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# WATER SORPTION OF DIFFERENT BIOACTIVE RESTORATIVE MATERIALS AT DIFFERENT TIME INTERVALS; AN IN VITRO COMPARATIVE STUDY

Hisham O. Mohamed<sup>1\*</sup>, Sameh M. Nabih<sup>2</sup>, Walaa M. Alsamolly<sup>3</sup>

## ABSTRACT

**Objective:** This study was performed to assess and compare water sorption of three different bioactive restorative materials; Resin Modified Glass Ionomer (RMGI), giomer, and Activa bioactive composite at different time intervals in vitro. **Materials and methods:** 45-disc shaped specimens were prepared and divided into three equal main groups (n=15) according to the type of restorative material; RMGI (Fuji II LC), giomer (Beautifil II), and Enhanced RMGI (Activa bioactive composite), then immersed in deionized water. Each main group was divided into three equal subgroups (n=5) according to the storage period (1 day, 3 months and 6 months), specimens were subjected to drying cycles and weighed for water sorption measurement. Data analysis was performed using Two-way ANOVA test, post-hoc test, Kruskal-Wallis test and Dunn's test. **Results:** After 24 hours; none of the three groups showed water sorption. After three and six months; there was a statistically significant difference between water sorption values in the three groups. Water sorption was statistically higher in Fuji II followed by Activa while Beautifil II showed the least one. **Conclusions:** RMGI, giomer, and Activa revealed water sorption values of accepted range in accordance with the ISO specification after 1 day, 3 months and 6 months storage periods.

KEYWORDS: Resin Modified Glass Ionomer; Giomer; Activa; Water sorption.

### **INTRODUCTION**

Nowadays, the resinous materials technology is one of the most important contributions to the dentistry due to the increased aesthetic demands. The physico-chemical and mechanical properties of these materials such as water sorption and solubility are influenced by the filler contents and the monomers structure and subsequently affect the durability of the restorations<sup>(1,2)</sup>. Generally, in a moist oral environment the polymers of resinous materials absorb water. The interaction between these materials and the oral cavity may cause two phenomena; the first phenomenon is water sorption which leads to an increase in weight of the material. On the other hand, the second phenomenon is dissolution of the material. This hydrolysis contributes to the reduction of the final weight of the material which is also known as Biodegradation<sup>(3,4)</sup>.

Innovations in the field of direct restorative materials' manufacturing are a continuous non stopping process. These advancements aim at combining the merits of good esthetics and mechanical properties

3. Assistant Professor of Operative Dentistry Department, Faculty of Dental Medicine, Boys, Cairo Al-Azhar University.

• Corresponding author: senna53hish@gmail.com

<sup>1.</sup> Ph.D. Founder in Operative Dentistry Department, Faculty of Dental Medicine, Boys, Cairo, Al-Azhar University.

<sup>2.</sup> Professor of Operative Dentistry Department, Faculty of Dental Medicine, Boys, Cairo, Al-Azhar University.

of resin composite and the fluoride-releasing capability of conventional glass ionomer restorative materials <sup>(5,6)</sup>. This open-ended effort has led to the emerging of bioactive restorative materials, such as Resin-Modified Glass Ionomer, giomers and Bioactive Composite <sup>(7)</sup>.

RMGIC (Fuji II) are hybrid materials that retain a significant acid-base reaction as part of their overall curing process <sup>(8)</sup>. They became clinically favorable due to their improved physical and mechanical properties compared to glass ionomer restorative materials in addition to their ability to act as a reservoir for direct fluoride release to susceptible tooth surfaces in high caries risk <sup>(9)</sup>.

Giomer (Beautifil II) is an anhydrous resinous restorative material which is known as "Pre-reacted glass-ionomer (PRG) composites". Giomer differs from compomer as it contains pre-reacted glass ionomer fillers (Flouro-alumino-silicate glass particles) incorporated into silica filled urethane resin matrix. The PRG fillers are fabricated by acid–base reactions between fluoride containing glass and polyacrylic acid in the presence of water. Giomers still have the need to absorb water to maintain fluoride release and recharge the properties of glass-ionomer cements <sup>(10,11,12)</sup>.

