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Evaluation of Water Sorption and Solubility of Different Dental Cements at Different Time Interval

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Abstract **Background:** The clinical durability of dental cements is determined by dimensional stability. Water sorption and solubility of dental cements in distilled water have not been tested sufficiently. **Aims:** To evaluate and compare the water sorption and solubility of four different dental cements for permanent cementation at different time interval. **Materials and Methods:** Four different dental cements were selected: 1. Resin modified glass ionomer cement (Rely X luting2), 2. Poly acid modified composite resin cement (PermaCem Compomer), 3. Self-etching, self-adhesive dual cure composite resin cement (BeautiCem SA) and 4. Self-adhesive, dual cure composite resin cement (PermaCem 2.0). 120 discs were prepared, 30 discs for each group using Teflon molds ($15^{\pm}0.1$mm in diameter and $1^{\pm}0.1$ mm in thickness). The discs were dried in a desiccator at 37cº and weighted daily until a stable weight (M_1) was achieved using an electronic digital balance with accuracy of 0.0001g. The discs were immersed in distilled water for different time intervals (1, 7, 14, 21, 28 and 35 days) and the weight (M_2) was recorded. The discs were replaced in a desiccator and weighed daily until stable weight (M_3) was achieved. Water sorption and solubility in µg/mm³ were calculated using the following equations: $W_{sp}=(M_2-M_3)/V$; $W_{sl}=(M_1-M_3)/V$. Data analyzed using ANOVA and Tukey's Post hoc test. **Results:** The results showed that there was a significant difference between the different dental cements and time intervals for both water sorption and solubility. RelyX luting2 exhibited the highest water sorption and solubility values at different time interval, whereas Permacem 2.0 showed the lowest values ($P \le 0.05$). **Conclusion:** Four types of dental cements of different compositions showed different behavior on water sorption and solubility. Resin cements are recommended for the permanent cementation of indirect restorations. Dental cements exhibited a time dependent increase in both water solubility and sorption.

Keywords: *resin cement, HEMA, water solubility, water sorption*

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1. Introduction

Dental treatments require attachment of indirect restorations to the teeth by means of the dental luting cement, which is a term that implies to a moldable substance that seal a space or to attach two components together such as teeth and dental prostheses. [1] The dental cements must have the ability of long term ageing in an oral environment, especially at the marginal gap of the indirect restorations, where the cement is in direct contact with a saliva which contains water soluble components. Clinically, the performance and durability of the dental cements depend on many factors such as the structural integrity and dimensional stability of the cements in the oral environment. [2,3] With the advancement of dental technology, The dental cements consisting of resin matrix such as a resin modified glass ionomer (RMGIC), a poly acid modified composite resin cement (Compomer) and self-etching, self-adhesive composite resin cement are becoming widely used for permanent cementation of metal-ceramic, all ceramic restorations, porcelain veneers, and more recently laminate, inlay, onlay restorations. [4,5]

Resin modified glass ionomer cement (RMGIC) has the advantages of conventional Glass Ionomer Cements (GIC), such as adhesion to the tooth structure and fluoride release, with the advantages of composite resin cement such as improvement of the strength, water sorption and solubility. [6] Compomer cement has recently developed as a new kind of dental cement. It also provides the combined benefits of composite resin cements the (comp) in their name and glass ionomer cements (omer). Self-etching and self-adhesive resin cements are newly introduced to minimize the steps required for application of the dental cements. [2,4]

Water sorption (W_{sp}) of resin materials is a diffusion controlled process that cause breakdown of an inorganic filler and organic matrix bond and release of un bonded monomers leading to chemical dissolution of the materials. [7] The elution of un bonded monomers leads to mass reduction of the cements and is considered as water solubility (W_{sl}). [2,8] The phenomena of water sorption and solubility of the cements may have detrimental effect such as degradation of cement, break in the margin contours leading to micro leakage that promotes secondary caries, pulpal inflammation, periodontal disease as well as fracture of the restoration by hygroscopic expansion. [2,9-13] Several studies demonstrated that water sorption and solubility of dental cements may also affect the mechanical properties of the dental cements such as the hardness, flexural strength. [14,15,16] For these reasons, the water solubility and sorption behavior of the cements should be tested clinically and in the laboratory to evaluate the compatibility of them for clinical applications.

