Original Article

In-Vitro Evaluation of Water Sorption and Solubility of G-Cem and FujiCem in Water and Acid

H. Heshmat [1], S. Banava [1], P. Zarandi [2], F. Faraji [3].

[1]Assistant Professor, Department of Restorative Dentistry, School of Dentistry, Islamic Azad University, Tehran, Iran
[2]Dentist
[3]Dentistry Student, School of Dentistry, Islamic Azad University, Tehran, Iran

Abstract

Background and Aim: Solubility is among the negative characteristics of dental materials leading to structural degradation of cements and decreased longevity of restorations. The present study sought to assess the water sorption and solubility of FujiCem resin-modified glass ionomer (RMGI) and G-Cem self-adhesive resin cement in water and acid.

Materials and Methods: In this experimental study, 5 discs were fabricated from each understudy cement for solubility testing in lactic acid and 5 other disc-shaped specimens were made for evaluation of water sorption and solubility. Specimens were immersed in distilled water for 7 days and immediately weighed afterwards (M2). Discs were then transferred to a desiccator again to reach a constant weight of M3. Water sorption and solubility were calculated using the respective equations. For acid solubility testing, specimens were immersed in lactic acid for 24h. Independent t-test was applied for statistical analysis of results.

Results: Solubility in distilled water was 0.0000139±0.0001μg/m^3 for FujiCem and 0.0000016±0.000005μg/m^3 for G-Cem (p=0.480). Water sorption was 0.0000197±0.000234 for FujiCem and 0.0000025±0.00004 for G-Cem ($p<0.001$). Acid solubility was 0.0059414±0.0016 for FujiCem and 0.0039115±0.0016μg/m^3 for G-Cem (p=0.348).

Conclusion: The two understudy cements had no significant difference in water or acid solubility but FujiCem RMGI showed greater water sorption than G-Cem self-adhesive resin cement.

Key Words: Acid solubility, Water solubility, Water sorption, Resin modified glass ionomer, Resin cement

Corresponding author:
F. Faraji, Dentistry Student, School of Dentistry, Islamic Azad University, Tehran, Iran
Foad.faraji@yahoo.com

Received: 7 Jul 2012
Accepted: 18 April 2013

Journal of Islamic Dental Association of IRAN (JIDAI) Autumn 2013 ;25, (4)

Introduction

Clinical success of luting cements depends on various factors such asmechanical properties, biological effects on soft tissue and dental pulp, chemical bond to tooth structure, water solubility and water sorption of cements [1].

Solubility is among the negative characteristics of dental materials that leads to structural degradationof cements and reduced longevity and survival of restorations [2]. Solubility is weight loss per area or volume unit due to dissolution or decomposition of material within a time period at specific temperature in saliva or oral fluids [3]. On the other hand, water sorption causes an increase in volume of dental material that may compensate for polymerization shrinkage. However, it is associated with a drop in mechanical properties compromising the cement-tooth bond and causing subsequent microleakage of saliva and cariogenic microorganisms via the cement-tooth interface. Acids produced from the metabolic activity of microorganisms lower the pH and lead to tooth hyper-sensitivity, discoloration, caries recurrence and pulp injury [4-14]. Furthermore, entry of some organic constituents of dental materials into the body following their dissolution causes local and systemic allergic reactions [4, 9, 15]. One important test for dental materials is the comparison of their water sorption because pulp irritation is most probably caused by the function of bacteria rather than direct effect of restorative materials [16].

Luting cements are used for cementation of restorations [12] and since 1952 methyl methacrylate-based resin cements have been used for this purpose [17]. Conventional glass ionomers are capable of chemical bonding to enamel and dentin and fluoride release. Additionally, their modulus of elasticity is similar to that of dentin and they have modulus of thermal expansion similar to that of tooth structure. They also possess relatively high opacity [2, 18, 19]; but they are susceptible to moisture contamination and have a difficult isolation [19]. RMGIswere introduced in 1980s. Theyare now among the most commonly used base and liners due to their easier application, greater resistance to abrasion and higher esthetics compared to conventional glass ionomers [2, 18]. Furthermore, due to faster setting,theyhave less technical sensitivity [19] while maintaining their ability to bond to tooth structure and release fluoride [18]. These cementsare available in two types of self-cure and light-cure and the pack contains powder andliquid bottles [15].

