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# Water Sorption and Solubility of Different Luting and Restorative Dental Cements

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**Aim:** The purpose of this study was to compare the water sorption and solubility of four provisional, three permanent luting cements and five restorative cements.

**Methods:** A split ring mould was fabricated for the preparation of specimen discs which were 15.0 mm in diameter and 1.5 mm thick. All specimens were manipulated according to the manufacturer's instructions and then subjected to water sorption and solubility tests. Data were analysed with Wilcoxon Signed Ranks Test. Scanning electron microscopy was used to evaluate surface topography and roughness.

**Results:** All tested materials demonstrated different degrees of sorption and solubility. The difference between sorption and solubility was statistically significant. Some materials were retained water in their structure and thus were compensated the loss of mass due to dissolution. It was found that zinc phosphate and zinc polycarboxylate cements were the most stable materials for solubility and sorption.

**Conclusion:** The most important properties of cements are their solubility and resistance to disintegration in saliva. This is an important concern for clinicians. If the cement dissolves or deteriorates under a restoration, leakage can result in sensitivity and caries in clinic and patients suffer from toothache.

Key Words: Water sorption, solubility, provisional cement, permenant luting cement, restorative cement

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## Farklı Yapıştırma ve Restoratif Dental Simanların Su Emilimi ve Çözünürlüğü

**Amaç:** Bu çalışmanın amacı dört geçiçi, üç daimi yapıştırma simanının ve beş restoratif simanın su emilim ve çözünürlüğünü karşılaştırmaktı.

**Yöntemler:** 15 mm çapında ve 1.5 mm kalınlığında örnek disklerin hazırlanması için halka şeklinde bir kalıp yapıldı. Siman örnekler üreticilerin önerilerine uygun olarak hazırlandı. Bu örneklere su emilimi ve çözünürlük testi uygulandı. Veriler Wilcoxon Singed Rang testi ile incelendi. Yüzey topografyası ve pürüzlülüğünü değerlendirmek için Taramalı (Scanning) Elektron Mikroskobu kullanıldı.

**Bulgular:** Test edilen bütün materyaller değişik derecelerde emilim ve çözünürlük gösterdi. Emilim ve çözünürlük arasındaki fark istatistiksel olarak önemliydi. Bazı materyaller yapılarında su tuttu ve böylece çözünmeden dolayı oluşan kütle kaybını kompanse ettiler. Çinkofosfat ve çinkopolikarboksilat simanlar çözünürlük ve emilim yönünden daha stabil materyaller olarak bulundu.

**Sonuç:** Simanların en önemli özellikleri çözünürlük ve tükürük içindeki bozulmaya dirençleridir. Klinisyenler için bu önemli bir kaygıdır. Bir restorasyon altındaki siman çözünür ya da bozulursa sızıntı nedeni ile klinik olarak hassasiyet ve çürük oluşabilir. Hastalar diş ağrısından şikayet ederler.

Anahtar Sözcükler: Su emilimi, Çözünürlük, Geçici siman, Daimi yapıştırma simanı, Restoratif siman

## Introduction

The solubility and water sorption charecteristics of restorative materials directly affects their selection criteria. Materials designed for the same clinical purpose differ in their behavior with respect to long-time aging in water.

The solubility of luting and restorative cements influences both their rate of degradation and their biocompatibility due to the nature of the eluates. Water sorption causes dimensional changes, staining, and break in margin contours. Both solubility and sorption contribute to the loss of marginal integrity, surface properties and aesthetics, resulting in restoration failure (1,2). Previous studies have been shown that the extensive amount of water sorption in the some cements is a cause of concern. This may affect mechanical behavior such the flexural strength, Vichers hardness, mechanical stability (3-5).

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According to adhesive potantial, permanent luting cements can be divided low (zinc phosphate, silicate cements), medium (polycarboxilate cement), high (glass ionomer cements and filled or unfilled resins) luting materials. Provisional luting materails are of two main types: calcium hyroxide and zinc oxide cements (with eugenol or alternative substances) (6).

The objective of the present study was to investigate the water sorption and solubility of provisional, permanent luting cements and restorative cements.

## Materials and Methods

Four provisional, three permanent luting cements and five restorative cements were selected (Table 1). All materials were manipulated according to the manufacturer's instructions. Six specimens were made for each product type. A split ring mould was fabricated for the preparation of disc specimens which were 15.0 mm in diameter and approximately 1.5 mm thick. The mould was first slighty over–filled with cement and then sandwiched between two glass plates to extrude the excess material. The specimens were set according to the recommended setting time between the glass plate. Set specimens were removed from their moulds. Specimens were stored set at room temperature for 30 min, weighted ($M_1$) on precision scale (Sartorius Genius, Germany) and placed in water bath maintained at 37 °C, until a constant mass ($M_2$) was obtained. For this, the measurements of weight were made day by day. The specimens were dried in a vacuum oven at 37 °C to until a constant weight was reached ($M_3$).

The density of the samples was determined by means of Archimedes principle (Buoyancy method). Archimedes principles state that a solid immersed in a liquid loses an amount of weight equal to the weight of the fluid it displaces. For this purpose a Mettler Toledo Density Kit (Item number 00033360, Germany) was used on the Shimadzu Electronic Balance (Type AX200, Readability 0.1 mg, Japan). The glass ve plate of the density kit was filled with n-hexane obtained from Merck.

