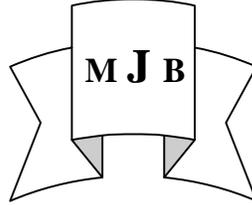


## Water Sorption and Solubility of Different Commercially Available Dental Cements. (An in Vitro Study)

Ahmed Ghanim M.

\* Dept. of Conservative Dentistry, College of Dentistry, University of Babylon.



### **Abstract**

**Background:** A variety of cements have been used in dentistry through many years for two primary purposes: as restorative filling materials, either alone or with other materials, and to retain restorations or appliances in a fixed position within the mouth. In addition, certain cements are used for specialized purposes in the restorative, endodontic, orthodontic, periodontic, and surgical fields of dentistry. Cement solubility and water sorption could be a primary cause of restoration failure.

**Aim of study:** To evaluate the water sorption and solubility of different commercially available dental cement materials.

**Materials and Methods:** Thirty disks (9×2 mm) of zinc polycarboxylate, zinc phosphate and glass ionomer cements (ten disks for each one) were prepared according to manufactures instructions. After setting, they were desiccated and weighed and each specimen were immersed in distil water for 7 days, then removed and weighed again. Then disks were again desiccated and weighed. Solubility and water sorption values were calculated from these different measures.

**Results and Conclusion:** The results shows that the polycarboxylate cement show the highest value of water sorption and water solubility followed by zinc phosphate cement. Glass ionomer cement shows the lowest values of the three materials tested regarding both water sorption and water solubility.

### **الخلاصة**

**الخلفية:** هنالك انواع مختلفة من الاسمنت قد استعملت في طب الاسنان على مر السنين لغرضين رئيسيين: كمواد تعويضية للحشوات اما لوحدها او مع مواد اخرى، وكذلك استعملت من اجل ابقاء التعويضات او التركيبات في موقع ثابت داخل الفم. بالإضافة الى ذلك، بعض انواع الاسمنت تستخدم لأغراض خاصة في المجالات التعويضية، التقويمية، طب اسنان الأطفال وفي مجال طب الاسنان الجراحي. ان امتصاص الاسمنت للماء و ذوبانه فيه قد يكون السبب الاول لفشل التعويضات. ان الغرض من هذا البحث هو لقياس امتصاص الماء و الذوبان فية لعدد من الاسمنت المتوافر في السوق.

**المواد و الطرق:** ثلاثين قرص (9×2) من فوسفات الزنك و بوليكاربوكسيلات الزنك و الكلاس ايونومر (عشرة اقراص لكل نوع) تم تحضيرها حسب توصيات المصنعين. بعد ان تصلبت تم تجفيفها ووزنها وكل عينة تم تغطيتها في ماء مقطر لمدة سبعة ايام ثم اخرجت و تم وزنها من جديد. ثم تم تجفيفها من جديد ووزنت. قيم امتصاص الماء و الذوبان فيه تم حسابها من الاختلاف ما بين هذه القياسات.

**النتائج و المناقشة:** النتائج اوضحت ان اسمنت البوليكاربوكسيلات اظهر اعلى قيم في امتصاص الماء و الذوبان فيه تبعه اسمنت الفوسفات. اسمنت الكلاس ايونومر اظهر اقل قيم بين المواد التي تم فحصها لكلا امتصاص الماء و الذوبان فيه.

### **Introduction**

Although dental cements are used in small quantities, they are the most important materials in clinical dentistry because of their application as luting agents, orthodontic attachments, cavity linings and bases,

and restorations for teeth. These multiple uses of dental cements require more than one type of cement; since no one material has yet been developed that can perform all the desirable requirements, these different applications require different physical

properties and appropriate clinical manipulative characteristics [1]. Solubility and water sorption is an important feature in assessing the clinical durability of dental cements. Consequently, solubility of dental cements has been widely evaluated both *in vitro* and *in vivo* [2-4].

