material needs to be resistant to several conditions in the oral cavity, such as water absorption and solubility. The RMGIC contains a matrix of polymer resins that can absorb liquids.^{2,4} Fluid absorption can

lead to hygroscopic expansion and plasticization effects that damage the polymer composition of the material and alter the physical/mechanical properties of the material, resulting in reduced hardness and wear resistance. This ability of water absorption is followed by the ability to dissolve components of unreacted material, such as nonpolymerized monomers. The solubility of this material component can affect wear resistance and can cause discoloration.^{1,7,8}

Each day, the salivary glands produce about 1–1.5 L saliva. Under normal circumstances, the pH of the saliva is approximately.⁷ However, during the consumption of certain foods or drinks, the pH of saliva in the mouth can lower and cause hydroxyapatite demineralization, resulting in the teeth caries. When the saliva reaches a value of pH 5.5, hydroxyapatite will be subjected to demineralization. Thus, a pH value of 5.5 is termed the critical pH of hydroxyapatite. When the salivary pH rises and normalizes, the reverse process of remineralization occurs. If during remineralization, a fluoride ion is present, fluoroapatite that has a critical pH of 4.5 will be formed.⁹

In a study that examined the water absorption rate and solubility of GIC, compomer, and RMGIC soaked in lemon juice, the rate of water absorption and solubility of the three materials increased to exceed the ISO 4049:2009 limit (water absorption $\leq 40 \mu\text{g}/\text{mm}^3$ and solubility $\leq 7.5 \mu\text{g}/\text{mm}^3$).¹⁰ Another study showed that the possibility of material degradation could increase the solubility of the RMGIC. Furthermore, the solubility depends on the composition of the monomers in the tested RMGIC material.¹¹ Previous studies reported that water absorption and solubility values of zinc phosphate cement, conventional GIC, RMGIC, and composite resins increased after these materials were immersed in artificial saliva with a pH value of 5. Thus, the salivary pH clearly influences the water absorption capacity and solubility of the RMGIC.¹¹⁻¹³ Generally, ERMGIC (ACTIVA™ BioACTIVE-RESTORATIVE) has lower water absorption and solubility after being immersed in water.^{4,14}

The influence of immersion period and liquid pH on water absorption capacity and solubility of the RMGIC raises the question of whether the critical pH of hydroxyapatite and fluoroapatite influences the water absorption capacity and solubility of ERMGI. This present study was designed to investigate the effect of immersion period and pH of artificial saliva on the water absorption capacity and solubility of ERMGI and RMGIC.

METHODS

A total of fifty-four RMGIC specimens (Fuji II LC Capsule, GC Corp, Japan) and 54 ERMGI specimens

(ACTIVA™ BioACTIVE-RESTORATIVE, Pulpdent Corp, Watertown, MA, USA) were made with plates of 15 mm diameter and 1 mm thickness, as per the ISO 4049:2009 guidelines.¹⁵ The material was superimposed on an acrylic mold using light-curing unit (MAX HILUX LED 700 mW/cm²) for 20 seconds for four times in four different areas. Thereafter, nine groups were created for each material. First, three groups were created as per the artificial saliva pH (pH 7, pH 5.5, and pH 4.5) and each of these groups was then subdivided into three groups as per the immersion time (1, 7, and 14 days), resulting in a total of nine groups (n = 6 in groups).

The prepared specimens were stored in a desiccator that contained silica gel and were stored in an incubator at a temperature of 37°C \pm 1°C for 22 h. These were then replaced in a desiccator at a temperature of 23°C \pm 1°C for 2 h. After the specimens were removed from the desiccator, the mass of the specimen was measured with analytical scales (Shimadzu AX-200, Japan) to determine the initial mass (m1). The specimen diameter was calculated using a digital caliper at two opposite points, and its thickness was measured at the center of the specimen. The cycle was conducted thrice for each specimen, and the initial mass value (m1) of each cycle is summed and averaged.