Activa Bioactive Restorative was regarded as an equivalent to resin reinforced glass ionomer containing glass particles and polyacid constituents of glass ionomer, which undergoes acid-base setting reaction <sup>(13)</sup>. They are also composed of a resin matrix, having both light and chemical polymerization ability. Such resin matrix is a patented bioactive shock absorbing rubberized ionic-resin (Embrace resin) matrix that includes a small percentage of water with no Bisphenol A (BPA), Bisphenol A-glycidyl methacrylate (Bis-GMA) or BPA derivatives <sup>(14)</sup>.

The aim of this study was to asses and compare water sorption of different bioactive restorative materials; RMGI (Fuji II LC), giomer (Beautifil II), and Enhanced RMGI (Activa bioactive composite) at different time intervals (1 day, 3 months and 6 months) in-vitro.

The null hypothesis was that there will be no difference in water sorption between the tested restorative materials.

### MATERIAL AND METHODS

#### I- Materials:

Three different bioactive restorative materials were used in this study as listed in (**Table 1**).

**TABLE** (1) Brand name, material specification, composition, manufacture and batch number of the material used:

Brand name and material specification	Composition	Manufacture and (batch No.)		
Fuji II LC (RMGIC)	HEMA, Polyacrylic acid and water. 58 wt% Fluoro-aluminumsilicate	GC, Tokyo, Japan (2103252)		
Beautifil II (Giomer)	Bis-GMA, UDMA, Bis-MPEPP, TEGDMA. 83.3 wt% Fluoro- silicate glass.	Shofu.Kyoto, Japan (041824)		
ACTIVA (Enhanced RMGIC)	Blend of diuerthane and other methacrylates with modified polyacrylic acid. 55.4 wt% bioactive glass and sodium fluoride.	Pulpdent. Watertown, MA. USA (180419)		

HEMA: hydroxyethyl methacrylate. Bis-GMA: bisphenol-A-diglycidyl-methacrylate. UDMA: urethane dimethacrylate. Bis-MPEPP: Bisphenol A polyethoxy Methacrylate. TEGDMA: triethyleneglycol dimethacrylate. RMGIC: resin modified glass-ionomer cement.

#### **II- Method:**

## II.1. Sample size:

Based on previous research <sup>(15)</sup>, a sample size of 30 in each group had an 80% power to detect a difference between means of 18.84 with a significance level (alpha) of 0.05 (two-tailed) at 95% confidence interval. In 80% (the power) of those experiments, the p value will be less than 0.05 (two-tailed) so the results will be deemed "statistically significant". In the remaining 20% of the experiments, the difference between means will be deemed "not statistically significant". Report created by Graphpad StatMate 2.00.

## II.2. Sample grouping:

A total number of 45 specimens were fabricated using a specially constructed standardized split Teflon molds. The specimens were divided into three equal main groups (n=15) according to the type of restorative material (Fuji II, Beautifil II and ACTIVA). Each main group was divided into three equal subgroups (n=5) according to the storage periods (1 day, 3 months and 6 months).

#### **II.3. Specimens Preparation:**

The specially constructed split Teflon mold (2mm thickness and 10mm diameter for fluoride release) was placed on the top of a sterile microscope glass slide and a celluloid strip, the tested materials were packed according to manufacture instruction into the mold using sterile gold plated instrument (Miltex, stainless Italy, 70-204 EELT 4). The second celluloid strip was used to cover the top side of the mold in order to prevent formation of oxygen inhibited layer (16). Another glass slide and 500gm load were applied over the second Mylar strip for 30s to guarantee a reliable packing of the prepared specimens and to extrude the excess material<sup>(17)</sup>, (Figure 1). The applied load and microscope slide were removed from the top surfaces before curing, Polymerization was performed using LED light-curing device (Elipar S10, 3M ESPE; USA,

wavelength 455 nm +/- 10 nm, light intensity 1200 mW/cm<sup>2</sup>). Light curing was done at one side according to materials manufacturer's instructions; 20 second for each material.