Extensive investigations have been carried out on the solubility and water sorption of resin cements and RMGIs yielding controversial results [4-14]. The advantage of resin cements over glass ionomer cements is their less solubility and water sorption [20]. Considering all the above, this study sought to assess and compare the solubility and water sorption of G-Cem self-adhesive resin cement and Fuji-Cem RMGI.

Materials and Methods

This in-vitro experimental study was conducted on two base and liner materials according to ISO 4049:2000 (E) standards for evaluation of water solubility and water sorption and ISO 9917-1:2007 (E) standards for the assessment of acid solubility [16, 21]. Characteristics of the understudy materials are presented in Table 1.

For water solubility and water sorption testing, 5 discs were fabricated of each material and a total of 20 disc-shaped specimens measuring 15 mm in diameter and one mm in thickness were fabricated in stainless steel molds as follows [16, 22, 23].

G-Cem is offered in the form of capsule. The capsule was placed in an amalgamator and mixed according to the manufacturer's instructions. The mixture was transferred to moulds using a spatula at 25°C temperature and 50% relative humidity. FujiCem is offered in the form of two pastes. Specific amounts of pastes were mixed according to the manufacturer's instructions on a glass slab using a spatula under similar conditions as mentioned above. The obtained mixture was transferred with a spatula to stainless steel moulds measuring 15 mm in diameter and one mm in thickness. The moulds were over-filled with the materials. A celluloid strip was placed over the moulds and topped with a glass slab [7, 15, 24]. The specimens were light-cured using a Coltolux 2.5 (Coltolux, USA) light-curing unit with400 mw/cm^2 intensity three times from each side for 40s in an overlapping fashion. The materials were cured for a total duration of 120s with 0.5 mm distance between the specimen surface and the tip of the device. Specimens were then transferred to a desiccator containing fresh silica gel and stored at 37±1°C for 22h. Next, the specimens were transferred to another desiccator and stored at 23±2°C for 2 h to achieve optimal curing.

In the next step, the specimens were weighed using an electronic digital scale (ANDMADRIN, Japan) with microgram readability. Drying cycle was continued for each disc until reaching a stable weight of M1. After 5 weeks, specimens reached M1 weight and diameter (D) and height (h) of each specimen at the center of disc and four extra points at equal distances from the center were measured using a digital caliper (Mitutoyo, Japan). Volume of each specimen was also calculated and reported in mm^3.

Specimens were then immersed in distilled water and stored in an incubator at 37°C. Seven days later, specimens were removed from distilled water, rinsed with fresh distilled water, dried with absorbing paper, shook in the air for 15 s and weighed

Table 1: Characteristics of the understudy materials

Material	Manufacturing company	Constituents
G-Cem self-adhesive resin cement	GC USA	Ester monomer, di-methacrylate, 4-MET, UDMA, phosphoric acid, initiator, fluoroaluminosilicate radiopaque powder, fluoride-releasing glass and water
FujiCem RMGI	GC USA	HEMA monomer, Maleic and acrylic acid copolymer, comphorquinone photo-initiator, liquid initiator, fluoroaluminosilicate radiopaque powder, fluoride-releasing glass and water

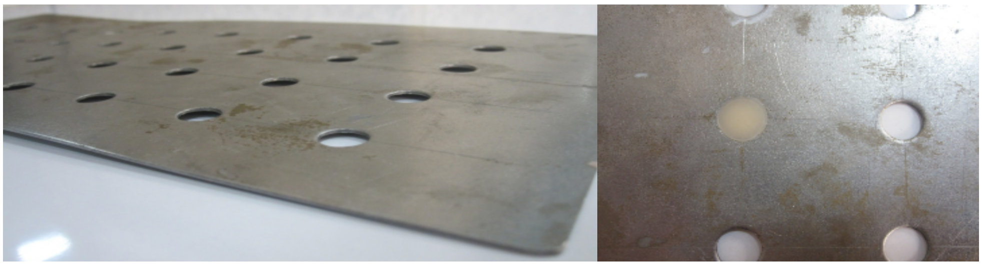

Figure 1: Disc-shaped moulds for fabrication of solubility testing specimens

one minute after removal from the incubator (M2). Specimens were then placed in a desiccator containing fresh silica gel and drying cycle was continued for 5 weeks until the specimens reached their final weight (M3).

Final volume of specimens (V2) was also calculated after the samples reached their final (M3) weight using the equation 1.