Water sorption and solubility may cause degradation of the cement, leading to debonding of the restoration and recurrent decay [2, 5, 9]. However, most of these tests are static solubility tests, unrelated to the conditions found in the oral environment, and in particular, applied only to short-term solubility. While some investigators study the solubility in dynamic state (different pH) [6, 7]. It's clear that the clinical success of fixed partial prostheses is heavily dependent on the cementation procedure, because dental cement must be used to act as a barrier against microleakage [8, 15]. Dental cements can degrade when exposed to saliva in the mouth, and the resulting gap between the tooth and the restoration predisposes the tooth to caries, post operative hypersensitivity, pulpal inflammation and periodontal disease [9]. Water sorption and solubility of cements leads to dimensional changes, loss of retention, staining and breaking in margin contours and may affect the mechanical behavior such as the flexural strength, Vickers hardness and mechanical stability [10-13].

The solubility of dental luting cements influences both their rate of degradation and their biological compatibility [14]. Because of this, the water sorption and solubility of dental cements are of considerable clinical importance and can not be overlooked.

#### **Materials and Methodes**

The ADA specification #8 (zinc phosphate cement solubility) [24] was adapted with few modification to

design the methodology used in this study, distilled water used as storage media as the ADA specification #8 suggests, and the storage time were 1 week [16].

The materials used in this study and its composition are listed in table (1) and shown in figure (1).

Thirty discs were prepared (ten discs for each material) measured (9 mm in diameter and 2 mm in thicknesses). The powder –liquid ratio and mixing of the components of each material was carried according to manufacturer instructions.

The samples were prepared using a specially designed plastic syringe (with a stopper on its body) (figure 2), the resulting space inside the syringe has 9 mm diameter and 2 mm thickness. The syringe were loaded with the cement material with slight excess and pressed against a polyester strip placed on glass slab, after setting of the cement we remove the excess material and remove the stopper of syringe and push the plunger to extrude the disc of cement. Then the samples weighed with precision weighing scale (Denver instruments MXX-123-USA) (figure 3), the initial weight is termed ( $W_1$ ). Immediately after weighing the samples, they were immersed in individually numbered distilled water tubes and held in stand (figure 4) at 37 °C for 1 week in an incubator (Mettler, Germany) (figure 5), removed and weighing again ( $W_2$ ). The samples then dehydrated in an oven at 37 °C for 24 h and weighed again ( $W_3$ ).

The loss of material (solubility) was obtained from the difference between the initial and final drying mass of each sample ( $W_1-W_3$ ).

The water sorption was obtained from the difference between initial weighing and the wet weighing ( $W_2-W_1$ ).

The values of water sorption ( $W_{sp}$ ) and solubility ( $W_{sol}$ ), in  $\mu\text{g}/\text{mm}^3$  for

each sample were calculated using the following equations [10, 17]:

$$W_{sp} = (W_2 - W_1) / V$$

$$W_{sol} = (W_1 - W_3) / V$$

Where **V** is the volume of sample in mm<sup>3</sup> = (127.17 mm<sup>3</sup>).

The data were subjected to one-way ANOVA, and LSD test at a 0.05 significance level.

**Table 1** Materials used in this study

Material	Compositions	Batch #	Manufacturer
Adhesor (zinc phosphate)	<b>Powder:</b> zinc oxide, magnesium oxide, aluminum trihydroxide and boron trioxide. <b>Liquid:</b> aqueous solution of phosphoric acid and aluminum orthophosphate.	N1-1911639 Exp. 12-2013	Sprofadental a.s. Marakova- CZ
Adhesor carbofine (zinc poly carboxylate)	<b>Powder:</b> oxides (Zn, Mg, Al), boric acid. <b>Liquid:</b> acrylic acid, maleic acid anhydride, distilled water.	1880391-2 Exp. 11-2013	Sprofadental a.s. Marakova- CZ
Medicem (glass ionomer )	Poly acrylic acid , fluoro silicate and parabens	0844212 Exp. 03-2014	Promedica Germany



**Figure 1** Materials used in this study



**Figure 2** the syringe used in this study



**Figure 3** Precision weighing device



**Figure 4** Labeled tubes and stand



**Figure 5** Incubator

### **Results**

The Mean values of W1, W2, W3, for all materials used in this study are shown in table (2).

The water sorption and water solubility are shown in table (3).

