

# The effect of different organic solvents on the degradation of restorative materials

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## ABSTRACT

**Objective:** To evaluate the solubility of three restorative materials exposed to the different endodontic solvents. **Materials and Methods:** The organic solvents eucalyptus oil, xylol, chloroform, and orange oil, with distilled water as the control group was utilized. The restorative materials light-cured resin (Filtek Z250/3M ESPE), light-cured-resin-reinforced glass ionomer (Riva Light Cure LC/Southern Dental Industries SDI) and resin-modified glass ionomer (Vitremer/3M ESPE) were analyzed. A total of 50 disks containing specimens (2 mm × 8 mm Ø) were prepared for each of the three classes of restorative materials, which were divided into 10 groups ( $n = 5$ ) for immersion in eucalyptus oil, xylol, chloroform, orange oil or distilled water for periods of either 2 min or 10 min. The means of restorative material disintegration in solvents were obtained by the difference between the original preimmersion weight and the postimmersion weight in a digital analytical scale. Data were statistically analyzed by two-way analysis of variance while the difference between the materials was analyzed by Student-Newman-Keuls test. The significance level set at 0.05. **Results:** Vitremer showed the highest solubility, followed by Riva LC, and these were statistically different from eucalyptus oil, xylol, chloroform, and distilled water ( $P < 0.05$ ). Regarding the immersion time in solvents, there were no significant differences between the two tested periods ( $P > 0.05$ ). **Conclusions:** The solvents minimally degraded the composite resin, although they did influence the degradation of both resin-modified glass ionomer resin and resin reinforced with glass ionomer.

**Key words:** Composite resin, modified glass ionomer, organic solvents, solubility

## INTRODUCTION

Despite their highly successful, some endodontic treatments do not respond to initial therapy for different reasons, which necessitates a new intervention. The removal of endodontic filling material from the root canal via endodontic solvents is a requirement for retreatment.<sup>[1-6]</sup>

Various methods for the removal of endodontic fillings from the root canals have been proposed, such as the use of hand tools, both with and without solvents, as well as the utilization of heated instruments and mechanical

and ultrasonic equipment.<sup>[1-3,7,8]</sup> Some previous studies have reported that chloroform is more effective than other agents on dissolving most of the endodontic filling materials,<sup>[5,9-13]</sup> although, no repercussions regarding its action or that of other solvents used in restorative materials has been demonstrated.

The mechanical properties of these restorative materials are vastly influenced not only by the chemical composition but also by the environment to which they are exposed.<sup>[14]</sup> The dissolution or elution of leachable components of the restorative materials, mainly inorganic ions or filler particles, may present

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a deleterious effect on the polymeric network of the material, leading to chemical or physical modifications of its structure.<sup>[15-19]</sup>

In this context, the selection of an ideal solvent during the endodontic retreatment requires the establishment of a balance between clinical safety with a lower toxicity and aggressiveness to tissues and effectiveness in chemical dissolving and the possibility of any surface degradation on the present restorative materials.<sup>[20,21]</sup> Based on the need to use the organic solvents in endodontic retreatment in restored teeth and given the generalized lack of knowledge about their effects on restorative materials, the aim of this study was to compare the solubility of restorative materials exposed to various organic solvents.

## MATERIALS AND METHODS

A nanohybrid composite resin (Filtek Z250, 3M ESPE, St. Paul, MN USA) shade A2 that contains bisphenol A glycidyl methacrylate (BisGMA) and ethoxylated bisphenol A glycol dimethacrylate (BisEMA) resins; a light-cured-resin-reinforced glass ionomer (Riva Light Cure [LC], SDI Ltd., Victoria, Australia) shade A3 that utilizes a radiopaque reactive glass filler; and a tri-cure glass ionomer (Vitremmer, 3M ESPE, St. Paul, MN USA) shade A2, which was composed of a radiopaque fluoroaluminosilicate glass, microencapsulated potassium persulfate and an ascorbic powder associated with an aqueous solution of a polycarboxylic acid (liquid), were selected for this study.

The selected solvents were eucalyptus oil (SS White, Rio de Janeiro, RJ, Brazil), orange oil (Orangeform, Formula and Açã, São Paulo, SP, Brazil), xylol (Labsynth, Diadema, SP, Brazil), chloroform (Labsynth, Diadema, SP, Brazil), and distilled water (Milli-Q, Millipore Corp., Bedford, USA).