The weight of the samples were determined directly on the electronic balance ($w_1$) and on the holder of the density kit immersed in n-hexane ($w_2$). The following equations were used for the determination of displaced n-hexane weight,

$$w_3 = w_1 - w_2$$

Table 1. Materials used for water sorption and solubility test.

| No | Material | Content | Manufacturer |
|---|---|---|---|
| 1 | Cavex Temporary Cement | Free eugenol (base and catalyst) | CAVEX, Hol. |
| 2 | Cavex ZOE Cement | Zincoxide-Eugenol | CAVEX, Hol. |
| 3 | Cavex Outline | Impression Paste Eugenol Free | CAVEX, Hol. |
| 4 | Medicem | Glass ionomer | PROMEDICA, Neumünster, Germ. |
| 5 | Dycal | Calcium hydroxite | Caulk Densply Milford, Del.U.S. |
| 6 | Logobond | Glass ionomer lining cement | PD Dental, Altenwalde, Germ. |
| 7 | Ionobond | Glass ionomer (for lining and core build-up) | VOCO, Cuxhaven, Germ. |
| 8 | Logofil-U | Polyalkenoate restorative cement | PD Dental, Altenwalde, Germ. |
| 9 | Adhesor Carbofine | Zinc Polycarboxylate | SPOFA DENTAL a.s. Praha, Cernok. |
| 10 | Medifil Silver | Glass ionomer (for lining and core build-up) | PROMEDICA, Neumünster, Germ. |
| 11 | Gem-Core | Glass ionomer core cement | Dental Composites Ltd, Belvedere, Kent, UK. |
| 12 | Adhesor | Zinc Phosphate | SPOFA DENTAL a.s Praha, Cernok. |

where, $w_3$ is the weight of n-hexane displaced, $w_1$ is the weight of sample and $w_2$ is the weight of sample in n-hexane. The volume of the n-hexane displaced was calculated from the following equation.

$$V_{n\text{-hexane}} = w_3 / d_{n\text{-hexane}}$$

where, $V_{n\text{-hexane}}$ is the volume of n-hexane and also the volume of the sample ($V_s$) and dn-hexane is the density of n-hexane.

The density of the sample ($d_s$) was calculated from following equation by using $w_1$ and $V_s$ values.

$$d_s = w_1 / V_s$$

The values for water sorption (Wsp) and solubility (Wsl), in μg/mm$^3$ for each of the specimens were calculated using the following equations[1]:

$$Wsp = M_2 - M_3 / V$$

$$Wsl = M_1 - M_3 / V$$

where $M_1$ is the conditioned mass, in μg, prior to immersion in water; $M_2$ is the mass of the specimen in μg, after immersion in water; $M_3$ is the reconditioned mass of the specimen, in μg after dry and V is the volume of the specimen in mm$^3$.

## Results

Tested materials and manufacturers are listed at Table 1. The mean water sorption and solubility values, standard deviations for Wsp and Wsl are presented at Table 2. The Wilcoxon Test is presented that the difference between Wsp and Wsl is statistically significant (Z=-3,059, p=0.002).

Table 2. Mean values, standard error and standard deviation for Wsp and Wsl.

| | | Wsp | Wsl |
|---|---|---|---|
| N | Valid | 12 | 12 |
| | Missing | 0 | 0 |
| Mean | | 276.61 | -6.20 |
| Std. Error of Mean | | 89.89 | 5.31 |
| Median | | 203.88 | -5.35 |
| Std. Deviation | | 311.40 | 18.40 |
| Range | | 1196.51 | 60.35 |
| Minimum | | 23.79 | -38.59 |
| Maximum | | 1220.30 | 21.76 |
| Percentiles | 25 | 141.54 | -21.19 |
| | 50 | 203.88 | -5.35 |
| | 75 | 237.73 | 4.83 |

Among the cements evaluated, glass ionomer cement (material 4) displayed the lowest water sorption values. There was slight difference between the glass ionomer cements (material 6, 7, 8, 10) except for materials 4 and 11. Three zinc oxide, with eugenol (material 2) and the eugenol free cement (material 1, 3) were different from each other. Zinc oxide free-eugenol materials were lower. Calcium hydroxide (material 5) was shown mean value. Comparison among different cements pointed out that material glass ionomer core cement (material 11) displayed the highest water sorption value. There was difference in sorption between the zinc polycarboxylate cement (material 9) and other cements (Figure 1) .

Zinc oxide eugenol and free-eugenol cements (material 1, 2, 3) and calcium hydroxide (material 5) and some glass ionomer cements (material 7, 10, 11) had negative values for solubility implying the uptake of water into the cement structure. Glass ionomer cement (material 4) showed a greater solubility. Zinc phosphate cement (material 12) was less soluble than zinc polycarboxylate cement (material 9 ) (Figure 2).

Electromicrographs revealed a change of the surface for all tested cements (Figures 3-14). Scanning electron

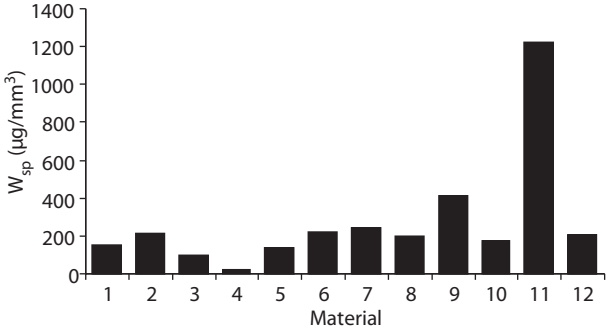


Figure 1. Mean water sorption (μg/mm$^3$) of tested materials.