The mean values shows that the polycarboxylate cement show the highest value of water sorption and water solubility followed by zinc phosphate cement. Glass ionomer cement shows the lowest values of the

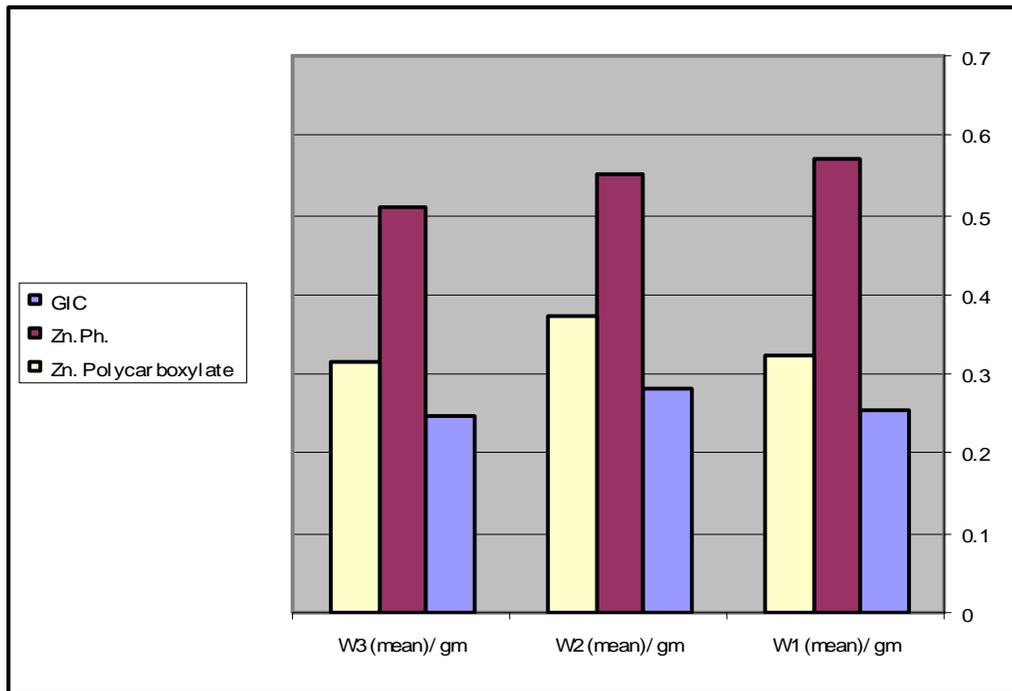
three materials tested regarding both water sorption and water solubility as shown in figure (6) and figure (7).

**Table 2** Mean values in gram for weighing.

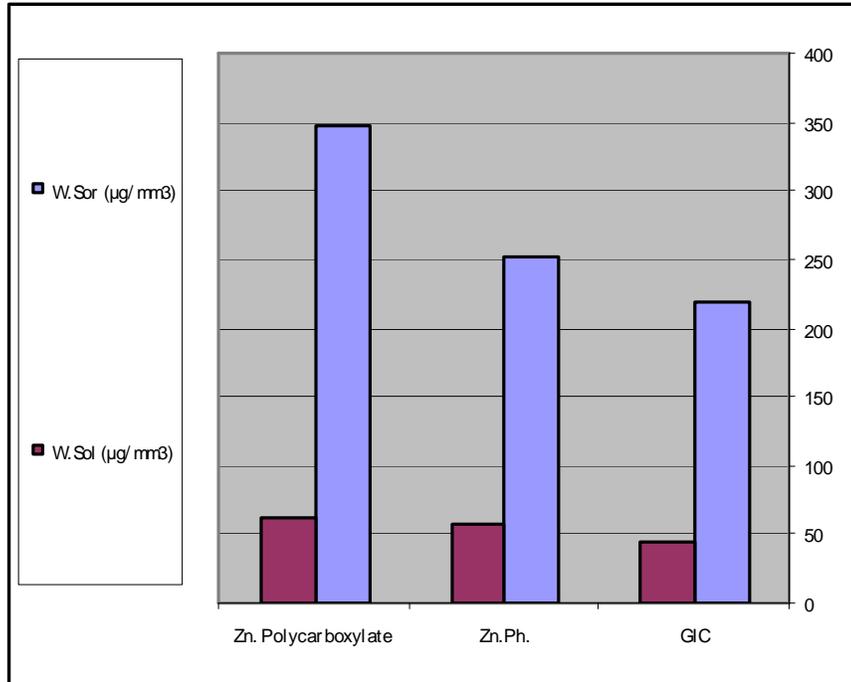
Material	W1		W2		W3	
	(mean)/gm	gμ	(mean)/gm	gμ	(mean)/gm	gμ
GIC	0.2538	253800	0.2804	280400	0.2481	248100
Zn.Ph.	0.5179	517900	0.5499	549900	0.5107	510700
Zn. Polycarboxylate	0.3234	323400	0.3723	372300	0.31478	314780