FIG (1) Applied load weight.

The guiding tip of the light curing unit was held centered in direct contact with the celluloid strips on the top surface of the mold perpendicular to it to standardize the curing distance. After photopolymerization, the cylindrical shaped specimens were removed from their molds and rinsed continuously with tap water for 1min and then rechecked for their diameter and thickness using a digital caliper, all specimens were then polished with sof-Lex polishing system (Sof-Lex polishing system, 3M ESPE, St. Paul, MN, USA) to remove the resin rich surface layer <sup>(18)</sup>.

### II.4. Storage of the specimens:

Each Sample was immersed in a plastic box filled with 5 mL of deionized water at 37°C (triple distilled water Anion H +Cation OH = $H_2O$  which is free of minerals) The deionized water was made especially for the experiment by Cairo University's faculty of pharmacy, pharmaceutical department. The deionized water was replaced daily for further equipoising. Water Sorption test was performed in compliance with the ISO 4049:2009 standards <sup>(17)</sup>. After the first 24h samples were removed, placed on a filter paper for one min, and weighed to an accuracy

of  $\pm$  0.1 mg with a digital electronic sensitive balance (SIGMA-ALDRICH, Germany) (Figure 2). The weight change should not exceed 0.2 mg and a constant mass  $(M_0)$  was gained. Any samples with a value less than 10 percent of the average weight were discarded due to the possible presence of internal voids. The diameter and thickness of each sample was measured three times at the same time with a caliper to calculate the sample volume (V) in mm<sup>3</sup> and the average was taken. After the second 24h the samples were removed from the box, excess water was removed with absorbent paper, air-dried for 15 s, and reweighed to obtain  $(M_1)$ , then the diameter and thickness was measured with a caliper to obtain  $(V_1)$ . Using the same protocol as for  $(M_1)$ , samples were restored again in deionized water for 3 months to record  $(M_2)$  and for 6 months to record  $(M_2)^{(19)}$ .



FIG (2) Digital electronic sensitive balance.

The volume (V) of samples was calculated by taking their diameters at two opposing points on the sample circumference with electronic digital caliper (CD6CS, Mitutoyo, Kanagawa, Japan), and their thickness at the center and at four equally spaced points <sup>(20)</sup>, (**Figure 3**). Water Sorption was calculated as follows <sup>(17)</sup>:

- Sorption after 1 day (SW1) =  $(M_1 M_0/V)$
- Sorption after 3 months (SW2) =  $(M_2 M_0/V)$
- Sorption after 6 months (SW3) =  $(M_3 M_0/V)$ .



FIG (3) Electronic digital caliper calculates sample diameters.

#### Statistical analysis

Data were presented as mean, standard deviation (SD), median and range values. Two-way ANOVA test was used to study the effect of material type, time and their interactions. Bonferroni's post-hoc test was used for pair-wise comparisons when ANOVA test is significant. For non-parametric data; Kruskal-Wallis test was used to compare between water sorption of the three materials as well as the three follow up times. The significance level was set at  $P \le 0.05$ .

#### RESULTS

#### Effect of the Restorative Materials

After 24 hours; none of the specimens in the three groups showed water sorption.

After three months; there was no statistically significant difference between median water sorption values in the three groups (P-value = 0.364, Effect size = 0.002).

After six months; there was a statistically significant difference between median water sorption values in the three groups (*P*-value = 0.026, Effect size = 0.443). Pair-wise comparisons between materials revealed that Fuji II group showed the statistically significantly highest median water sorption. Activa group showed statistically significantly lower median value. Beautifil II group showed the statistically significantly lowest median water sorption (**Table 2**).