Fifty cylindrical specimens of each material were prepared according to the manufacturer's specifications using a split stainless steel mold 8 mm in diameter and 2 mm in height. The restorative material Filtek Z250 was light-cured for 60 s on their top surfaces through a clear polyester matrix strip using a visible-light-curing unit (Emitter A, Schuster Dental Equipments, Santa Maria, RS, Brazil) with an intensity of 750 mW/cm<sup>2</sup> as determined by a radiometer (Curing Lightmeter 105, DMC Equipments, São Carlos, SP, Brazil). The light-cured glass ionomer Vitremmer and Riva LC were manipulated in accordance with the manufacturers' instructions and carefully introduced into sample molds using a Centrix injector (Centrix Incorporated,

CT, USA) until it was completely filled and excess material was removed with a metallic spatula Goldstein Flexi-Thin (Hu-Friedy, Chicago, IL, USA). Subsequently, the mixed materials were light-cured individually in accordance with the manufacturer's recommendations.

Any excess material was then trimmed to the surface level of the mold with a scalpel, and the residues were removed with soft brushes and air spray. For each restorative material, 50 samples were prepared, which were then divided into five groups. We further divided each of those groups into two subgroups of five each, according to the immersion period (2 min and 10 min). Thus, we prepared a total of 150 samples for this study.

The samples were immediately transferred to individual containers and were left untouched for 48 h at a constant temperature of 37 ± 1°C (Fanem, São Paulo, SP, Brazil). The samples were weighed in grams (up to four decimal places) on a precision scale (Mark 210A, Bel Engineering, Monza, Italy) prior to immersion in the solvent to obtain the initial mass. The weights were recorded in duplicate. At room temperature (20 ± 1°C), the sealer samples were immersed in 20 ml of solvent stored in an amber glass bottle with a screw cap. The immersion was such that both surfaces of each sample were readily accessible to the solvent. Distilled water, obtained from the Milli-Q water system (Millipore Corp., Bedford, USA), was used as a negative solvent control. After the specified immersion period, the samples were removed from the glass vials, rinsed with 50 ml of double-distilled water, and then blotted dry with absorbent paper. The samples were allowed to dry for 24 h at 37 ± 1°C in an oven and kept in a dehumidifier/desiccator with silica gel. They were later weighed, and the amount of restorative material removed from the specimen was determined as the difference between the original weight of the restorative material and its final weight.

The mean and standard deviation of dissolution (weight loss) in grams were calculated at each time interval for each specimen group. The values were compared by factorial analysis of variance using the SPSS software (SPSS Incorporated, Chicago, USA). When the F-tests were significant, *post-hoc* Student-Newman-Keuls multiple comparison intervals were performed to identify statistically homogeneous subsets ( $P = 0.05$ ). Additionally, the surface texture of two randomly selected specimens and two control samples from the three restorative materials were qualitatively evaluated by SEM, using

a digital scanning microscope (Zeiss Digital Scanning Microscope 940A, Carl Zeiss, Oberkochen, Germany).

## RESULTS

The mean solubility data for the restorative materials are listed in the Table 1. Regarding the solubility data for the different restorative materials, there were no statistically significant differences ( $P > 0.05$ ) between composite resin and glass ionomer Riva LC when immersed in the tested solvents for either 2 min or 10 min. For the glass ionomer Vitremer, the orange oil in both evaluation periods provided the lowest mean solubility values compared to other solvents ( $P < 0.05$ ).

Comparisons between different restorative materials showed that Vitremer showed the highest solubility, followed by Riva LC glass ionomer, which was statistically different from eucalyptus oil, xylol, chloroform, and distilled water ( $P < 0.05$ ). Composite resin presented the lowest solubility ( $P < 0.05$ ).

Regarding the immersion time in the solvents, there were no significant differences between the two tested modes ( $P > 0.05$ ).

The SEM examinations of the selected specimens kept in a solvent environment showed few surface alterations. The most evident was the presence of voids and porosities in some areas, though there was no apparent loss of fillers or topography alterations after the aging time [Figures 1-3].

## DISCUSSION

Considering the great chance of success in endodontic reinterventions, retreatment becomes a conservative clinical procedure in comparison to more radical procedures such as periapical surgeries.<sup>[22]</sup> Although, there are only a few reports in the literature

investigating the solubility of restorative materials immersed in organic solvents, it was interesting to observe the comparison of the effects on some groups of modified glass-ionomer restorative materials and composite resins.