After the initial mass (m1) was calculated, the specimens were placed into a plastic pot containing 10 mL artificial saliva (pH 7, pH 5.5, and pH 4.5) and stored in an incubator at a temperature of 37°C \pm 1°C for the respective immersion period applicable to each group (1, 7, and 14 days). Thereafter, the specimen was removed and dried with drying paper, and the mass (m2) was measured. The same desiccation cycle was repeated, and the final mass (m3) was obtained. The water absorption and solubility values of each specimen were then calculated following the water absorption and solubility values equation.¹⁰

Water Absorption Value Formula:

$$W_{sp} = \frac{m^2 - m^3}{v}$$

Solubility Value Formula:

$$W_{si} = \frac{m^1 - m^3}{v}$$

Legend:

W_{sp} : Water Absorption Value ($\mu\text{g}/\text{mm}^3$)

W_{si} : Solubility value ($\mu\text{g}/\text{mm}^3$)

m¹ : Initial mass of specimen after insertion into desiccator (μg)

m² : Specimen mass after immersion (μg)

m³ : Final mass specimen after soaking and put in a desiccator (μg)

V : Volume (mm^3)

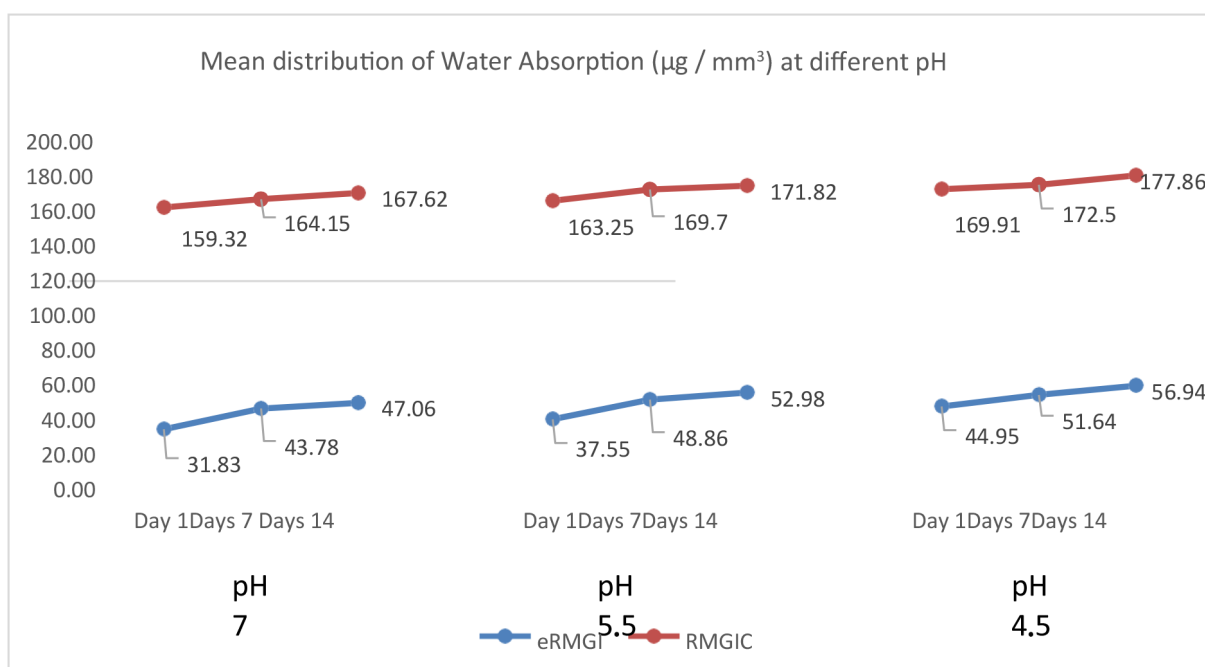


Figure 1. Graph of Distribution of Average Water Absorption at pH 7, pH 5.5, and pH 4.5 for Immersion Time of 1 day, 7 days, and 14 days.

Table 1. Distribution of the mean water absorption of the specimen at different pH and immersion period

Specimen Group and Immersion period (day)	Water Absorption Value (µg/mm ³)			P	
	Mean ERMGI –RMGIC	95% Confidence Interval			
pH 7	1	127.495	115.736	139.253	0.000 *
	7	120.372	113.910	126.833	0.000 *
	14	120.560	112.284	128.836	0.000 *
pH 5.5	1	127.495	106.303	145.090	0.000 *
	7	120.37	105.910	135.763	0.000 *
	14	124.963	112.797	137.130	0.000 *
pH 4.5	1	124.963	112.797	137.129	0.000 *
	7	120.868	113.426	128.310	0.000 *
	14	121.508	118.611	124.405	0.000 *

Significantly different at p <0.001

All the data were analyzed using the SPSS 23.0 application. The normality test was conducted using the Kolmogorov–Smirnov test and the homogeneity test was done using the Levene static test. Thereafter, one-way analysis of variance, followed by the Tamhane post-hoc test and the independent t-test were conducted for the normal data. Kruskal–Wallis test and Mann–Whitney test were used for the non-normal data to compare the water absorption capacity and solubility of the two materials under the same conditions.