Equation 1: $V = [(D)/2]^2 \times \pi \times h$

Water sorption (W_{sp}) and water solubility (W_{sl}) were also calculated according to ISO 4049 2000 standard in $\mu g/mm^3$ using the following equations:

Equation 2: $W_{sp}=(M2-M3)/V2$

Equation 3: $W_{sl}=(M1-M3)/V2$

Acid solubility was measured according to ISO 9917-1: 2007 (E) standard using the erosion test as follows:

In order to prepare theerosive material, 8.27 g lactic acid (general purpose reagent grade or purer) and 0.92 g sodium lactate (general purpose reagent grade or purer) (Merck, Germany) were mixed with grade 3 water at least 18 h prior to the experiment according to ISO 3696:1987 standard. The pH of the solution was 2.74±0.02 and adjusted with 1 M/L sodium lactate solution or 1 M/L lactic acid using a digital pH-meter (Denver Instrument, USA).

Five square-shapedmoulds measuring 30x30 mm with 5 mm thickness or disc-shapedmoulds measuring 5±0.05 mm in diameter and 2±0.5 mm in depth at the center were fabricated of poly methyl methacrylate. Understudy materials were mixed according to the manufacturer's instructions at 23±1°C. To prevent voids, first one part of the mould was filled with the material and then the remaining material was appliedto the opposite end of the mould. Specimens were cured according to the recommended intensity and timing. The mouldswere placed on a plate and a glass slabwas placed over the cement surface. The entire complex was pressed using a clamp. After storage in an incubator at 37°C for 24h with relative humidity of 90%, the plate and the glass slab were separated from the mould and specimen surface was ground until reaching the level of mould using a 1200 grit abrasive discs under water irrigation. The acceptable difference between the level of specimen and mould was 5 microns. The height of mould was measured at 4 points near the center of specimen using a digital caliper. These 4 points had at least 90° distance from one another. The mean of these values was then calculated. Specimen thickness at the center was measured as well. The obtained two values were subtracted and D0 was obtained. Each mould along with its respective specimen was vertically placed in a separate tube containing 30 ml of acidic solution in a way that the specimen surface was facing up and at least 10±3 mm of the acidic solution covered the specimen surface. The tube was sealed and stored at 37°C for 24h. After this time period, the specimens were removed from the tubes and rinsed with distilled water. The depth at the center of each specimen was measured as the reference point with the use of mould margins to calculate the new depth (D_t). By subtracting the two values of D0 (depth at the center of specimen before placemen in acid in mm) and D_t (depth at the center of specimen after acid immersion in mm), the D value (depth of corrosion) was calculated as follows:

$D=D_t-D_0$

Data were analyzed using independent t-test.

amount of soluble residual monomer in the material standard. Monomer polymerization degree, the and comparable to those of other tests using the same and natural solubility of polymers and offer results materials. These tests evaluate the water dur ing mixing prevent polymer polymerization at the

Results

In this study, 5 specimens of each material and a total of 10 samples were evaluated in terms of water sorption and solubility in two groups of water and acid. No statistically significant difference was found between the two groups of G-Cem and Fuji-Cem in terms of water solubility (p=0.480) (Table 2). Table 2 also presentswater sorption of G-Cem and FujiCem. As observed in Table 2, a significant difference existed between the two cements and FujiCem showed higher water sorption (p=0.001). Table 3 reveals that no significant difference existed between the two understudy cements in terms of acid solubility (p=0.348).

Table 2: The mean and standard deviation of water solubility and water sorption in the understudy groups (mg/mm^3)

Cement	Water solubility Mean ±SD	Water sorption Mean ±SD
FujiCem	0/0000139±0/000100	0/0000197±0/000234
G-Cem	0/0000016±0/000005	0/0000025±0/000040
P value	0/480	0/001

Table 3: The mean and standard deviation of acid solubility in understudy groups (mm)

Cement/Solubility	Mean ±SD	P value
FujiCem	0/0059414±0/001600	
G-Cem	0/0039115±0/001600	0/348

Discussion

This experimental study aimed to compare water and acid solubility and water sorption of FujiCem RMGI and G-Cem self-adhesive resin cement. FujiCem had higher water sorption than G-Cem but in terms of water or acid solubility, no significant difference was observed between the two cements.

Filler content, size of filler particles, surface area of filler particles, coupling agents and type of particles can all affect the solubility of cements [1]. Assessment of solubility according to ISO standard is usually suggested for primary testing of different sorption speed of polymerization affect the solubility of materials. Voids entrapped in the material void surface and increase the solubility of the cement [4].