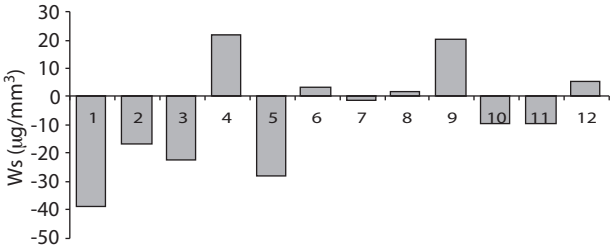


Figure 2. Mean water solubility (μg/mm$^3$) of tested materials.

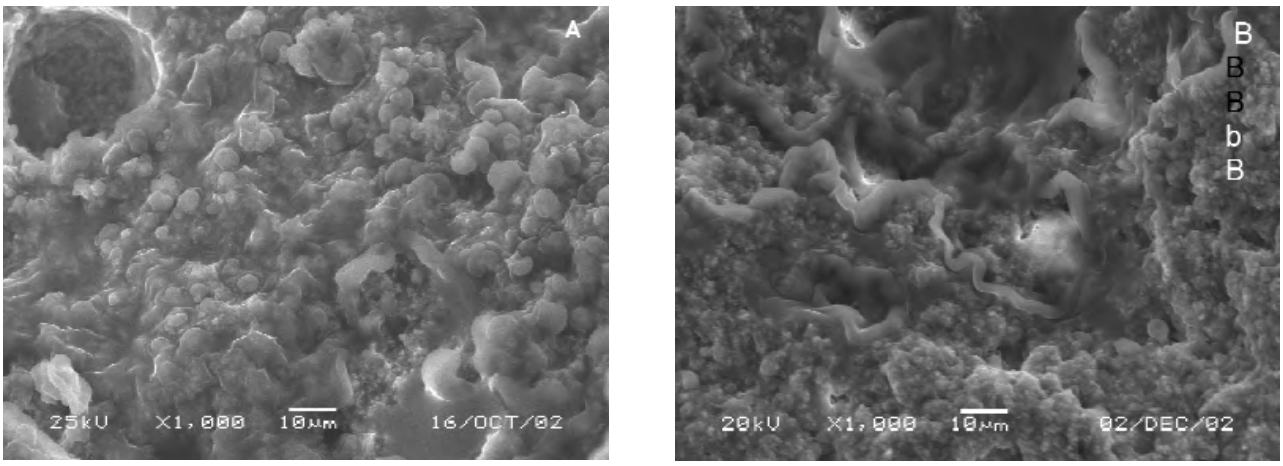


Figure 3. Scanning electron micrograph of zinc oxide free eugenol cement (material 1) surface. A. before water sorption, B. after water sorption.

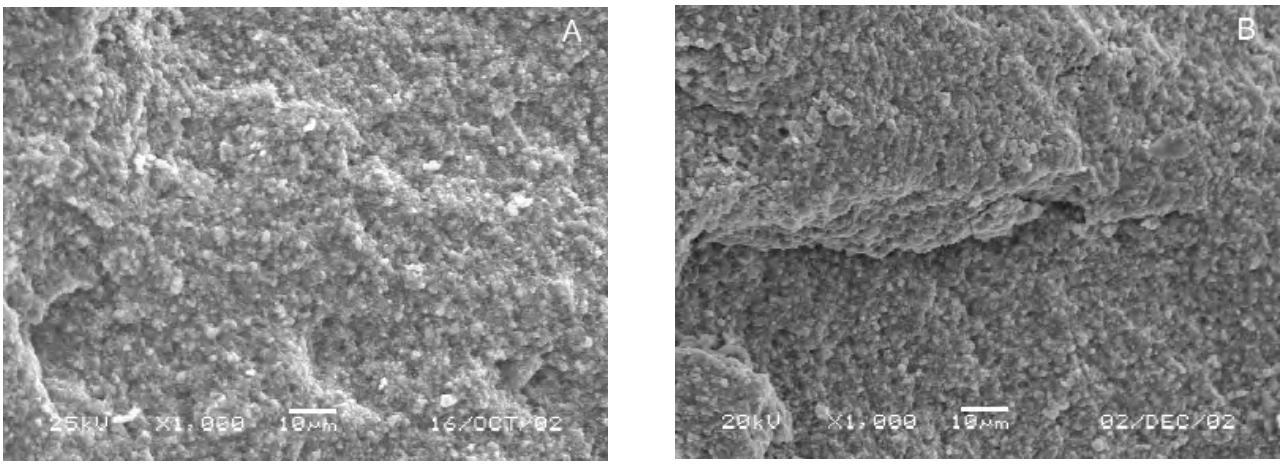


Figure 4. Scanning electron micrograph of zinc oxide eugenol cement (material 2) surface. A. before water sorption, B. after water sorption.