RESULTS

In this section, we present the calculated values for water absorption and solubility values for the RMGIC and ERMGI restorative materials.

Figure 1 shows an increasing tendency of the water absorption values along with the immersion time with pH 7, pH 5.5, and pH 4.5. The distribution of the average difference is listed in Table 1.

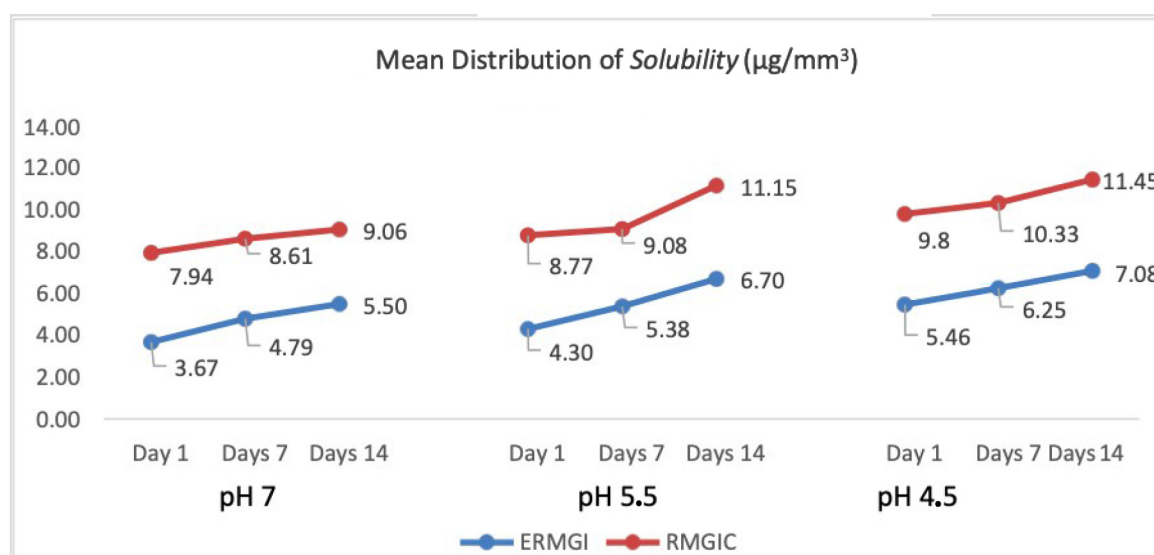


Figure 2. Graphical distribution of the mean solubility at pH 7, pH 5.5, and 4.5 for immersion Period of 1, 7, and 14 days.

Table 2. Distribution of the mean solubility of the specimens at different pH and immersion period

Specimen Group and Immersion period (day)	Water Solubility Value (µg/mm³)			p	
	Mean	ERMGI	RMGIC		95% Confidence Interval
pH 7	1	3.928	2.822	5.034	0.000 *
	7	3.615	2.810	4.420	
	14	3.570	2.944	4.196	
pH 5.5	1	4.425	3.910	4.940	0.000 *
	7	4.140	3.143	5.137	
	14	4.308	3.206	4.961	
pH 4.5	1	4.010	3.519	4.501	0.000 *
	7	4.083	3.521	5.096	
	14	4.248	3.602	4.895	

* Significantly different p <0.001

As shown in Table 1, there was an increase in the water absorption value from the ERMGI and RMGIC restorative materials after immersion in artificial saliva with pH 4.5 for 14 days. Differences in the mean values between restorative materials were significant, with p values <0.001.

As seen in Figure 2, there was a tendency of the solubility value to progressively increase with increased immersion period at pH 7, pH 5.5, and pH 4.5. The distribution of the average differences is presented in Table 2.

DISCUSSION

In this study, the water absorption value of ERMGIC (Activa Bioactive Restorative) was significantly lower than that of RMGIC (Fuji II LC) under all the tested

conditions of pH and immersion time. We found that all the RMGIC water absorption values after immersion in artificial saliva under various pH conditions and immersion periods were much higher than the water absorption value limit set by ISO 4049:2009 (max. 40 µg/mm³, i.e., 159.32–171.82 µg/mm³). Meanwhile, with respect to the water absorption value of the ERMGI after immersion in artificial saliva under various conditions, some values exceeded the ISO 4049:2009 limit (max. 40µg/mm³), whereas others were within the limit (31.83–56.94 µg/mm³). The water absorption values that were below the ISO 4049:2009 limit were from the specimens that were immersed for 1 day in artificial saliva with pH 7 (31.83 ± 9.27µg/mm³) and those immersed for 1 day in artificial saliva with pH 5.5 (37.55 ± 6.3µg/mm³). The significant difference in the water absorption value of the two materials was due to the composition of each material.¹⁶