The null hypothesis to be tested in this study was neither the type of dental cements nor storage time interval will affect the water solubility and sorption of the dental cements. The aims of this study was to evaluate and compare the water sorption and solubility of four different dental cements for permanent cementation at different time interval.

2. Materials and Methods

This an in vitro study was conducted on four different dental cements for evaluation of water sorption and solubility properties. The tested dental cements, (Figure 1) were used: 1. A resin modified glass ionomer cement (RelyX luting2, 3M ESPE, USA) composed of paste A: fluoroaluminosilicate glass, HEMA, water. Paste B: Methacrylated poly carboxylic acid, BIS-GMA, HEMA, water, potassium per sulfate, zirconia silica filler. 2. A poly acid modified composite resin cement (PermaCem Compomer, DMG, Hamburg, Germany) composed of: Ionomer glass in a BIS-GMA based matrix consisting of dental resins, HEMA. 3. Fluoride releasing, self-etching, self-adhesive dual cure composite resin cement (BeautiCem SA, SHOFU Inc Corp., Japan) composed of: paste A:UDMA, fluoroboroaluminosilicate glass, silicate glass. Paste B: HEMA, UDMA, BIS-GMA, carboxylic acid monomer, phosphoric acid monomer, zirconium silicate. Inorganic filler contents: 59.6 wt% and 4. Self-adhesive, dual cure composite resin cement (PermaCem 2.0, DMG, Hamburg, Germany) composed of: BIS-GMA based matrix, TEGDMA, Barium glass filler. Inorganic filler contents: 69 wt%.

Figure 1. Tested dental cements

2.1. Specimen Preparation

One hundred twenty specimens were fabricated representing four main groups according to the type of cements, thirty specimens for each cement as follows:

Group I: RelyX luting2 (resin modified glass ionomer cement).

Group II: PermaCem Compomer (polyacid modified composite resin cement).

Group III: BeautiCem SA (self-etching, self-adhesive composite resin cement).

Group IV: PermaCem 2.0 (self-adhesive composite resin cement).

Each main group divided into six subgroups of five specimens according to storage periods in distilled water (one, seven, fourteen, twenty one, twenty eight and thirty five days). All specimens were prepared according to manufactures' instructions using Teflon molds $15^{\pm}0.1$mm in diameter and $1^{\pm}0.1$ mm in thickness. Preparation of specimens of the cements was carried out as follows: a thin celluloid matrix strip was put on a glass slide and over it the mold in which the cement was packed. Another celluloid matrix strip was put on top of the cement in the mold and was covered with a second glass slide. The cements were light cured for 20 seconds for each side using light curing unit with a light output of 1000 mw/cm^2 (LED. F, WOOD PECKER, China). The cements were allowed to chemically set for 5 minutes and then were removed from the mold.

2.2. Water Sorption and Solubility Test

Figure 2. Electronic digital balance

The discs were immediately weighed using an electronic digital balance with an accuracy of 0.0001g (KERN & Sohn GmbH, Version 1.3, Germany) (Figure 2), then the specimens were placed into the individual containers containing fresh silica gel at 37°C and weighted daily until a constant weight (M1) was achieved. Then the discs were immersed individually in a poly propylene containers with 10 ml of distilled water and stored in an incubator at 37°C for different time intervals. At the end of each storage period, the discs were removed from the containers, dried with absorbed paper and weighted (M_2). After that the specimens were placed into the individual desiccators at 37°C and weighted until constant weight was achieved (M_3). The volume V (mm^3) was calculated according to the equation V=$\pi * r^2 *$h. The value of water sorption (W_{sp}) and solubility (W_{sl}) in µg/mm³ was calculated according to the equations:

$$W_{sp} = \left(M_2 - M_3\right) / V$$

$$W_{sl} = \left(M_1 - M_3\right) / V$$

Where
M_1=Specimen weight before immersion in (µg).
M_2 = Specimen weight after immersion in (µg).
M_3= Specimen weight after immersion and desiccation in (µg).
V = Specimen volume in (mm³).

