

Comparative Study of the Solubility and Sorption Properties of Resin Modified Glass Ionomer and a Bioactive Liner

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Abstract

Background and Aim: This study aimed to compare the sorption and solubility properties of ACTIVA BioACTIVE liner and resin modified glass ionomer (RMGI).

Materials and Methods: In this in vitro study, a total of 30 samples were fabricated from each liner measuring 15 mm in diameter and 1 mm in thickness according to ISO 2009:4049. They were then divided into 6 subgroups (n=5) to assess their water solubility, water sorption, and acid solubility after 1 week and 8 weeks. The samples were then weighed, and placed in vials containing artificial saliva or lactic acid and incubated at 37°C for 1 week or 8 weeks according to specimen grouping. Afterwards, each specimen was weighed again. Sorption and solubility were calculated according to difference in weight of the samples. Data were analyzed by two-way ANOVA ($\alpha=0.05$).

Results: RMGI showed greater water solubility and sorption compared with ACTIVA BioACTIVE liner after 1 week of storage ($P=0.00$). No significant difference was found in water solubility ($P=0.64$) and sorption ($P=0.15$) after 8 weeks of storage. There was no significant difference in acid solubility of RMGI and ACTIVA BioACTIVE liner after 1 week ($P=0.30$) or 8 weeks of storage ($P=0.60$).

Conclusion: Water sorption and solubility of RMGI were greater than those of ACTIVA BioACTIVE within the first week after setting but they were similar in long-term assessment. RMGI and ACTIVA BioACTIVE were not different in terms of acid resistance.

Key Words: Adsorption; Pulp Capping; Pulpectomy Agents; Solubility

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Introduction

Protecting and preserving the pulp vitality are of great importance in modern restorative dentistry [1]. Vital pulp therapy can increase the longevity of teeth because the dental pulp is responsible for protecting and nourishing

the tooth [2]. Successful pulp capping preserves the vitality of the tooth, and induces dentinal bridge formation. On the other hand, it is more cost effective than root canal therapy [3,4].

Calcium hydroxide [$\text{Ca}(\text{OH})_2$] has been the

gold standard of pulp capping for decades because of its ability to assist dentin formation and superb antibacterial properties [5]. However, $\text{Ca}(\text{OH})_2$ shows poor mechanical properties, has no inherent adhesion to dentin, and is highly soluble. The high solubility of $\text{Ca}(\text{OH})_2$ may lead to dissolution of this liner and insufficient sealing ability [6]. Due to the drawbacks of $\text{Ca}(\text{OH})_2$, materials with more desirable properties were developed and introduced to the market [7]. Resin modified glass ionomers (RMGIs) such as Fuji Lining (GC Corporation, Tokyo, Japan) are among such materials. RMGIs have some noticeable advantages including chemical adhesion to enamel and dentin, fluoride release, which improves antibacterial properties and inhibits further demineralization of the adjacent tooth structure, and optimal biocompatibility. Owing to the desirable properties of RMGIs, they are used as a base material, or alternatively as liner for indirect pulp capping [8,9]. Although RMGIs have high biocompatibility in deep cavities, contemporary dentistry searches for bioactive rather than only biocompatible materials [10]. Today, bioactivity is a topic of paramount interest in restorative dentistry. Bioactive materials interact with cells and cause them to respond favorably. An example of this optimal cellular response is the formation of dentinal bridge in deep cavities [11]. Recently, ACTIVA BioACTIVE Base/Liner (Pulpdent, Watertown, MA, USA) was introduced as a light-curable resin-modified calcium silicate with bioactive glass as filler [12]. ACTIVA BioACTIVE is composed of diurethane and methacrylate-based monomers with a modified polyacrylic acid [13]. The amount of calcium and fluoride release from ACTIVA BioACTIVE is greater than that from glass ionomers, and its adherence to dentin improves its sealing ability. It also promotes dentinal bridge formation close to dental pulp. It does not contain bisphenol A or Bis-GMA [14].

Pulp capping materials are in close contact with dental pulp; thus, they must have special

properties especially low sorption and solubility properties. Water sorption leads to hydrolysis of pulp liners, and changes their properties such as their mechanical and structural strength, and weakens the bond between the liner and cavity walls [15,16]. The absorbed water serves as a plasticizer and causes deterioration of liner. Solubility also leads to gradual degradation of the liner so it is not effective any more. As a consequence, microleakage and pulp damage occur. Insolubility and low sorption in oral fluids are of paramount importance especially in cases that sealing ability of the restoration is compromised due to various reasons. On the other hand, due to proximity of liner to dental pulp, this material is exposed to moisture [17]. Sealing ability of a liner is one of the most important properties for preserving dental pulp vitality. The sealing ability contributes to solubility of dental cavity liners. Several studies investigated the sealing ability and bacterial leakage of cavity liners in dentistry, and concluded that insolubility is directly related to lower bacterial leakage [18,19]. On the other hand, dissolution of some organic contents of dental materials into the saliva and their oral intake may cause local or systemic reactions [20].