Time	Fuji II		Activa		Beautifil II			Effect size
	Mean (SD)	Median (Range)	Mean (SD)	Median (Range)	Mean (SD)	Median (Range)	P-value	(Eta squared)
24 hours	0 (0)	0 (0-0)	0 (0)	0 (0-0)	0 (0)	0 (0-0)	1	0
3 months	0.000034 (0.000025)	0.000042 (0-0.00006)	0.0002 (0.000019)	0.000028 (0-0.00004)	0.00002 (0.000017)	0.000012 (0-0.00004)	0.364	0.002
6 months	0.0001 (0.00002)	0.00011 <sup>A</sup> (0.00007-0.00012)	0.000035 (0.000023)	0.000042 <sup>в</sup> (0-0.00006)	0.000035 (0.000048)	0.000012 <sup>c</sup> (0-0.00011)	0.026*	0.443

**TABLE (2)** Descriptive statistics and results of Kruskal-Wallis test for comparison between water sorption of the three materials:

\*: Significant at  $P \le 0.05$ , Different superscripts in the same row indicate statistically significant difference between materials

### • Effect of storage period

As regard to Fuji II; there was a statistically significant difference between median water sorption values at different times (P-value = 0.002, Effect size = 0.844). Pair-wise comparisons between time periods revealed that 24 hours' period showed no water sorption. Three months' period showed statistically significantly higher median water sorption. Six months' period showed the statistically significantly highest median water sorption.

significant difference between median water sorption values at different times (*P*-value = 0.039, Effect size = 0.373). Pair-wise comparisons between time periods revealed that 24 hours' period showed no water sorption. There was no statistically significant difference between three and six months' periods; both showed statistically significantly higher median water sorption values than 24 hours' period.

While for Beautifil II; there was no statistically significant difference between median water sorption values at different times (*P*-value = 0.067, Effect size = 0.285) (**Table 3**).

As regard to Activa; there was a statistically

	Fuji II		Activa		Beautifil II	
Time	Mean (SD)	Median (Range)	Mean (SD)	Median (Range)	Mean (SD)	Median (Range)
24 hours	0 (0)	0 (0-0) <sup>c</sup>	0 (0)	0 (0-0) в	0 (0)	0 (0-0)
3 months	0.000034 (0.000025)	0.000042 <sup>в</sup> (0-0.00006)	0.0002 (0.000019)	0.000028 <sup>A</sup> (0-0.00004)	0.00002 (0.000017)	0.000012 (0-0.00004)
6 months	0.0001 (0.00002)	0.00011 <sup>A</sup> (0.00007-0.00012)	0.000035 (0.000023)	0.000042 <sup>A</sup> (0-0.00006)	0.000035 (0.000048)	0.000012 (0-0.00011)
P-value	0.002*		0.039*		0.067	
Effect size (Eta squared)	0.844		0.373		0.285	

**TABLE (3)** Descriptive statistics and results of Kruskal-Wallis test for comparison between water sorption at different times within each material:

\*: Significant at  $P \le 0.05$ , Different superscripts in the same column indicate statistically significant difference between materials.

## DISCUSSION

Water sorption is an important test to characterize resin-based materials. Sorption and solubility affect the materials' biocompatibility, structural integrity, mechanical properties, dimensional and color stability <sup>(21)</sup>.

There are two methods for calculating water sorption identified in previous studies. Some studies calculate water sorption by subtracting the conditioned-initial dry mass prior to immersion from the saturated mass of specimen after immersion. However, this method does not take into consideration the amount of leached out materials. Therefore, to be more precise, in this study, the water sorption was calculated by subtracting the reconditioned-final dry mass from the saturated mass of specimen after immersion<sup>(22,23)</sup>. Also, the water sorption of each specimen was calculated in accordance with ISO4049:2009<sup>(17)</sup> which is considered acceptable when water sorption was  $\leq 40 \mu g/mm3$ .

Our findings revealed that after six months Fuji II group showed significant highest water sorption followed by Activa group, while Beautifil II group showed significant lowest median water sorption. These results agreed with Sufyan et al <sup>(24)</sup> who found that the water sorption after 37 days of Fuji II was the highest followed by Activa, while Beautifil II showed the least water sorption. They explained that the presence of hydrophilic constituent HEMA in GC Fuji II can increase its water sorption ability. Moreover, the method of mixing may generate air voids, which may accelerate the water sorption of this material and increases the surface exposed to moisture, this may lead to inhibition zones with unpolymerized material <sup>(24, 25)</sup>.