**Table 3** mean values in  $\mu\text{g}/\text{mm}^3$  of W.Sor and W.Sol. for all materials.

Material	W.Sor ( $\mu\text{g}/\text{mm}^3$ )	W.Sol ( $\mu\text{g}/\text{mm}^3$ )
GIC	219.17	44.82
Zn.Ph.	251.63	56.62
Zn. Polycarboxylate	345.99	61.34



**Figure 6** Bar chart showing the mean values for weighing in grams.



**Figure 7** Bar chart showing water sorption and water solubility in  $\mu\text{g}/\text{mm}^3$ .

The water sorption values showed correlation with the solubility values: the higher the water sorption, the greater the solubility for the evaluated materials. The data were subjected to

one-way ANOVA, and LSD test at a 0.05 significance level.

For water sorption, ANOVA test shows significant difference among the materials tested ( $p < 0.05$ ) (table (4)).

**Table 4** ANOVA test for sorption values

	Sum Squares	df	Mean Square	F	Sig.
Between Groups	156289.021	2	78144.511	7.836	.002
Within Groups	259289.805	26	9972.685		
Total	415578.826	28			

Post hoc (LSD) test for water sorption values shows a significant difference between all materials ( $p < 0.05$ ) except

between zinc phosphate and glass ionomer cements (table 5).

**Table 5** LSD test for sorption values

(I) Material Type	(J) Material Type	Mean Difference (I-J)	Std. Error	Sig.	95% Confidence Interval	
					Lower Bound	Upper Bound
GIC	Zn.Ph	-42.462845-	44.660239	.350	-134.26328-	49.33759
	Zn.Poly	-1.752685E2	45.884034	.001	-269.58443-	-80.95247-
Zn.Ph	GIC	42.462845	44.660239	.350	-49.33759-	134.26328
	Zn.Poly	-1.328056E2	45.884034	.008	-227.12159-	-38.48962-
Zn.Poly	GIC	175.268451*	45.884034	.001	80.95247	269.58443
	Zn.Ph	132.805606*	45.884034	.008	38.48962	227.12159

\*. The mean difference is significant at the 0.05 level.

For water solubility, ANOVA test shows non significant difference among the materials tested ( $p < 0.05$ ) (table (6)), however the mean values

shows that zinc poly carboxylate has greater solubility values followed by zinc phosphate, the glass ionomer has the least values.

**Table 6** ANOVA test for solubility values

	Sum of Squares	df	Mean Square	F	Sig.
Between Groups	2581.483	2	1290.741	.378	.689
Within Groups	88775.752	26	3414.452		
Total	91357.234	28			

Post hoc (LSD) test for water solubility values shows a non

significant difference between all materials ( $p < 0.05$ ) (table (7)).

**Table 7** LSD test for solubility values

(I) Material Type	(J) Material Type	Mean Difference (I-J)	Std. Error	Sig.	95% Confidence Interval	
					Lower Bound	Upper Bound
GIC	Zn.Ph	-11.795235-	26.132172	.655	-65.51068-	41.92021
	Zn.Poly	-23.328353-	26.848254	.393	-78.51573-	31.85902
Zn.Ph	GIC	11.795235	26.132172	.655	-41.92021-	65.51068
	Zn.Poly	-11.533118-	26.848254	.671	-66.72050-	43.65426
Zn.Poly	GIC	23.328353	26.848254	.393	-31.85902-	78.51573
	Zn.Ph	11.533118	26.848254	.671	-43.65426-	66.72050

**Discussion**

The water sorption and solubility of dental restorative materials are of considerable clinical importance and cannot be neglected [10, 18]. According to Tae Hyung Kim [19], high strength and low solubility are

desirable for any base or lining material. A strong base material is needed to support the overlying restoration and the subsequent occlusal forces acting upon it during clinical function. Some reduction observed in compressive strength may be attributed

to dissolution or water sorption during storage. Moreover, marginal infiltration may influence on liners hardness, which reinforces the need for studies related to their physical properties.