To this day, there is inadequate evidence about solubility and sorption properties of ACTIVA BioACTIVE. Therefore, the aim of this study was to investigate the solubility and sorption properties of ACTIVA BioACTIVE in artificial saliva and acidic solution and compare these properties with those of one of the most commonly used liners i.e. Fuji Lining. The null hypothesis was that there would be no difference in terms of solubility and sorption properties between ACTIVA BioACTIVE and Fuji Lining.

Materials and Methods

Specimen preparation:

In this in vitro study, water solubility, acid solubility, and water sorption of Fuji Lining

RMGI (GC Corporation, Tokyo, Japan) and ACTIVA BioACTIVE Base/Liner (Pulpdent Corporation, Watertown, MA, USA) were assessed after 1 and 8 weeks of storage. Table 1 presents the characteristics of the materials used in this study. The sample size was calculated to be 5 in each subgroup based on a study by Francisconi et al. [21] assuming $\alpha=0.05$, $\beta=0.2$ and 80% study power. The Bioethics Committee of Islamic Azad University of Tehran Medical Sciences approved this study.

A total of 60 disc-shaped samples were fabricated using stainless steel molds measuring 15 mm in diameter and 1 mm in thickness according to ISO 2009:4049 [22]. In order to fabricate Fuji Lining samples, the powder and liquid were mixed on a glass slab using a stainless steel spatula according to the manufacturer's instructions, and the mixture was then applied into the molds. ACTIVA BioACTIVE is supplied in a syringe and was directly injected into the molds. For both materials, the molds were placed on a Mylar strip on top of a glass slab [22]. The mold was slightly overfilled to minimize voids. Another Mylar strip was placed on top of the mold and then pressure was applied using a second glass slab over it for 30 seconds to allow the excess material to leak out. The samples were light-cured using a LED light-curing unit (Demetron LC; Kerr, Orange, CA, USA) with 600 mW/cm² intensity for 40 seconds. The light intensity was calibrated with a radiometer (Kerr Corporation, Orange, CA, USA) before each time of use. The samples were removed from the molds and gentle dry polishing was done on both sides with 600-grit silicon carbide paper for 5 seconds. They were then randomly divided into the following groups [22]:

Group A (n=30): ACTIVA BioACTIVE Base/Liner

Subgroup A-WSl 1 (n=5): water solubility of ACTIVA after 1 week

Subgroup A-WSl 8 (n=5): water solubility of ACTIVA after 8 weeks

Subgroup A-WSO1 (n=5): water sorption of ACTIVA after 1 week

Subgroup A-WSO 8 (n=5): water sorption of ACTIVA after 8 weeks

Subgroup A-AS1 (n=5): acid solubility of ACTIVA after 1 week

Subgroup A-AS 8 (n=5): acid solubility of ACTIVA after 8 weeks

Group F (n=30): Fuji Lining RMGI

Subgroup F-WSl 1 (n=5): water solubility of Fuji Lining after 1 week

Subgroup F-WSl 8 (n=5): water solubility of Fuji Lining after 8 weeks

Subgroup F-WSO1 (n=5): water sorption of Fuji Lining after 1 week

Subgroup F-WSO 8 (n=5): water sorption of Fuji Lining after 8 weeks

Subgroup F-AS 1 (n=5): acid solubility of Fuji Lining after 1 week

Subgroup F-AS 8 (n=5): acid solubility of Fuji Lining after 8 weeks

Evaluation of sorption and solubility in artificial saliva:

Evaluation of water sorption and water solubility was done according to ISO 2009:4049 [22]. The samples were transferred into a desiccator containing fresh silica gel and stored at 37°C for 22 hours. They were then transferred to another desiccator and remained at 23°C for 2 hours. The samples were then weighed using a digital scale (Madrin, Japan) with 0.1 mg accuracy. The drying cycle continued until each disc reached a constant initial weight (W1).