Moreover, the Fuji II is a hydrophilic material which is sensitive to moisture and water absorption is necessary for the acid-base setting reaction. Water sorption values of Activa might be due to the bioactive ionic resin matrix which shows a degree of hydrophilicity as claimed by the manufacturer <sup>(7)</sup>. However, Beautifil II contains surface pre reacted glass ionomer (S-PRG) as a fluoride component. The fluoride glass within Beautifil II has little or no glass ionomer matrix phase, because of the lack of any significant acid base reaction. As S-PRG has been reacted with fluoroaluminosilicate glass and acid, water sorption is not critical in the acid base reaction <sup>(26)</sup>.

This finding also in agreement with Eriwati et al <sup>(27)</sup>, who stated that the water absorption value of Activa was significantly lower than that of RMGIC (Fuji II LC) may be due to the composition of each material. As the Fuji II has HEMA monomer, whereas Activa has UDMA.

Furthermore, the HEMA in Fuji II is very hydrophilic and higher water absorption is observed inmaterials with higher HEMA. The HEMA can cause water absorption up to about 80% of its weight<sup>(28)</sup>. Meanwhile, UDMA in Activa 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. With more liquid absorbed in the immersion, the mass of the material increases <sup>(25)</sup>. Moreover, the lower filler content of RMGI than Activa indicates a higher resin content in RMGI so RMGI can absorb more water than Activa <sup>(29)</sup>.

Moreover, our findings were in agreement with Kandil et al <sup>(7)</sup>, who concluded that water sorption values of Activa was higher than that of Beautifil II, Activa is considered a hydrophilic enhanced RMGIC supplemented with bioglass and strengthened with a patented rubberized polymer resin. However, the water sorption values of Activa complies the ISO 4049:2009 requirements in which water sorption of resin-based materials should not exceed 40µg/mm3.

Besides that, our findings were with Gorka et al<sup>(30)</sup> who found that the water absorption of Giomer was comparatively less than that of RMGIC. They explained that the Giomer fillers were pretreated

with polyalkenoic acids in presence of water before being interfaced with organic matrix. Therefore, no absorption of moisture is required in the matrix. Thus, glass ionomer phase in Giomers is not affected by water uptake in the restoration, whereas it was

significantly affected by water uptake in RMGIC.

In contrast to resin modified glass ionomer, the acid-base reaction in Giomer occurred in S-PRG fillers during manufacturing, thus resulting in a surface modified layer which is described as consequently protecting the glass core from the damaging effects of moisture. Moreover, the stability of the Giomer might also be attributed, at least partially, to the incorporation of different types of filler in the material amounting in total to 83.3 wt%, including, besides S-PRG fillers, also large pre-polymerized fillers (31). While the RMGIC has filler size range of 1.8 µm, with filler loading 76 wt.% by volume. Therefore, it can be expected that Giomer having a lower resin content compared to RMGIC may lead to increase water sorption in RMGIC (32). Based on our results, the null hypothesis was rejected as Giomers and Bioactive Resin Restorative revealed less water sorption than Resin Modified Glass Ionomer.

In our study the tested materials showed significantly different water sorption behavior by time, all the tested materials showed acceptable water sorption behavior even after 6 months, which is below 40mg/mm3 (the maximum water sorption stated by the ISO 4049). On the other hand, our findings were in contrast with Harhash et al <sup>(17)</sup> who found that Giomer showed significantly greater water absorption than Fluoride-releasing composite due to the type of the resin matrix which is a main factor in water sorption of resinous restorations, which may control both water diffusion rate and the amount of sorption in the matrix.

A possible limitation of the present study is that the dynamic nature of conditions found actually in the oral cavity such as salivary flow characteristics, presence of plaque, oral hygiene and dietary habits utilized by the patient which can lead to results that may be different from what have been found in the current study. Thus, further studies employing clinical trials are important <sup>(33)</sup>.

## CONCLUSIONS

- 1. All materials used in the study revealed water sorption values of the accepted range in accordance with the ISO specification.
- 2. Water Sorption is material and time dependent.

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