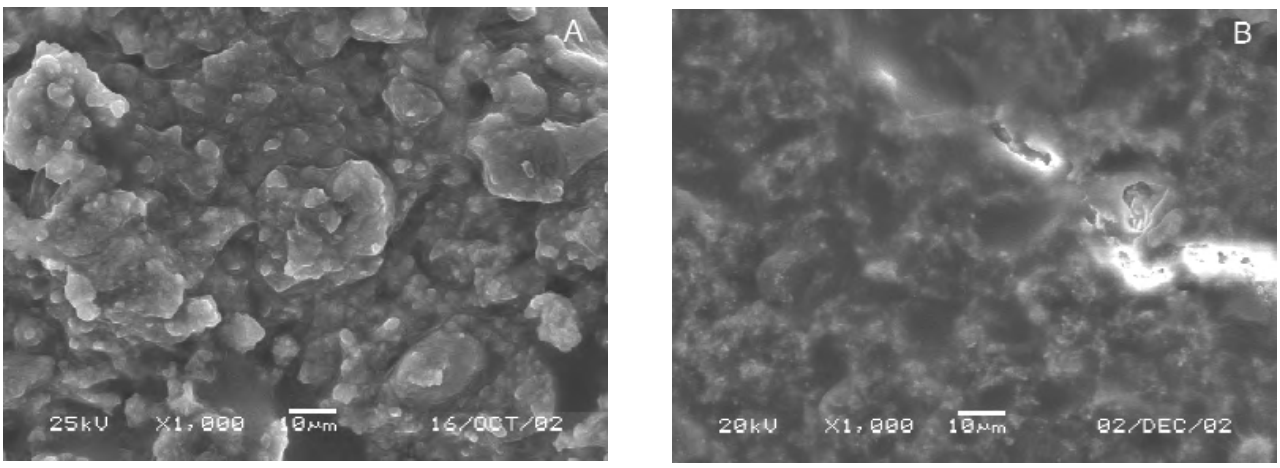


Figure 5. Scanning electron micrograph of zinc oxide free eugenol (impression paste) (material 3) surface. A. before water sorption, B. after water sorption.

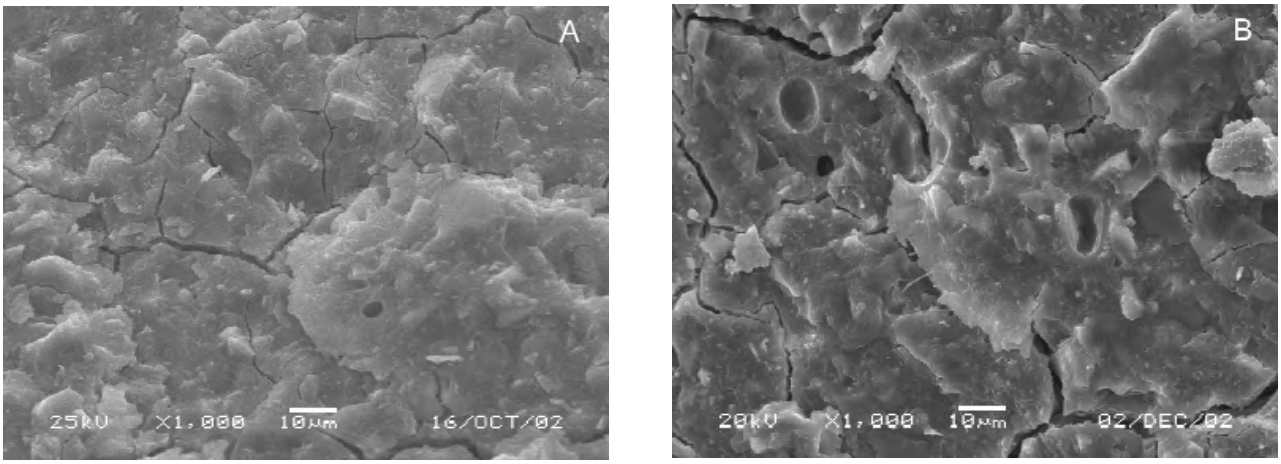


Figure 6. Scanning electron micrograph of glass ionomer cement (material 4) surface. A. before water sorption, B. after water sorption.

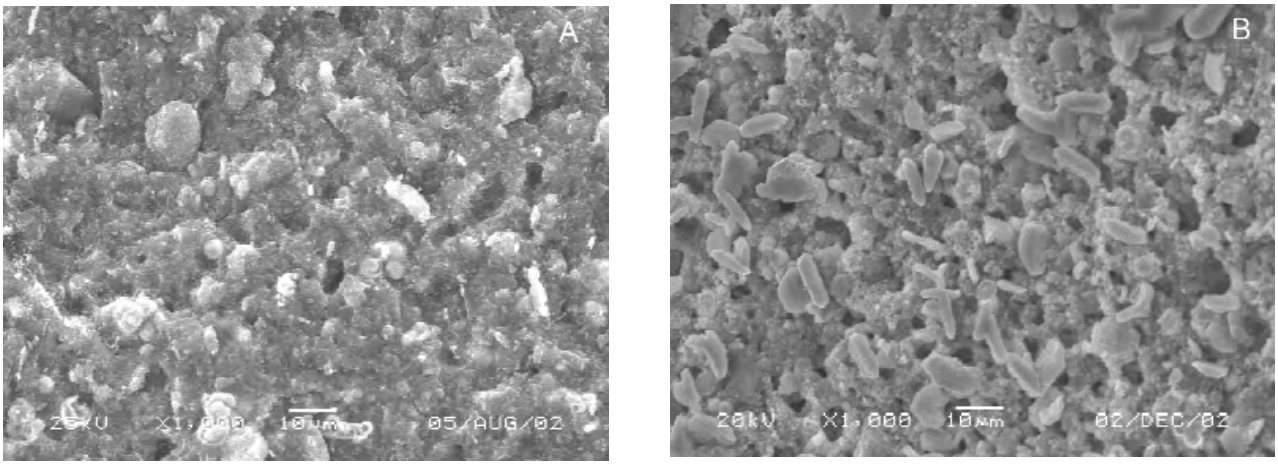


Figure 7. Scanning electron micrograph of calcium hydroxide (material 5) surface. A. before water sorption, B. after water sorption.