Samples in each group were separately placed in 50 mL vials containing artificial saliva, capped, and incubated at 37°C for 1 week or 8 weeks according to specimen grouping discussed earlier. The artificial saliva used consisted of KCl (0.4 g/L), NaCl (0.4 g/L), CaCl₂.2H₂O (0.906 g/L), NaH₂PO₄.2H₂O (0.690 g/L), Na₂S₉H₂O (0.005 g/L), and urea

Table 1. Characteristics of the materials used in this study

Material	Manufacturer	Composition
Fuji Lining resin-modified glass ionomer cement	GC Corporation Tokyo, Japan	Powder: aluminofluorosilicate glass Liquid: polyacrylic acid, HEMA, metadimethacrylate, water Patented bioactive ionic resin
ACTIVA BioACTIVE Base/Liner	Pulpdent Corporation, Watertown, MA USA	Patented rubberized resin bioactive glass ionomer with blend of diurethane and other methacrylates with modified polyacrylic acid amorphous silica sodium fluoride

(1 g/L) with a pH of 6.5 [23].

Afterwards, each specimen was removed from the artificial saliva, rinsed with fresh distilled water, dried with absorbent paper, air-dried for 15 seconds, and weighed again (W2). The samples were then placed in a desiccator containing fresh silica gel to achieve their final weight (W3).

The diameter (d) and height (h) of each sample were measured by taking the means of measurements at the center and at four points with equal distance from the center of each disc using a digital caliper (Mitutoyo, Japan). The volume (V) of each sample was calculated in cubic millimeters using the formula below:

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

Water sorption and water solubility were calculated in micrograms per cubic millimeters ($\mu\text{g}/\text{mm}^3$) using the formula below:

$$\text{Water sorption} = (W2 - W1) / V$$

$$\text{Water solubility} = (W1 - W3) / V$$

Evaluation of solubility in acid:

The samples were transferred into a desiccator containing fresh silica gel and stored at 37°C for 22 hours. They were then transferred to another desiccator and remained at 23°C for 2 hours. The samples were then weighed using a digital scale (Madrin, Japan) with 0.1 mg accuracy. The drying cycle continued until each disc reached a constant initial weight (W1).

To prepare the acidic solution, 8.27 g of lactic acid and 0.92 g of sodium lactate (Merck, Germany) were added to grade 3 water at least solution was adjusted at 3 using a digital

pH-meter (Denver Instrument, USA).

Each sample was separately placed in vials containing 50 mL of acidic solution, capped, and incubated at 37°C for 1 week or 8 weeks according to specimen grouping discussed earlier.

Afterwards, each specimen was removed from the acidic solution, rinsed with fresh distilled water, dried with absorbent paper, air dried for 15 seconds, and weighed again (W2). The samples were then placed in a desiccator containing fresh silica gel to achieve their final weight (W3).

The volume (V) of each sample was calculated in cubic millimeters as described in the previous section.

Solubility in acidic solution was calculated in micrograms per cubic millimeters ($\mu\text{g}/\text{mm}^3$) using the formula below:

$$\text{Acid solubility} = (W1 - W3) / V$$

Statistical analyses were carried out using SPSS version 22 (SPSS Inc., IL, USA). The groups were compared using two-way ANOVA. The significance level was set at 0.05

Results

Water solubility and sorption:

Table 2 presents the mean water solubility and sorption of Fuji Lining and ACTIVA BioACTIVE after 1 and 8 weeks of storage. The effect of time on water solubility and sorption was significant as both materials showed significantly greater water solubility and sorption after 8 weeks of storage compared with 1 week ($P=0.0001$). Also, the effect of type

Table 2. Mean solubility and sorption ($\mu\text{g}/\text{mm}^3$) of the groups

Storage medium	Storage time	Type of material	Mean \pm SD
Solubility in artificial saliva	1 week	Fuji Lining	0.45 \pm 0.10
		ACTIVA	0.11 \pm 0.02
	8 weeks	Fuji Lining	0.74 \pm 0.02
		ACTIVA	0.37 \pm 0.05
Sorption in artificial saliva	1 week	Fuji Lining	0.57 \pm 0.10
		ACTIVA	0.13 \pm 0.02
	8 weeks	Fuji Lining	0.92 \pm 0.01
		ACTIVA	0.39 \pm 0.06
Solubility in acid	1 week	Fuji Lining	0.66 \pm 0.02
		ACTIVA	0.49 \pm 0.05
	8 weeks	Fuji lining	0.83 \pm 0.10
		ACTIVA	0.67 \pm 0.02

of material on water solubility and sorption was significant as Fuji Lining showed greater water solubility and sorption compared with ACTIVA BioACTIVE after 1 week of storage ($P=0.0001$). However, the interaction effect of time and type of material on water solubility and sorption was not significant as no significant difference was found between water solubility ($P=0.64$) and sorption ($P=0.15$) of Fuji Lining and ACTIVA after 8 weeks of storage.