Similar values were detected in the solubility data for all tested solvents and periods, with the exception of orange oil in the glass ionomer Vitremer. These results are interesting because the control group (distilled water) showed solvency power equal to or even greater than that of the other tested solutions. This may indicate that Vitremer was more susceptible to water absorption, leading to mass gain that may have masked its real solubility. This does not mean "solubility" did not occur, though its water absorption was greater than its solubility. This characteristic of high hydrophilicity of the composite materials has been observed in other studies.<sup>[23,24]</sup>

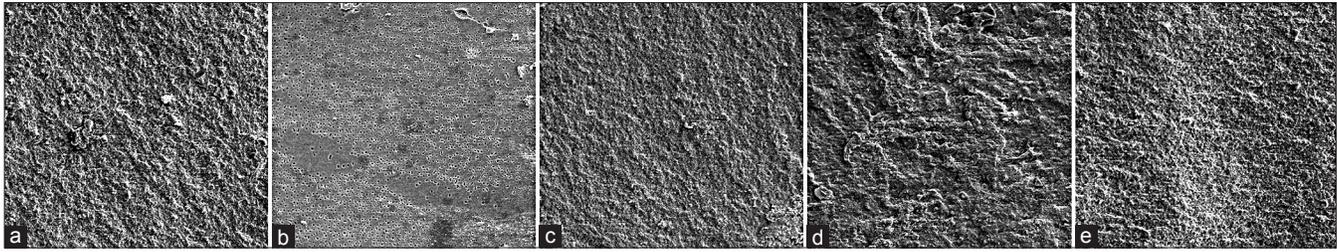
Comparing the materials with each other we found that the composite resin and resin-modified glass-ionomer material (Vitremer/3M ESPE and Riva LC/SDI) showed statistically significant differences regarding its solvency between them ( $P < 0.05$ ). It is possible that the chemical characteristics of Filtek Z250 and Riva resulted in lower values of material loss through solvency. The resin blend has a higher molecular weight, which may reduce polymerization shrinkage, as well as any aging effects. Filtek Z250 is also purported to be quite hydrophobic and therefore, less sensitive to atmospheric moisture.

The clinical performance of BisGMA-based materials is to a great extent, dependent on their mechanical properties and resistance to chemical degradation by acids and other organic substances found in the oral cavity.<sup>[25]</sup> The results of this investigation showed that all three materials stored in different solvents suffered minimal disintegration that was not statistically significant ( $P > 0.05$ ) when comparing

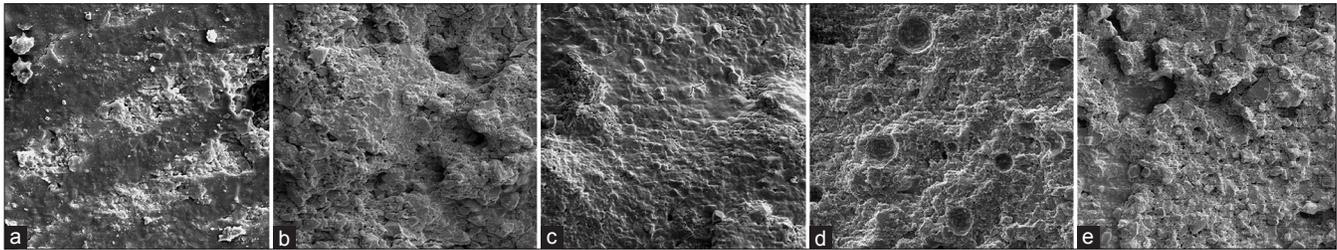
**Table 1: Means with SD ( $\pm$ ) of weight loss (grams) for each restorative material with the different tested solvents and contact times**