The water sorption measurements actually measured the net gain in weight of a specimen as a result of the ingress of water molecules and egress of monomers and other small molecules [20]. From an atomic point of view diffusion mechanisms are a stepwise migration from one site to another. Generally two patterns are known for diffusion of water through polymeric materials [21]: one is the pattern following the (**free volumetric theory**), in which the water diffuses through a microvoids without any mutual relationship to the polar molecules in the material. The other pattern is called (**interaction theory**), in which water diffuses through material binding successively to the hydrophilic groups. In the case were there was a negative correlation between the diffusion and equilibrium water uptake, the later pattern of diffusion was supposed to occur mainly. Recently it has been assumed that both approaches could be valid, each one for a defined specimen family or both simultaneously.

Solubility is the ability of a substance to dissolve in another, expressed as the concentration of saturated solution of the former in the latter. When solubility is tested, there is no particle in suspension (the solvent remains limp) [30].

This study was aimed at elucidating essential values for the evaluation of the quality of each employed material, which is of important clinical applicability. In fact, lining, base and luting materials have to be resistant to dissolution in water, organic solvents and acid-etching solutions, in order to maintain their pulp protective effect

[22]. In addition, dissolved and smeared cement may contaminate acid etched enamel, and produce an inferior bond, which is not desirable [23].

The choice for the three kinds of cements here evaluated was based on the fact that Zinc phosphate, Zinc polycarboxylate and Glass ionomer cement are the most commonly used conventional lining and luting materials in the clinical practice.

Water sorption and solubility tests were applied according to the ADA's #8 specification [24], though with few alterations to meet the objectives of this study.

The results of this study shows higher mean values (both sorption and solubility) for poly carboxylate cement followed by zinc phosphate and the least values were for glass ionomer cement.

The results of Yoruc and Karaaslan [1] showed that commercial dental polycarboxylate cements absorbed most of the water within 1st day of water storage. They continued to absorb the water at a slower rate for 28 days until equilibrium was reached. They further assumed that the results of their investigation showed that the water absorption of the commercial dental polycarboxylate cements was significantly depend on material composition.

Zinc polycarboxylate cement is a water-based material that hardens following an acid-base reactions between zinc-rich powder and an aqueous solution of polyacrylic acid [25]. The hydrophilic nature of a polymer is a function of the chemistry of its monomers and polymerization linkages. The presence of hydroxyl, carboxyl and phosphate groups in monomers and their resultant polymer make them more hydrophilic and more prone to water sorption [13], these cements include water in their formulation.

Glass ionomer cements are sensitive to water erosion [26]; it may be due to same hydrolysis of the cement components, this phenomenon is apparently aggravated in oral environment due to presence of aggressive compounds in saliva. Clinical success of glass ionomer cements depends on early protection from hydration and dehydration; it's weakened by early exposure to moisture, while desiccation on the other hand causes shrinkage and cracks [10]. Deniz et al [27] found that higher levels of solubility were associated with earlier exposures of mixed cement to water, and glass ionomer luting cements were highly sensitive to water contact during the first 6 minutes after mixing.

It was reported in previous studies that long-time storage of dental cements in water affected the mechanical properties of the cements [1, 14]. Cattani-Lorente et al [11] found that deterioration of the physical properties of the cements after long-term storage in an aqueous environment could be related to the water absorption of these materials. Part of the absorbed water acted as a plasticizer, inducing a decrease in strength. Weakening resulted from erosion and plasticizing effect of water.

The results of this study are agreed with that of Hajmiragha et al [6], Yanikoglu et al [7], Keyf et al [10], Tuna et al [14], Nomoto et al [29], Nomoto et al [30] and Eisenburger et al [31], however some of these researchers use different storing solution and different specimen preparation technique and size.

Hajmiragha et al [6] uses artificial saliva at pH 5 and pH 3 and found that Weight changes of polycarboxylate cement were greatest, and there were significant differences among all the materials ( $P < 0.05$ ). Solubility of the cements in the two medium decreased

in the following order: polycarboxylate, zinc phosphate and glass ionomer. Solubility of the cements were more in the acidic medium ( $P < 0.05$ ).