Solubility in acid:

Table 2 presents the mean acid solubility of Fuji Lining and ACTIVA BioACTIVE after 1 and 8 weeks of storage. The effect of time on acid solubility was not significant as there was no significant difference between acid solubility of the two materials after 8 weeks of storage compared with 1 week ($P=0.30$). Also, the effect of type of material on acid solubility was not significant as there was no significant difference between acid solubility of Fuji Lining and ACTIVA BioACTIVE after 1 week of storage ($P=0.30$). The interaction effect of time and type of material on acid solubility was not significant either as no significant difference was found between acid solubility of Fuji Lining and ACTIVA after 8 weeks of storage ($P=0.60$)

Discussion

This study assessed sorption and solubility properties of Fuji Lining and ACTIVA BioACTIV,

and showed that Fuji Lining had significantly higher water solubility and water sorption than ACTIVA BioACTIVE after 1 week of storage; thus, the null hypothesis was rejected.

Pulp capping is intended to preserve tooth vitality. The properties of pulp liners such as water solubility and sorption play an important role in success of this treatment [12].

Water sorption by polymeric materials is explained based on two theories; according to the free volumetric theory, water is absorbed through the voids in the material. According to the interaction theory, water sorption is due to the presence of hydrophilic parts in the polymer [24]. The hydrophilicity of a polymer is determined by its composition, cross links between polymer chains, and the percentage of hydroxyl, carboxyl, or phosphate groups. The more hydrophilic the polymer, the higher its water sorption would be [25]. Thus, higher water sorption and solubility of RMGI can be attributed to higher content of 2-hydroxyethyl methacrylate which is a hydrophilic monomer. The higher the 2-hydroxyethyl methacrylate content of a material, the higher its water sorption would be [26]. On the other hand, a triple setting mechanism occurs in ACTIVA including light-cure resin polymerization, self-cure resin polymerization, and acid-

base reaction, which may explain its superior sorption and solubility resistance [27]. However, the precise composition of its resin matrix has not been disclosed by the manufacturer. In accordance to our results, Zankuli et al. [28] showed significantly higher water sorption of RMGI cement compared with composite resins.

In the present study, water sorption and solubility of ACTIVA BioACTIVE and Fuji Lining were not different after 8 weeks of storage; this may be attributed to the balance in water sorption over time [24]. Similarly, Espir et al. [29] evaluated the solubility of several cavity liners and concluded that after 1 week of storage, zinc oxide/eugenol (ZOE) presented the highest solubility; but after one month of storage, no difference was observed among the studied liners.

In RMGI, water sorption initially displaces aluminum and calcium ions, during which, reaction with polyacrylic acid occurs; but excessive water sorption over time causes the material to degrade which may lead to the loss of desirable properties as a pulp liner [26].

In the present study, water sorption and solubility were measured after 1 week of storage, as it was shown in previous studies that maximum water sorption occurs within the first 7 days after setting [25,30].

In the present study, both liners showed higher water solubility and sorption after 8 weeks compared with 1 week of storage. Similarly, Palin et al. [31] showed greater solubility of the studied materials as the storage time increased. Similarly, Zankuli et al. [28] reported higher solubility and water sorption after 1 year compared with 1 month.

The methodology of the current study was in accordance with ISO 2009:4049, but the samples were stored in artificial saliva to simulate normal clinical environment. The solubility of liners in acidic solution was

also investigated. No significant difference was found between acid solubility of studied liners after 1 week and 8 weeks of storage. To assess the acid resistance of the studied liners, lactic acid solution (pH = 3.0) was used. The purpose of using acidic solution was to simulate conditions created by bacterial biofilm and subsequent acid production adjacent to margins of a pulp capped tooth. In fact, we tried to simulate the changes in the pH of saliva in the oral cavity [32-34].

This study had an in vitro design. Thus, generalization of results to the clinical setting must be done with caution. Further studies are required on other properties of these materials. Also, clinical trials are recommended to assess the efficacy of ACTIVA BioACTIVE in comparison with other liners in the clinical situation.

Conclusion

Within the limitations of this in vitro study, it can be concluded that water sorption and solubility of Fuji Lining and ACTIVA increased over time. Water sorption and solubility of Fuji Lining were greater than those of ACTIVA within the first week after setting but they were similar in long-term assessment. Fuji Lining and ACTIVA were not different in terms of acid resistance.

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