2.3. Statistical Analysis

The collected data from the water sorption and solubility tests were analyzed statistically using the SPSS software (version 19.0; SPSS Inc., Chicago, IL, USA). One-way ANOVA is applied in order to determine if there was a significant difference among groups and Tukey's Post hoc test to compare between significant groups. All statistical tests were conducted at 0.05 levels of significant.

3. Results

The mean and standard deviation of water sorption and solubility for the different dental cements at different storage times are listed in (Table 1 and Table 2). For each storage time, One-way ANOVA showed that there was a significant difference in the mean values of water sorption and solubility among the different dental cements ($P \leq 0.05$). Tukey's Post hoc test showed that RelyX luting2 had the highest mean values of both parameters among the different dental cements followed by PermaCem Compomer and BeautiCem SA, while PermaCem 2.0 showed the lowest mean values as shown in (Table 1 and Table 2).

For each dental cement, One-way ANOVA showed that there was a significant difference in the mean values of water sorption and solubility among different storage times ($P \leq 0.05$). Thirty five days storage time of all dental cements showed significantly higher means of both parameters than other storage time. Generally, the increase of storage periods leaded to increase of water sorption and solubility of all cements as shown in (Figure 3 and Figure 4).

Table 1. Mean, standard deviation of water sorption (µg/mm³) for the different dental cements at each time interval.

Time	Materials	Means ±SD	F value	P value
1 day	RelyX luting2	323.8499 ± 7.65 **A**	2810.159	.000
	PermaCem Compomer	132.0311 ± 2.44 **B**		
	BeautiCem SA	72.5831 ± 6.37 **C**		
	PermaCem 2.0	52.6538 ± 1.91 **D**		
7 days	RelyX luting2	431.5357 ± 9.66 **A**	2655.728	.000
	PermaCem Compomer	182.4203 ± 4.48 **B**		
	BeautiCem SA	103.3828 ± 8.07 **C**		
	PermaCem 2.0	78.2448 ± 4.14 **D**		
14 days	RelyX luting2	517.3672 ± 7.81 **A**	1783.772	.000
	PermaCem Compomer	261.0049 ± 14.39 **B**		
	BeautiCem SA	197.0275 ± 5.01 **C**		
	PermaCem 2.0	146.7515 ± 3.26 **D**		
21 days	RelyX luting2	545.6758 ± 19.63 **A**	428.748	.000
	PermaCem Compomer	328.9454 ± 15.66 **B**		
	BeautiCem SA	265.7607 ± 18.89 **C**		
	PermaCem 2.0	207.4451 ± 5.49 **D**		
28 days	RelyX luting2	570.1344 ± 5.06 **A**	3159.119	.000
	PermaCem Compomer	364.2745 ± 3.09 **B**		
	BeautiCem SA	300.8633 ± 9.23 **C**		
	PermaCem 2.0	245.2653 ± 2.63 **D**		
35 days	RelyX luting2	593.0077 ± 3.33 **A**	12141.457	.000
	PermaCem Compomer	381.7126 ± 1.08 **B**		
	BeautiCem SA	331.2101 ± 2.62 **C**		
	PermaCem 2.0	274.0268 ± 3.55 **D**		

SD= Standard deviation, different letters are statistically significantly different according to Tukey's test.

Table 2. Mean, standard deviation of water solubility (μg/mm³) for the different dental cements at each time interval