From an atomic point of view, the diffusion mechanism is gradual migration of atoms in the specimen volume. In general, in diffusion phenomenon, water penetrates into the polymeric compounds. In this phenomenon, called free volumetric theory, water penetrates into the porosities of the material without involvement with the polar molecules of the polymer [4].

In interaction theory, water penetrates into the material and successfully bonds with the hydrophilic groups of the compound [4]. Polymers have variable degrees of water sorption based on their microscopic and molecular structure. For instance, polarity of molecules, presence of hydroxyl groups that can form hydrogen bonds with water, matrix polymerization degree, presence of water absorbing constituents in the composition of material and type, volume percent and solubility of filler particles can all affect the water sorption of a material [1]. Curing and setting of RMGIs are based on acid-base reactions similar to conventional cements and also polymerization of free radicals in the resin part of these materials. Thus, as our study results showed as well, these cements are expected to have greater water sorption. In the resin part of Fuji Cem, a considerable amount of hydrophilic groups such as hydroxy ethyl methacrylate (HEMA) are present that act like hydrogen and absorb greater amounts of water [4].

In our study as well as many others, the highest amount of water sorption occurredwithin the first hours and days. For RMGIs, this increase in volume continued in the following days as well. This phenomenon is due to the completion of polymerization processwithin the mentioned time periods. Studies have reported variable results in terms of solubility depending on the physicochemical properties of different polymers [4]. That is the reason why cements manufactured by different companies show different results despite the similarity of their main constituents [13]. In other words, water sorption and solubility mainly depend on the type of material and the different results yielded by various studies on the same material are usually attributed to the difference in resin matrix compositions [25].

Mese et al, in their study in 2008 evaluated water sorption and solubility of luting cements. The results showed that GC Fuji required 3-4 weeks for stabilization in water. In our study, we allowed 5 weeks to reach a stable weight. Also, GC Fuji had the highest water sorption and percentage of changes in resin cements was significantly less than GC Fuji RMGI. Overall, their obtained results were in accord with ours [5].

Toledano et al, in their study evaluated water sorption and solubility of Fuji II LC RMGI in comparison to composite resins like Z100. As mentioned earlier, water sorption is a diffusion-dependent phenomenon that occurs in the organic resin matrix. Therefore, it depends on resin constituents and can significantly increase water sorption. Based on their obtained results, Vitremer and Fuji II LC that are both RMGIs had higher water sorption than Z100 and Prodigy composite resins due to the presence of hydrophilic compounds like HEMA in their composition [25].

Our study was carried out in accordance with ISO 4049: 2000 (E) and ISO 9917-1 2007 (E) standards. Therefore, our obtained results are comparable with those of other studies using the same standards.

Kanchanvasita et al, study was totally in accord with ISO 4049 standard and reported higher water sorption and water solubility of RMGIs compared to other resin-based materials [15].

Banava et al, in their study conducted in accordance with ISO 4049 and ISO 9917-1 standards compared Fuji lining GClight-cure RMGI and a base containing hydroxyapatite (Lime-Lite) in terms of solubility in distilled water, acid and artificial saliva and found no significant difference between the two [23].

Using profilometry, Eisenburger et al. compared the erosion of GI and zinc phosphate cements in presence of citric acid at different pH values and concluded that Ketac-Cem GI and resin-based Dual-Cement had less surface erosion than zinc phosphate cement [1].

Marghalini evaluated water and acid solubility of self-adhesive resin cements. All 4 types of understudy cements had greater solubility in acid than in water. GC cement had the highest and Rely X Unicem had the lowest solubility. He attributed higher solubility of G-Cem to the presence of UDMA that has been demonstrated to have higher solubility than Bis-GMA in many studies [26].

Conclusion

FujiCem RMGI had higher water sorption than G-Cem self-adhesive resin cement but the two cements were not significantly different in terms of solubility in water and acid.

Acknowledgement

The authors would like to express their gratitude to Dr. Mohammad JavadKharaziFard for statistical consultation, Dr. Farzaneh Agha Jaani and personnel of Tehran University Dental Research Center.

References

1- Eisenburger M, Addy M, Rossbach A. Acid solubility of luting cements. J Dent. 2003 Feb; 31 (2):137-42.

2- Roberson TM, Heymann H, Swift EJ. Sturtevant's art & science: Operative dentistry. 5th ed. USA: Mosby; 2006, 140. 174-182, 196-205, 310-312.

3- Powers JM, Sakaguchi R. Craig's restorative dental material. 12th ed. USA; Mosby; 2006, 46-47, 484-504.