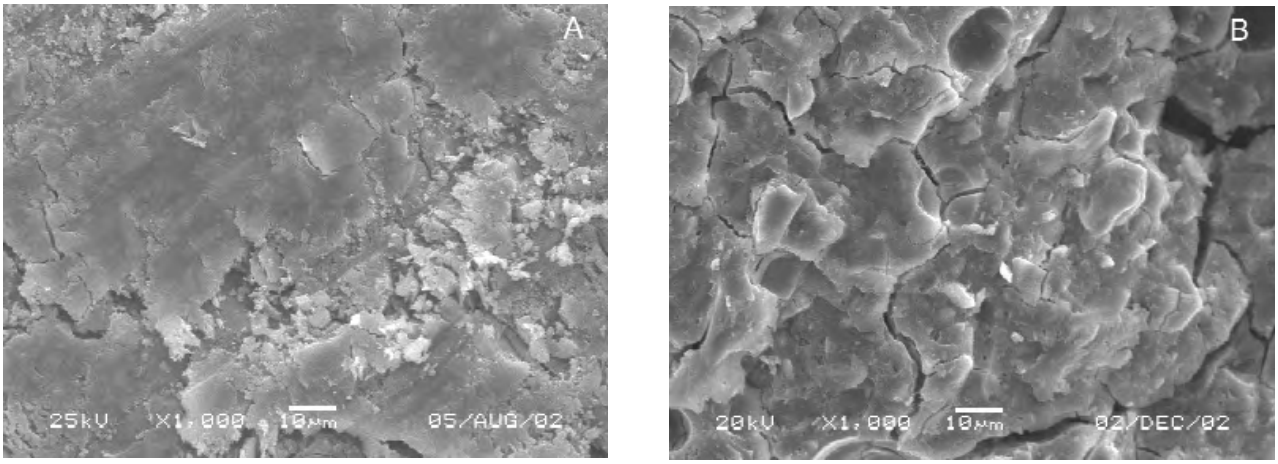


Figure 8. Scanning electron micrograph of glass ionomer lining cement (material 6) surface. A. before water sorption, B. after water sorption.

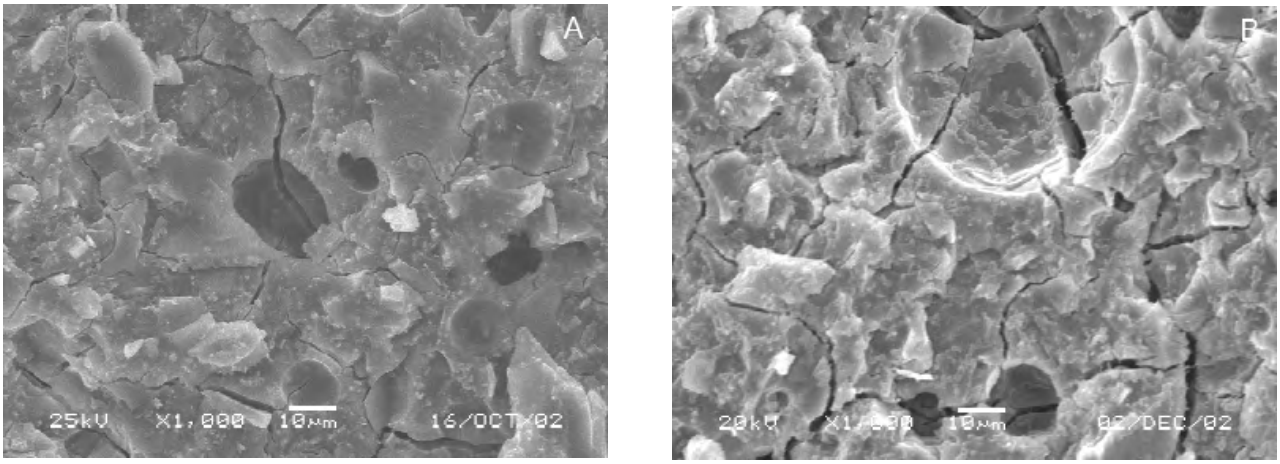


Figure 9. Scanning electron micrograph of glass ionomer cement (for lining and core build-up) (material 7) surface. A. before water sorption, B. after water sorption.

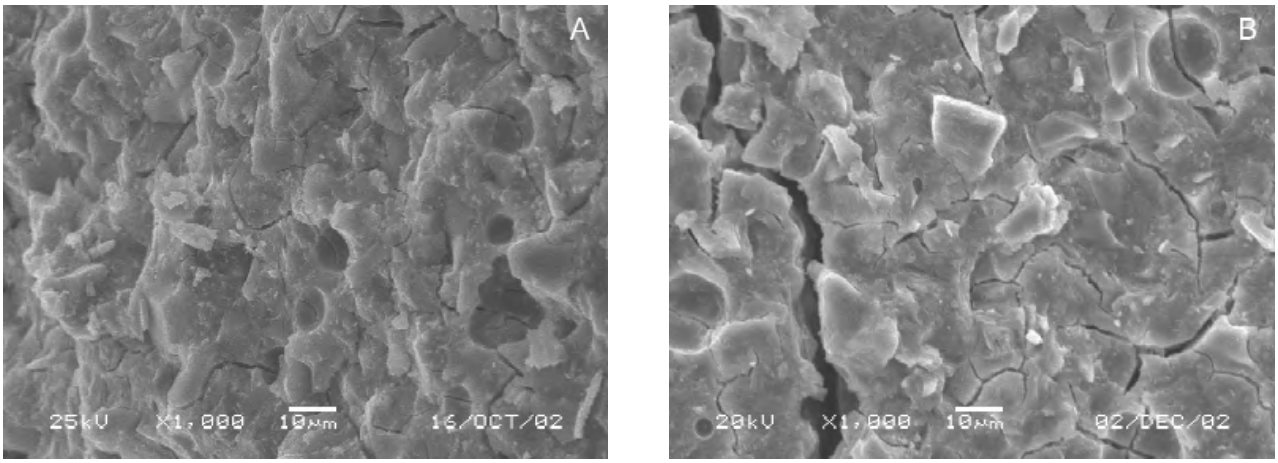


Figure 10. Scanning electron micrograph of polyalkenoate restorative cement (material 8) surface. A. before water sorption, B. after water sorption.