Time	Materials	Means ± SD	F value	P value
1 day	RelyX luting2	121.1605 ± 4.58 **A**	709.545	.000
	PermaCem Compomer	83.6800 ± 2.75 **B**		
	BeautiCem SA	43.4819 ± 3.33 **C**		
	PermaCem 2.0	28.7614 ± 3.03 **D**		
7 days	RelyX luting2	146.1853 ± 2.57 **A**	329.943	.000
	PermaCem Compomer	99.8726 ± 1.52 **B**		
	BeautiCem SA	74.1683 ± 8.13 **C**		
	PermaCem 2.0	58.9950 ± 3.63 **D**		
14 days	RelyX luting2	172.6821 ± 3.15 **A**	435.528	.000
	PermaCem Compomer	141.4295 ± 4.18 **B**		
	BeautiCem SA	99.7593 ± 7.10 **C**		
	PermaCem 2.0	76.4333 ± 2.53 **D**		
21 days	RelyX luting2	208.0112 ± 7.95 **A**	415.302	.000
	PermaCem Compomer	166.7940 ± 3.01 **B**		
	BeautiCem SA	130.5586 ± 5.67 **C**		
	PermaCem 2.0	95.1167 ± 2.91 **D**		
28 days	RelyX luting2	298.1457 ± 7.38 **A**	1457.330	.000
	PermaCem Compomer	198.6128 ± 2.97 **B**		
	BeautiCem SA	147.4309 ± 3.26 **C**		
	PermaCem 2.0	115.0459 ± 3.69 **D**		
35 days	RelyX luting2	321.8117 ± 3.76 **A**	1444.439	.000
	PermaCem Compomer	234.9610 ± 7.91 **B**		
	BeautiCem SA	176.1924 ± 2.97 **C**		
	PermaCem 2.0	132.8237 ± 2.61 **D**		

SD= Standard deviation, different letters are statistically significantly different according to Tukey's test.

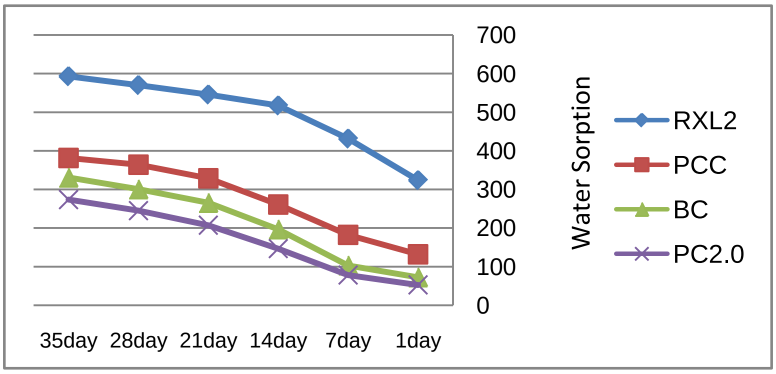

Figure 3. Water sorption (μg/mm³) of each cement at different time intervals

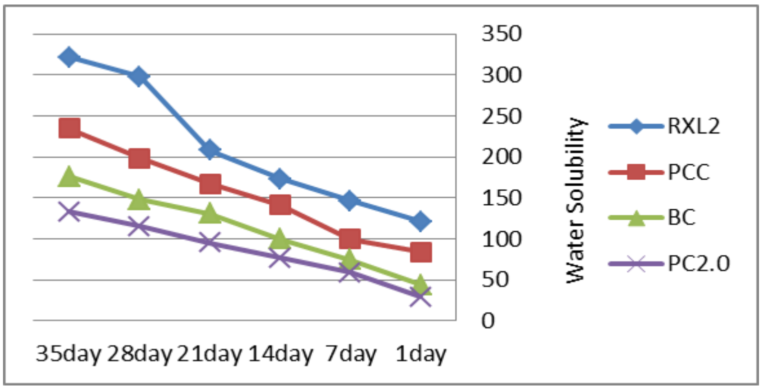

Figure 4. Water solubility (μg/mm³) of each cement at different time intervals

4. Discussion

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, solubility, flexural and bond strength are influenced by the filler contents and the monomers structure and subsequently affect the durability of the restorations. [17,18] Generally, in a moist oral environment the polymers of resinous materials absorb water. The interaction between these materials and water includes two phenomena: (1) Water sorption which leads to an increase in weight of the material. (2) Dissolution of the material (fillers or monomers) and release them into the water contributing to the reduction of the final weight of the material that can be measured as solubility. [19,20]