Yanikoglu et al [7] uses artificial saliva at different pH values and found that statistically significant differences were found among the specimens stored in acidic, basic and neutral artificial saliva, it was observed that the cements were more soluble in acidic media and more stable at pH 7. The highest solubility found in zinc phosphate followed by zinc polycarboxylate and the least is glass ionomer cement.

Keyf et al [10] found that the water sorption of zinc poly carboxylate more than zinc phosphate and the two is more than glass ionomer cement, while for solubility he found that glass ionomer has greater solubility than zinc poly carboxylate and the least is zinc phosphate.

Tuna et al [14] has exactly the same finding of Keyf et al [10].

Nomoto et al [29] found that the erosion (using 0.1 and 0.02 lactic acid solution) of three different kinds of cement; zinc phosphate, polycarboxylate and glass ionomer, were evaluated by measuring the depth loss of the cement in a cavity. Differences in the eroded depths of the three types of cements clearly emerged. The depth losses of polycarboxylate cements (up to 300  $\mu\text{m}$ ) were more than those of zinc phosphate cements (up to 200  $\mu\text{m}$ ), which were more than those of glass ionomer cements (up to 100  $\mu\text{m}$ ) after 24 h immersion in 0.1 M buffer solution.

Nomoto et al [30] found that the eroded depth are in the same order zinc polycarboxylate more than zinc phosphate more than glass ionomer cement and stated that the volumetric

method for investigation are more applicable than gravimetric method.

Eisenburger et al [31] stated that profilometric measurements show a higher susceptibility of zinc phosphate cement than glass ionomer cement for acid erosion. Comparison with erosion depth of enamel and dentine measured in vitro reveals a higher substance loss of zinc phosphate cement at all pH values, whereas glass ionomer cement shows a lower erosion depth than the dental tissues.

### **Conclusion**

Within the limitation of this study, zinc poly carboxylate has greater values of water sorption and solubility than zinc phosphate, and the least values were found in glass ionomer cement.

### **References**

1. Yoruc H.A.B., Karaaslan A.: Effect of water storage on the mechanical properties of zinc poly carboxylate cements. *Digest J. of Nanomaterials and Biostructures* 2007; 2(2): 243-52.
2. Yoshida K, Tanagawa M, Atsuta M.: Solubility of three types of resin and conventional luting cements. *J Oral Rehabil* 1998 Apr; 25(4): 285-91.
3. Cefaly D.F.G., Wang L., deMello L.C., Santos J.L., Santos J.R. and Lauris J.R.P.: Water sorption of resin-modified glass-ionomer cements photoactivated with LED. *Braz oral research* 2006; 20(4):342-6.
4. Anya B.E., Celenk S., Bolgul B.S., Atakul F. and Uysal E.: Water sorption and water solubility of various restorative materials. *Turkiye Klinkleri J Dental Sci*, 2006; 12:43-6.
5. Daugela P., Oziunas R. and Zekonis G.: Antibacterial potential of contemporary dental luting cements. *Stomatologija, Baltic Dental and Maxillofacial Journal*, 2008; 10: 16-21.
6. Hajmiragha H., Nokor S., Alikhasi M., and Nikzad S., Dorriz H.: Solubility of three luting cements in dynamic artificial saliva. *J. of Dentistry, Tehran* 2008; 5(3): 95-98.
7. Yanikoglu N. and Dymus Y.Z.: Evaluation of the solubility of dental cements in artificial saliva of different pH values. *Den. Mat. J.* 2007; 26(1): 62-67.
8. Diaz-Arnold AM, Vargas MA, Haselton DR.: Current status of luting agents for fixed prosthodontics. *J Prosthet Dent* 1999; 81:135-141.
9. Piwowarczyk A, Lauer HC, Sorensen JA. Microleakage of various cementing agents for full cast crowns. *Dent Mater.* 2005; 21 (5):445-53.
10. Keyf F., Tuna S.H., Sen M., Safrany A.: Water sorption and solubility of different luting and restorative dental cement. *Turk J Med Sci*; 2006; 36(1): 47-55.
11. Cattani-lorente MA, Dupuis V, Payan J, Moya F, and Meyer JM: Effect of water on the physical properties resin modified glass ionomer cements. *Dent Mat* 1999; 15 Jan.: 71-79.
12. Musanje L, Shu M, Darvell BW. : Water sorption and mechanical behavior of cosmetic direct restorative material in artificial saliva. *Dent Mat* 2001; 17(5): 394-401.
13. Malacrane J, Carvalho RM, De Goes MF, Svirzero N, Pashley DH, Tay F. et al : Water sorption/solubility of dental adhesive resins. *Dent Mat* 2006; 22(10): 973-980.
14. Tuna S.H., Keyf F.: Water sorption and solubility of provisional and permanent luting cement. *Hacettepe Dishekimligi Fakultesi Dergisi* 2006; 30(3): 19-24.
15. Berglund A, Hulterström AK, Gruffman E, van Dijken JW. Dimensional change of a calcium aluminate cement for posterior restorations in aqueous and dry media. *Dent Mater* 2006; 22: 470-476.
16. Francisconi L.F., Freitas A.P., Scaffa P.M.C., Mondelli R.F.,