4- Gerdolle DA, Mortier E, Jacquot B, Panighi MM. Water Sorption and water solubility of current luting cements: An in vitro study. Quintessence Int. 2008 Mar; 39(3):107-14.

5- Mese A, Burrow MF. Jyus MJ. Sorption and solubility of luting Cements in different solutions. Dent Material J. 2008 Sept; 27(5):702-9.

6- Archegas LR, Caldas DB, Rached RN, Vieira S. Sorption and solubility of composites cured with quartz tungsten halogen and light emitting diode light-curing Units. J Contemp Dent Pract. 2008 Feb; 1;9(2):73-80.

7- Zhang Y, Xu J. Effect of immersion in various media on the sorption, solubility, elution of unreacted monomers, and flexural properties of tow model dental composite composition. J Mater Sci Mater Med. 2008 Jun; 19(6):2477-2483.

8- McCabe J. Walls A. Applied dental materials. UK: Blackwell Oxford; 2008, Chap 23, 24.

9- Cefaly DF, Wang L, de Mello LL, dos Santos JL, dos Santos JR, Lauris JR. Water sorption of resin-modified glass-ionomer cements photo activated with LED. Braz Oral Res. 2006 Oct-Dec; 20 (4):342-6.

10-Malacarne J, Carvalho RM, de Goes MF, Svizero N, Pashley DH, Tay FR, Yiu CK, Carrilho MR. Water sorption /solubility of dental adhesive resins. Dent Mater. 11. 2006 Oct;22(10):973-80.

11- Ortengren U, Wellendorf H, Karlsson S, Ruyter IE. Water Sorption and solubility of dental composites and identification of monomers released in an aqueous environment. J Oral Rehabil. 2001 Dec; 28(12):1106-1115.

12- Kells BE, Kennedy JG. Temperature and humidity: Significant factors in resin/enamel bonding. Eur J Prosthodont Restor Dent. 1995 Dec; 3 (6):269-74.

13- Knoblock L A, Kerby RE, McMillen K. Clelland N. Solubility and sorption of resin–based luting cements. Oper Dent. 2000 Sep-Oct;25(5):434-40.

14- Yoshida K, Tanagawa M, Atsuta M. In-vitro solubility of three types of resin and conventional luting cements. J Oral Rehabil. 1998 Apr; 25(4): 285-91.

15- Kanchanavasita W, Anstice HM, Pearson GJ. Water sorption Characteristics of resin-modified glass ionomer cements. Biomat. 1997 Feb; 18(4): 343-9.

16- International standardization organization. ISO 4049: 2000 (E) Dental material: Guidance on testing of water sorption and solubility. Published in Switzerland. Order number: 220-2711 Downloaded. 2009-02-21.

17- Gerdolle DA, Invitro evaluation of microleakage of indirect composite inlays cemented with four luting agents. J Prosthet Dent. 2005 Jun; 93(6):563-70.

18- Craig RG, Powers JM. Restorative dental material. 11th ed. USA: Mosby; 2006, Chapter 4, 20.

19- Summit JB, Robbinson W, Schwartz R. A Fundamental of operative dentistry. 1st ed. China: Quintessence; 2001, 94, 96, 103, 219-224.

20- Eliades T, Editore Zohairy A, Feilzer AJ. Dent hard tissue and bonding. 1st.Berlin: Springer; 2005, 169.

21-International standardization organization. ISO 9917-1: 2007 (E) Dental Material: Guidance of testing of Acid Erosion. Published in Switzerland. Order number: 220-2711 Downloaded. 2009-02-21.

22-Cefaly DF, Wang L, de Mello LL, dos Santos JL, dos Santos JR, Lauris JR.Water sorption of resin-modified glass-ionomer cements photoactivated with LED. Braz Oral Res. 2006 Oct-Dec; 20(4):342-6.

23-Banava S, Noohi S, Aghajani F, Poorbaghi P, Kharazifard MJ. In vitro comparision of solubility of resin modified glass ionomer and a base containing hydroxyapatite in water, acid and artificial saliva. J Islamic Dent Ass. 2011 Winter; 23(4):262 -269. (Persian)

24-Toledano M, Osorio R, Osorio E, Fuentes V, Prati C, Garcia-Godoy F. Sorotion and solubility of resin-based restorative dental materials. J Dent. 2003 Jan; 31(1):43-50.

25-Marghalani HY. Sorption and solubility characteristics of self-adhesive resin cements. Dent Mater.2012 Oct; 28(10):187-98.