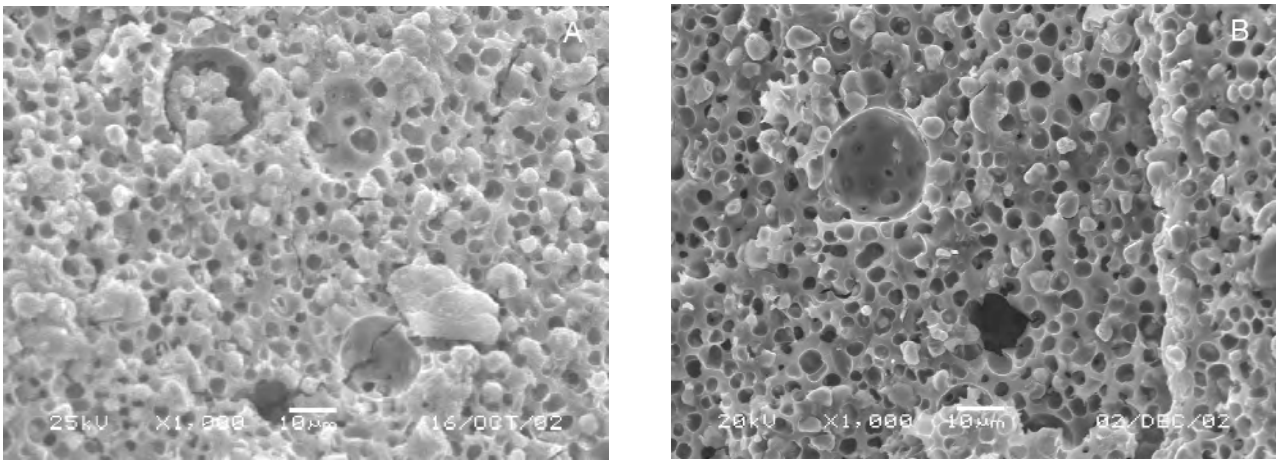


Figure 11. Scanning electron micrograph of zinc polycarboxylate cement (material 9) surface. A. before water sorption, B. after water sorption.

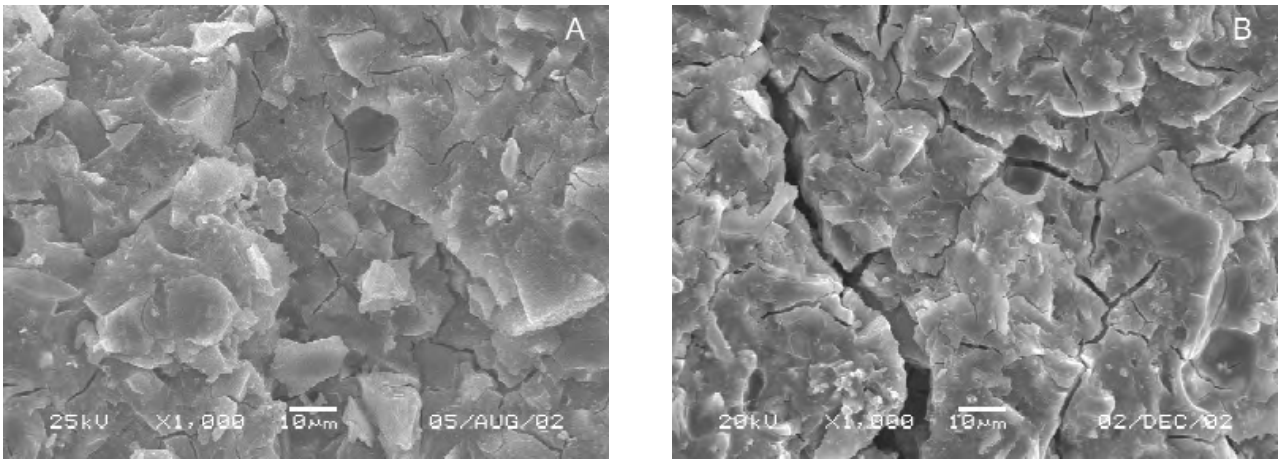


Figure 12. Scanning electron micrograph of glass ionomer cement (for lining and core build-up) (material 10) surface. A. before water sorption, B. after water sorption.