In this study, the two materials had different types of resins: the RMGIC has hydroxyethyl methacrylate (HEMA) monomer, whereas ERMGI has diurethane dimethacrylate monomer, also known as urethane dimethacrylate (UDMA).^{1,4} Hydroxyethyl methacrylate (HEMA) in RMGIC is very hydrophilic with a hydroxyl group (–OH) in its compound arrangement.¹³ Hydrogen ions from the water enter through the gap between the arrangement of the polymer chains and bind with the hydroxyl group (–OH) on the HEMA monomer, forming hydrogen bonding. Meanwhile, UDMA in ERMGI has a urethane group (–NH–) that can also bind to the hydrogen ions in the water. However, its hydrophilicity is lower compared with that of HEMA.¹⁶ With more liquid absorbed in the immersion, the mass of the material increases.

In this present study, the water absorption value of both materials was higher after they were immersed in artificial saliva that had a lower (more acidic) pH. This finding is in agreement with previous studies that have shown greater increase in the water absorption value in RMGIC after immersion in artificial saliva with pH 3 compared to that after immersion in artificial saliva with pH 7.4.^{11,13} This may be due to the degradation of hydrolysis, that is, hydrolysis degradation in HEMA caused by the hydrogen ions from artificial saliva.^{11,13,17} Hydrolysis degradation in RMGIC material can cause microcrack formation, facilitating the entry of the liquid into the polymer matrix, resulting in increased water absorption.¹³

Degradation also occurs in the enhanced resin-modified glass ionomers. After stirring and polymerization of the monomer, a salt bridge is formed due to the reaction of fluoro-alumino-silicate glass and a polycarboxylic acid. Hydrogen ions release the bonds between the metal cations and the carboxyl groups that already exist in salt bridges. The cations that are released from the salt bridge diffuse out. If this phenomenon continues, more cations are released from the glass component, damaging the glass component. Hydrogen ions can also damage the Si–O–Si bond in the glass component, resulting in more microvoids. The present results indicate that the water absorption values of RMGIC and ERMGI restorative materials are higher with a longer immersion time, caused by more liquid binding with the monomer structure. These results are in agreement with those reported by Lima et al (2018) that showed increased water absorption with longer immersion time (1, 7, 14, and 40 days).¹¹

The solubility value calculated from the mass loss in the specimen after immersion and division by the volume of the specimen in RMGIC was significantly higher than that of ERMGI. The solubility value of RMGIC restorative material in this study exceeds the maximum limit of $\leq 40\mu\text{g}/\text{mm}^3$ set by ISO 4049:2009. The solubility value of ERMGI under various pH conditions and immersion times met the requirements

of ISO 4049:2009. Liquids that diffuse into the polymer matrix can dissolve monomer components that are not polymerized in the material. This nonpolymerized monomer is usually located between the polymer chains and inside the microvoid.¹⁸ Moreover, one of the factors that can affect the solubility of restorative material is the degree of conversion of the material. A higher degree of conversion is associated with a lower solubility value of the material.¹⁸ Münchow et al (2014) showed that UDMA monomers have a higher degree of conversion than HEMA.¹⁹ This could explain the higher solubility of the RMGIC that involves a HEMA monomer whose degree of conversion is lower than that of an ERMGI that has a UDMA monomer.

Our results are also in accordance with results in higher RMGIC solubility values when immersed in artificial saliva with lower pH.^{10,16} This may be because the degradation or hydrolysis that occurs in RMGIC and ERMGIC increases with a decreasing pH. Acidic pH conditions can increase the plasticization effect observed in resin components to reduce the bonding between polymer chains in the dimethacrylate matrix. This weakened bond between the polymer chains can cause more resin components, such as monomers, to become nonpolymerized and detached, thereby increasing the solubility of the restorative material.¹⁶

CONCLUSION

Our findings show that the water absorption and solubility values of ERMGIC materials were higher after being immersed in artificial saliva with lower pH and longer immersion period. Thus, the ERMGIC materials have significantly lower water absorption and solubility values than the RMGIC materials at various immersion times and pH values of artificial saliva.

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CONFLICT OF INTEREST

The authors report no conflict of interest.

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