The results of statistical analysis (Table 1 and Table 2) revealed that there was a significant difference in water sorption and solubility values among four different dental cements tested at all storage period; this was believed to be related to several factors such as the chemical composition of the cements, hydrophilicity of their monomers, the concentration and nature of the fillers. [2,8,9,17,21]

A resin modified glass ionomer cement (RelyX Luting2) revealed significantly higher water sorption and solubility values at all storage periods compared to the other dental cements were tested. This may be associated with a dual mode of setting reactions of RMGIC that involving an acid-base reaction and a free radical polymerization. It absorbs lots of water at the begging, then sorption process decrease due to the presence of the resin compounds which minimize the diffusion of water. [18,22,23,24] The main polymerized structure of RMGIC is hydroxyl ethyl methacrylate monomer (HEMA) which is a highly hydrophilic monomer and having high polarity and level of hydrogen bonds with water. It responsible for absorb huge amount of water both in cured and uncured states. [25,26] The clinical implications of a high W_{sp} of RMGIC is the elevation of hygroscopic expansion that leads to unfavorable forces on the tooth structure and the restorations. Therefore, the use of this type of dental cement for permanent cementation particularly for All Ceramic restorations remains questionable.

PermaCem Compomer showed high water sorption and solubility values with significant differences when compared to BeautiCem SA and PermaCem 2.0 because it consists of HEMA monomer and fluoroaluminosilicate glass which is an ion-leachable glass which embedded to a polymeric matrix as in RMGIC that have hydrophilic nature and would be anticipated to reveal more water sorption. [22,23] While, it had lower values than RelyX Luting2 because it contains less concentration of HEMA and it modified by the attachment of polymerizable acid methacrylates that increase the number of inter network links and decease water uptake. [18,22]

BeautiCem SA exhibited higher water sorption and solubility than PermaCem 2.0 at all storage periods. This is attributed to the differences in their compositions and reactions with water. BeautiCemSA is a self-etching resin cement which consists of carboxyl, phosphate acidic functional groups and Urethane dimethacrylate (UDMA) monomers that have strong hydrophilic characteristics and more prone to water sorption. [2,18,26,27,28] While, PermaCem 2.0 had the minimal values of both parameters because it depends on Bisphenol A glycidyl Methacrylate monomer (BIS-GMA) and Triethylene glycol dimethacrylate monomer (TEGDMA) that are basically hydrophobic in nature. In addition, PermaCem 2.0 has higher inorganic filler contents 69 wt% . While, BeautiCem SA 59.6 wt% of inorganic fillers. This was in agreement with other studies that demonstrated that the resin materials with a low filler contents showed higher water sorption and solubility. [17,18,29,30] The results of this study agree with many previous studies that reported RMGIC and Compomer that immersed in distilled water showed high W_{sp} and W_{sl} as compared with resin cements. [16,22,25,31,32,33]

Regarding the effect of storage times on water sorption and solubility of the dental cements, the results showed that the storage times had a significant effect on both parameters. In general, there was continues increasing in water sorption and solubility from 1to35 days for the different cements as shown in (Figure 3 and 4). This may be due to the continues diffusion of a lot of water along the filler matrix interface, this diffusion requires a longer time in contrast to a reaction at the surface. [30] The increase of storage period leads to increase of water sorption and solubility of resin materials. [34] In addition, the tested cements showed high water uptake between 1and14 days of immersion. After that, the specimens absorbed constantly amount of water until they reached saturation state. This may be due to continuous setting reaction which occur after the initial light exposure. It was demonstrated that the rate of water sorption of resin materials depends upon the concentration of the polymer network and increased proportionally to HEMA concentrations. [35] This study supports several previous studies that proved the water sorption of resinous materials significantly increase at 1,7 and14 days followed by gradual sorption over a long period of time until a balance state was obtained. [9,30,36,37]

5. Conclusion

Under the limitation of this study, four types of dental cements of different compositions showed different behavior on water sorption and solubility. Resin cements are recommended for the permanent cementation of indirect restorations. While, a resin modified glass ionomer cement may be unsuitable. Dental cements exhibited a time dependent increase in both water sorption and solubility.

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