- Francisconi P.A.S.: Water sorption and solubility of different calcium hydroxide cements. *J Appl Oral Sci.* 2009; 17(5): 427-31.
17. Gerdolle D.A., Mortier E., Jacqout B. and Panighi M.: Water sorption and water solubility of current luting cements 9an in vitro study). *Quintessence int.* 2008; 39:256.e107-114.
18. Rosenstiel S.F, Martin F. Land and Bruce J. Crispin: Dental luting agents: A review of the current literature. *J Prosth Den* 1998; 80:280-301.
19. Tae Hyung Kim, Sajid A. Jivraj and Terry E. Donovan: Selection of Luting Agents: Part 2. *Californian dental association journal* 2006; 34(2): 161-66.
20. Chai J, Takahashi Y, Hisama K, Shimizu H: water sorption and dimensional stability of three glass fiber reinforced composites. *Int J Prosthodont* 2004, 17: 195-9.
21. Bellenger V, Verdu J, and Morel E: Structure properties relationships for densely cross linked epoxy-amine system based on epoxide or amine mixtures. *J Mater Sci* 1989; 24: 63-68.
22. Von-Fraunhofer JA, Marshall KR, Holman BG. The effect of base/ liner use on restoration leakage. *Gen Dent.* 2006; 54(2):106-9, cited by Francisconi etal no. 16.
23. Sajid A. Jivraj, Tae Hyung Kim and Terry E. Donovan: Selection of Luting Agents: Part 1. *Californian dental association journal* 2006; 34(2): 149-60.
24. ANSI/ADA Specification No. 8 – 1977.
25. D Xie, M Faddah, J G Park: Novel amino acid modified zinc polycarboxylates for improved dental cements *Dent. Mater.* 2005. 21 (8): 739-748.
26. Ymazaki A., Yasushi H., M. Honda, Y. Nagasawa, Y. Hasegawa, J. Omatsu, T. Yamaga and H. Nakajima: Effect of Water on Shear Strength of Glass Ionomer Cements for Luting. *Dental Materials Journal*, 2007; 2 (6 5) : 708—712.
27. Deniz G., Binnaz Y., Mutlu O. and Hasan N. Alkumru: Effect of early water contact on solubility of glass ionomer luting cements. *J Prosthet Dent* 1998; 80:474-8.
28. Carvalho J.R., Guemaraes L.F., Correr L., Pecora J.D. and Sousa-Neto M.D.: Evaluation of Solubility, Disintegration, and Dimensional Alterations of a Glass Ionomer Root Canal Sealer. *Braz Dent J* (2003) 14(2): 114-118.
29. Nomoto R. and McCabe J.F.: A simple acid erosion test for dental water-based cements. *Dental Materials* (2001); 17: 53-59.
30. Nomoto R., Uchida K., Momi Y. and McCabe J.F.: Erosion of water based evaluated by volumetric and gravimetric methods. *Dent Mater.* 2003; 19: 240-244.
31. Eisenburger M., Addy M., RoBbach A.: Acid solubility of luting cements. *Journal of Dentistry* (2003) 31: 137–142.