Solvents	Orange oil		Eucalyptus oil		Xylol		Chloroform		Distilled water	
	2 min	10 min	2 min	10 min	2 min	10 min	2 min	10 min	2 min	10 min
Filtek	0.0007 <sup>A,a</sup>	0.0011 <sup>A,a</sup>	0.0015 <sup>A,a</sup>	0.0017 <sup>A,a</sup>	0.0014 <sup>A,a</sup>	0.002 <sup>A,a</sup>	0.0015 <sup>A,a</sup>	0.0012 <sup>A,a</sup>	0.0008 <sup>A,a</sup>	0.0009 <sup>A,a</sup>
Z250	( $\pm 0.0004$ )	( $\pm 0.0005$ )	( $\pm 0.0004$ )	( $\pm 0.0007$ )	( $\pm 0.0003$ )	( $\pm 0.0004$ )	( $\pm 0.001$ )	( $\pm 0.0005$ )	( $\pm 0.0007$ )	( $\pm 0.0004$ )
Riva LC	0.0031 <sup>A,b</sup>	0.0041 <sup>A,c</sup>	0.0036 <sup>A,b</sup>	0.0032 <sup>A,b</sup>	0.0034 <sup>A,b</sup>	0.0037 <sup>A,b</sup>	0.0032 <sup>A,b</sup>	0.0032 <sup>A,b</sup>	0.0029 <sup>A,b</sup>	0.0034 <sup>A,b</sup>
	( $\pm 0.0006$ )	( $\pm 0.0006$ )	( $\pm 0.0003$ )	( $\pm 0.0006$ )	( $\pm 0.0008$ )	( $\pm 0.0002$ )	( $\pm 0.0007$ )	( $\pm 0.0007$ )	( $\pm 0.0004$ )	( $\pm 0.0005$ )
Vitremer	0.0038 <sup>A,bc</sup>	0.0038 <sup>A,bc</sup>	0.0049 <sup>B,c</sup>	0.005 <sup>B,c</sup>	0.0061 <sup>B,c</sup>	0.0058 <sup>B,c</sup>	0.0064 <sup>B,c</sup>	0.0056 <sup>B,c</sup>	0.0053 <sup>B,c</sup>	0.0059 <sup>B,c</sup>
	( $\pm 0.0005$ )	( $\pm 0.0004$ )	( $\pm 0.0008$ )	( $\pm 0.0008$ )	( $\pm 0.0001$ )	( $\pm 0.0007$ )	( $\pm 0.001$ )	( $\pm 0.0004$ )	( $\pm 0.001$ )	( $\pm 0.0002$ )

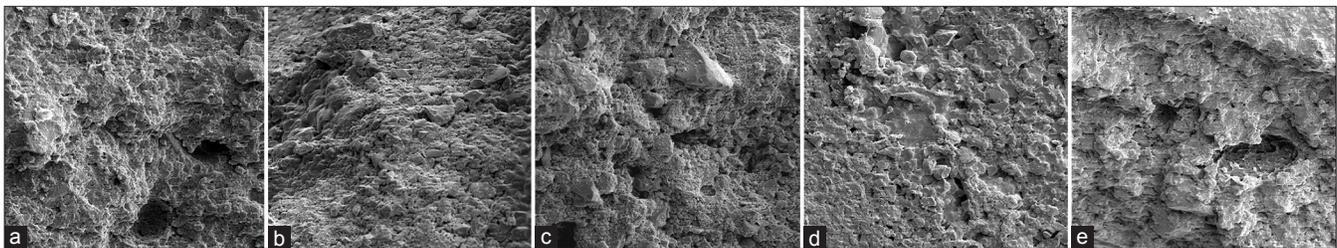
Means followed by the same superscript upper case letter in the rows indicate no statistically significant difference among the solvents for each restorative material ( $P < 0.05$ ). In the columns, same superscript lower case letter indicates no statistically significant difference among the restorative materials for each solvent ( $P < 0.05$ ). LC: Light cure



**Figure 1:** Scanning electron microscopy of filtek after 10 min immersion (sequence from left to right): (a) Control, (b) orange oil, (c) eucalyptus oil, (d) chloroform, and (e) xylol (500 × 10 kv 30 mm)



**Figure 2:** Scanning electron microscopy of vitremer after 10 min immersion (sequence from left to right): (a) Control, (b) orange oil, (c) eucalyptus oil, (d) chloroform, and (e) xylol (500 × 10 kv 30 mm)