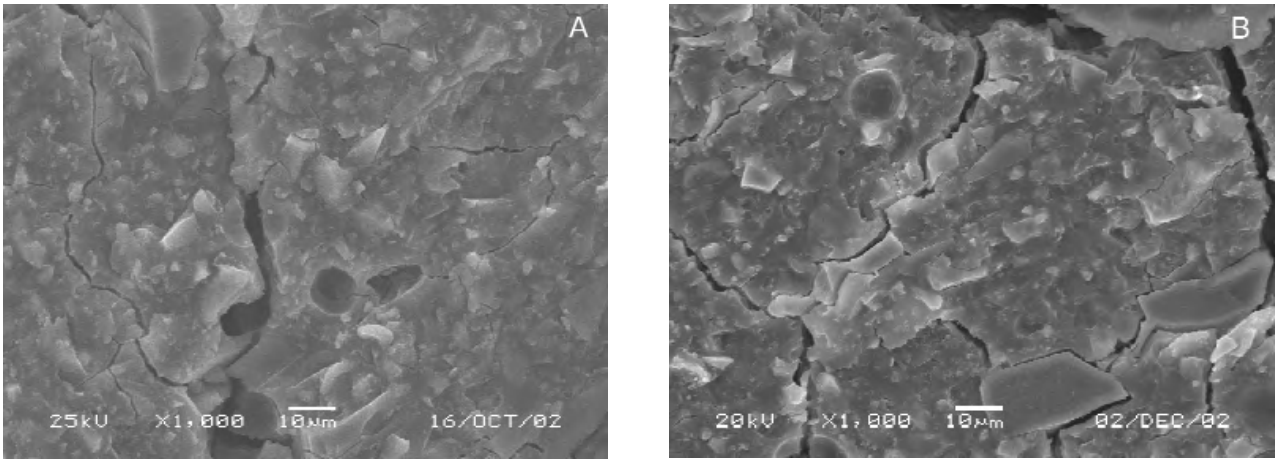


Figure 13. Scanning electron micrograph of glass ionomer cement (core build-up) (material 11) surface. A. before water sorption, B. after water sorption.

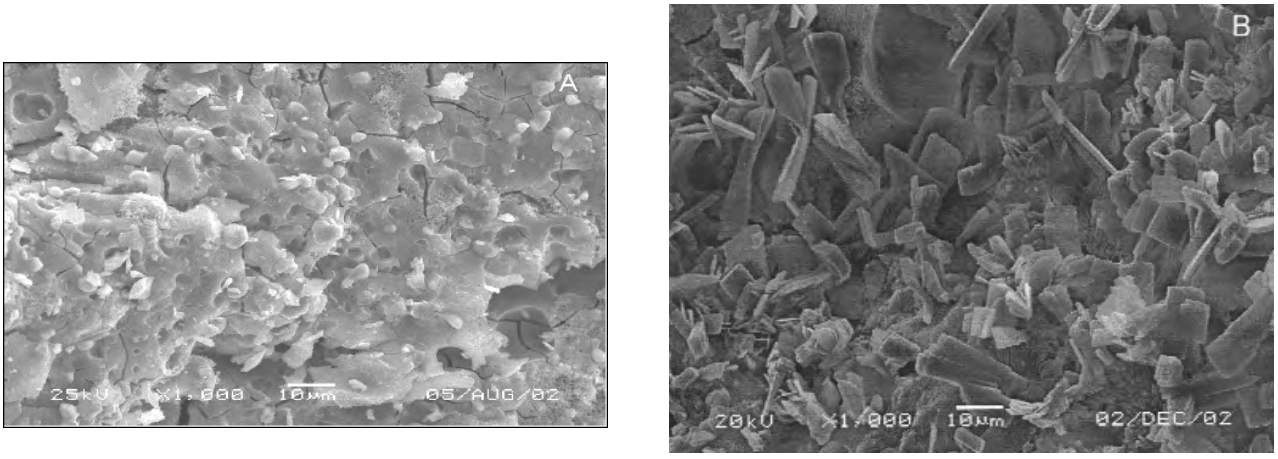


Figure 14. Scanning electron micrograph of zinc phosphate cement (material 12) surface. A. before water sorption, B. after water sorption.

micrographs of cements (x1000) showed more porous surfaces after water-treated.

## Discussion

The water sorption and solubility of dental restorative materials are of considerable clinical importance and cannot be neglegted.

Some studies perfomed to test the behaviour of dental cements have used water, acids and other solvents to simulate the contaminating environment of mouth (1,2,7,8). The chemical structure of the solutions used for in vitro tests is important because it has to simulate the complexity of the oral environment. The in vitro tests made are only static solubility tests because they do not simulate the pH and temperature changes of the oral cavity (1,2,7-12). Clinical conditions vary, even within the same person, making it virtually impossible to reproduce a natural environment (13).

It was reported in previous studies that long–time storage in water affected the mechanical properties of the cements (9-12). Cattani-Lorente et al. (5) 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 decreas in strength. Weakening resulted to erosion and plasticizing effect of water.

Studies about water sorption and solubility were less than did on mechanical properties of cements. Provisional, permanent and restorative cements were not studied together up to now. In this in vitro study, sorption and solubility of cements were measured. Zinc phosphate, zinc polycarboxylate, glass ionomer, calcium hidroxide, zinc oxide eugenol and free eugenol cements are used for luting purposes. Due to the limited strength of zinc oxide eugenol and free eugenol cements, they are only accepted for provisional cementation. However, due to implants do not decay, zinc oxide cements may often be used as the definitive cement and permits an easier retrieval of prosthesis, should intermediate or long-term complications result (14).

One of luting agents evaluated in this study contained eugenol, two provisional cements did not contain eugenol. There was not significant difference between the eugenol-containing and eugenol-free luting agents in regard of sorption. Materials designed for the same clinical purpose may differ in their behavior for solubility. The glass ionomer (material 4) and zinc polycarboxylate cements (material 9) showed more solubility than the other cements. Among permanent cements evaluated, glass ionomer cement (material 4) exhibited the least sorption than did zinc phosphate (material 12) and zinc polycarboxylate cement (material 9). A statistical comparison between Wsl and Wsp showed that they were statistically different.

Many commercial glass ionomer cement products are now used in restorative and crown / core build–up applications. In restorative applications, these materials are constantly exposed to oral fluids at physiological temperature. Except for glass ionomer cement (material 11) had medium sorption values.

Glass ionomer cements are sensitive to water erosion. It may probably be due to same hydrolysis of the cement components. This phenomenon is apparently aggravated in oral environment due to the presence of aggressive compounds in the saliva (7). Clinical sucsess with glass ionomer cements depens on early protection from both hydration and dehydration. It is weakened by early exposure to moisture, while desiccation, on the other hand, produces shringage cracs in the resently set cement (15).

In this study, five glass ionomer cements for restorative filling or core build-up applications were studied. Both materials 10 and 11 present the similar solubility properties.

Some studies conclude that glass ionomer cements are more resistant to degradation than zinc phosphate cements, although Knibbs and Walls (16) reported that marginal defects around crowns appeared sooner with glass ionomer than with zinc phosphate, possibly because of the greater susceptibility of glass ionomer to contamination by moisture. Contaminated glass ionomer is more susceptible to erosion (16,17), and glass ionomer aged in water is mechanically weaker (18).

An in vivo study with patients wearing luting specimens in the lingual flanges of inferior complete dentures showed that polycarboxylate and zinc phosphate cement dissolved more than a glass ionomer cement. Under scanning electron microscopy, glass ionomer and polycarboxylate cements showed pits and extensive cracks on their surfaces, while zinc phosphate showed a large number of pits (19).

Plaque accumulation in dental cements is mainly related to the possibility of achieving and maintaining a highly polished surface of exposed, highly porous lutting material (20).

In our study, scanning electron micrographs of zinc oxide free-eugenol cement surface (material 1, 3) are revealed that the surfaces were much more rough after water sorption (Figures 3, 5). Figure 4 shows cracks on zinc oxide eugenol cement surface (material 2) after water sorption.

Figures 6, 8, 9, 10, 12, 13 show microcraks and many micropores on the glass ionomer cement surfaces after water sorption.

Calcium hyroxide cements have low mechanical properties compared with cements used as high-strengh bases, but they are stronger than zinc oxide-eugenol. Solubility in water and in acid varies considerably among producs (21)

Scanning electron micrograph of calcium hydroxide cement surface (material 5) display many micropores, large and shallow cavities (Figure 7).

Scanning electron micrograph of water stored zinc polycarboxylate cement shows more apparent pores (Figure 11).

After water sorption zinc phosphate cement showed apparent microcracks and a roughest surface (Figure 14).

## Conclusion

Selection of the most suitable restorative material is important, for the longevity of the restoration. Comparisons among different restorative cements indicated that the water sorption and solubility of these materials were different. It was found that zinc phosphate and zinc polycarboxylate cements were the most stable materials for solubility and sorption.

## References


1. Yap A, Lee CM. Water sorption and solubility of resin-modified polyalkenoate cements. J Oral Rehabil 1997; 24: 310-314.
2. Mojon P, Kaltio R, Feduik D, Hawbolt EB, McEntee MI. Short-term contamination of luting cements by water and saliva. Dent Mater 1996; 12: 83-87.
3. Cattani-Lorente MA, Dupuis V, Payan J, Moya F, Meyer JM. Effect of water on the physical properties of resin-modified glass ionomer cements. Dent Mater 1999; 15: 71- 79.
4. Musanje L, Shu M, Darvell BW. Water sorption and mechanical behaviour of cosmetic direct restorative materials in artificial saliva. Dent Mater 2001; 17: 394-401.
5. Malacarne J, Carvalho RM, De Goes MF, Svirzero N, Pashley DH, Tay F et al. Water sorption/solubility of dental adhesive resins. Dent Mater 2006; 22: 973-980.
6. Macorra JC, Praides G. Conventional and adhesive luting cements. Clin Oral Invest 2002; 6: 198-204.
7. Fukasawa M, Matsuya S, Yamane M. The mechanism for erosion of glass-ionomer cements in organic-acid buffer solutions. J Dent Res 1990; 69: 1175-1179.
8. Murakomi H, Matsuya Y, Matsuya S. Dissolution mechanism of zinc phosphate dental cement in acetic and lactic acid buffers. Biomaterials 1990; 11: 435-440.
9. Cattani-Lorente MA, Godin C, Meyer JM. Mechanical behavior of glass ionomer cements affected by long–term storage in water. Dent Mater 1994; 10: 37-44.
10. Mesu FP. Degradation of luting cements measured in vitro. J Dent Res 1982; 61: 665-672.
11. Mitra SB, Kedrowski BL. Long-term mechanical properties of glass ionomers. Dent Mater 1994; 10: 78-82.
12. Uno S, Finger WJ, Fritz U. Long-term mechanical characteristics of resin-modified glass ionomer restorative materials. Dental Mater 1996; 12: 64-69.
13. Macorra JC, Praides G. Conventional and adhesive luting cements. Clin Oral Invest 2002; 6: 198-204.
14. Misch CE. Contemporary Implant Dentistry. Mosby Co. Second ed. St Louis, Missorui 1999.
15. McLean JW Clinical applications of glass ionomer cements. Oper Dent 1992; 17: 184-190.
16. Knibbs PJ, Walls AWG. A laboratory and clinical evaluation of three dental luting cements. J Oral Rehabil 1989; 16: 467-473.
17. Earl MSA, Ibbetson RJ. The clinical disintegration of a glass-ionomer cement. British Dent J 1986; 161: 287-291.
18. Negm MN, Beech DR, Grant AA. An evaluation of mechanical and adhesive properties of polycarboxylate and glass ionomer cements. J Oral Rehabil 1982; 9: 161-167.
19. Hersek N, Canay S. Invivo solubility of three types of luting cement. Quint Int 1996; 27: 211-216.
20. Macorra JC, Praides G. Conventional and adhesive luting cements. Clin Oral Invest 2002; 6: 198-204.
21. Craig RG, Powers JM, Wataha JC. Dental materials properties and manipulation. Mosby, Inc. Seventh ed. St.Louis, Missouri, 2000.