**Figure 3:** Scanning electron microscopy of riva light cure after 10 min immersion (sequence from left to right): (a) Control, (b) orange oil, (c) eucalyptus oil, (d) chloroform, and (e) xylol (500 × 10 kv 30 mm)

the difference among the solvents for each restorative material. The mechanism of hydrolytic degradation is enhanced, if the filler particles have metallic ions in their composition.<sup>[26]</sup> An explanation of this effect is that some ions in the filler particles, such as barium and zinc, are electropositive and tend to react with the aqueous solution. With the loss of these elements into the solution, the charge balance inside the silica network changes and is reestablished with the penetration of hydrogen ions from the aqueous solution into the spaces occupied by zinc and barium. As a result of the increase in the concentration of hydroxyl ions, the siloxane bonds of the silica network start to disintegrate, potentially forming an autocatalytic cycle of surface degradation.<sup>[14,26,27]</sup>

This mechanism may perhaps explain the real solubility of the tested materials, despite being statistically insignificant ( $P > 0.05$ ). With respect to the contact time, the solvents were similarly effective when used for both 2 and 10 min.

In addition, the SEM evaluation of the specimens kept in a solvent environment revealed changes in the surface texture. The solvent-stored samples were significantly rougher than the control specimens and also showed a fine, highly porous structure. This surface roughness appeared to be a discernible loss of material and crack formation. As long as the inorganic fillers of the types currently used clinically are present, the surfaces of composite resins will be rough, either because of loss or projection of particles.<sup>[28]</sup>

Today, the most commonly used solvents have a good capacity for removing the gutta-percha and also have an effect on the filling cements. There are several alternative auxiliary chemical agents for the dissolution of endodontic filling materials. These chemicals are chosen according to two fundamental criteria: Solvent effectiveness and toxicity level.<sup>[20,21]</sup> In addition to the existence of alternative solvents to replace those with high levels of systemic and tissue-related toxicities, it is important to emphasize the possibility of other auxiliary methods, such as

the use of manual endodontic instruments, rotary instruments, and equipment such as ultrasound, to remove the cement.<sup>[1-3,7,8,29]</sup>

Considering the similarities between eucalyptus oil, chloroform, xylol, and orange oil ( $P > 0.05$ ), we could presume an extension of the clinical use of orange and eucalyptus oils because of their low toxicity to tissues.<sup>[12]</sup> The field of action of a solvent must be primarily limited to the proximity of the periapical area to prevent the occurrence of chemical pericementitis. Thus, careful utilization of solvents, as well as the use of short gauge and active endodontic files, is critical in facilitating the chemical-mechanical removal of endodontic cements.

It is important to note that the contact time of these solutions in an already restored tooth should be as brief as possible to maintain safety. Although, longer exposure times showed no significant effect in this study, this does not mean that we should not aim for as short a contact time as possible. Different ways to minimize any contact of these solutions during the retreatment would include the application of some type of insulation like vaseline or dentin adhesive in the restorations prior to the use of a solvent along with the use of abundant and successive irrigations with the sodium hypochlorite.

One study, the limitation was the use of only three commercially available restorative materials. Further studies should use a wider range of permanent restorative materials and explore the effects of longer contact times of the solvents used in endodontics.

## CONCLUSIONS

The results obtained and the analyses conducted in the present study concluded that the tested solvents minimally degraded the composite resin, although they did cause the degradation of resin-modified and resin-reinforced glass ionomers.

## REFERENCES

1. Wilcox LR. Endodontic retreatment with halothane versus chloroform solvent. *J Endod* 1995;21:305-7.
2. Gilbert BO Jr, Rice RT. Re-treatment in endodontics. *Oral Surg Oral Med Oral Pathol* 1987;64:333-8.
3. Mandel E, Friedman S. Endodontic retreatment: A rational approach to root canal instrumentation. *J Endod* 1992;18:565-9.
4. Hansen MG. Relative efficiency of solvents used in endodontics. *J Endod* 1998;24:38-40.
5. Schäfer E, Zandbiglari T. A comparison of the effectiveness of chloroform and eucalyptus oil in dissolving root canal sealers. *Oral Surg Oral Med Oral Pathol Oral Radiol Endod* 2002;93:611-6.
6. Johann J, Martos J, Silveira LF, Del Pino FA. Use of organic solvents in endodontics: A review. *Rev Clin Pesq Odontol* 2006;2:393-9.
7. Ladley RW, Campbell AD, Hicks ML, Li SH. Effectiveness of halothane

- used with ultrasonic or hand instrumentation to remove gutta-percha from the root canal. *J Endod* 1991;17:221-4.
8. Friedman S, Moshonov J, Trope M. Efficacy of removing glass ionomer cement, zinc oxide eugenol, and epoxy resin sealers from retreated root canals. *Oral Surg Oral Med Oral Pathol* 1992;73:609-12.
9. Wennberg A, Orstavik D. Evaluation of alternatives to chloroform in endodontic practice. *Endod Dent Traumatol* 1989;5:234-7.
10. Görduşus MO, Taşman F, Tuncer S, Etikan I. Solubilizing efficiency of different gutta-percha solvents: A comparative study. *J Nihon Univ Sch Dent* 1997;39:133-5.
11. Whitworth JM, Boursin EM. Dissolution of root canal sealer cements in volatile solvents. *Int Endod J* 2000;33:19-24.
12. Martos J, Gastal MT, Sommer L, Lund RG, Del Pino FA, Osinaga PW. Dissolving efficacy of organic solvents on root canal sealers. *Clin Oral Investig* 2006;10:50-4.
13. Magalhães BS, Johann JE, Lund RG, Martos J, Del Pino FA. Dissolving efficacy of some organic solvents on gutta-percha. *Braz Oral Res* 2007;21:303-7.
14. Martos J, Osinaga PW, Oliveira E, Castro LA. Hydrolytic degradation of composite resins: Effects on the microhardness. *Mater Res* 2003;6:599-604.
15. Silveira LF, Ferreto MA, Rosa FV, Martos JM, César Neto JB. Impact of sodium hypochlorite and edta on the quality sealing of composite resin restoration performed in human dentin. *Rev Pós-Grad USP* 2008;15:103-9.
16. Asmussen E, Peutzfeldt A. Influence of selected components on crosslink density in polymer structures. *Eur J Oral Sci* 2001;109:282-5.
17. Söderholm KJ, Yang MC, Garcea I. Filler particle leachability of experimental dental composites. *Eur J Oral Sci* 2000;108:555-60.
18. Söderholm KJ, Mukherjee R, Longmate J. Filler leachability of composites stored in distilled water or artificial saliva. *J Dent Res* 1996;75:1692-9.
19. Ferracane JL. Hygroscopic and hydrolytic effects in dental polymer networks. *Dent Mater* 2006;22:211-22.
20. Ribeiro DA, Matsumoto MA, Marques ME, Salvadori DM. Biocompatibility of gutta-percha solvents using *in vitro* mammalian test-system. *Oral Surg Oral Med Oral Pathol Oral Radiol Endod* 2007;103:e106-9.
21. Erdemir A, Adanir N, Belli S. *In vitro* evaluation of the dissolving effect of solvents on root canal sealers. *J Oral Sci* 2003;45:123-6.
22. Sundqvist G, Figdor D, Persson S, Sjögren U. Microbiologic analysis of teeth with failed endodontic treatment and the outcome of conservative re-treatment. *Oral Surg Oral Med Oral Pathol Oral Radiol Endod* 1998;85:86-93.
23. Malacarne J, Carvalho RM, de Goes MF, Svizero N, Pashley DH, Tay FR, *et al.* Water sorption/solubility of dental adhesive resins. *Dent Mater* 2006;22:973-80.
24. Lopes LG, Franco EB, Pereira JC, Mondelli RF. Effect of light-curing units and activation mode on polymerization shrinkage and shrinkage stress of composite resins. *J Appl Oral Sci* 2008;16:35-42.
25. Witzel MF, Calheiros FC, Gonçalves F, Kawano Y, Braga RR. Influence of photoactivation method on conversion, mechanical properties, degradation in ethanol and contraction stress of resin-based materials. *J Dent* 2005;33:773-9.
26. Söderholm KJ, Zigan M, Ragan M, Fischlschweiger W, Bergman M. Hydrolytic degradation of dental composites. *J Dent Res* 1984;63:1248-54.
27. Lovell MR, Dalgliesh M, Richardson RM, Barnes AC, Enderby JE, Evans B. Depth of water incorporation into float glass surfaces studied by neutron reflection. *Phys Chem Chem Phys* 1999;1:2379-81.
28. Sarrett DC, Ray S. The effect of water on polymer matrix and composite wear. *Dent Mater* 1994;10:5-10.
29. Friedman S, Stabholz A, Tamse A. Endodontic retreatment: Case selection and technique. 3. Retreatment techniques. *J Endod* 1990;16:543-9.

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