ORIGINAL ARTICLE

Comparison of sorption, solubility, and flexural strength of four resin luting cements in three different media: An *in vitro* study

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**Abstract**

**Background:** The clinical success and durability of luting cements are determined by structural integrity and dimensional stability. Sorption and solubility resin luting cements in food-simulating solutions has not been studied sufficiently.

**Aims and Objectives:** To compare the sorption, solubility, and flexural strength of resin luting cements in artificial saliva, ethanol:water (1:1) and distilled water at 37°C.

**Materials and Methods:** Four commercially available resin cements were selected. Disk- and rectangular-shaped samples will be prepared according to ISO standard-4049. Out of 160 specimens, 120 disc-shaped specimens will be subjected to sorption and solubility analysis. 40 rectangular-shaped specimens will be subjected for flexural strength analysis.

**Results:** The result of the study clearly depicted that highest water sorption was observed for GC Fuji Cem in distilled water and artificial saliva media. For Composite Ionomer highest sorption was observed in ethanol:water medium. Panavia F showed least sorption in all three media. GC Fuji Cem exhibited more solubility in artificial saliva and ethanol:water (1:1) media, Composite Ionomer showed more solubility in distilled water medium. Secure luting agent showed least solubility in all three media. Highest flexural strength was observed by Panavia F and least by Tokeyoma Ionomer.

**Conclusion:** Within the limitation of this study resin luting (Panavia F and Secure) agents showed least sorption, solubility, and highest flexural strength than compared to resin reinforced glass ionomer cement (GC Fuji Cem and Tokeyoma Ionomer).

**Introduction**

Various dental treatments require attachment of prostheses to the teeth by means of a luting agent. The word luting implies to seal a space or to cement two components together by use of a moldable substance; hence, the term is descriptive of dental cementing agents. Ceramics in the oral environment are continuously exposed to a variety of acids produced by microorganisms during the breakdown of fermentable carbohydrates. Both the pH and temperature of the oral cavity fluctuate. This complexity of the oral environment, coupled with the fact that different cements behave in different ways, has hindered the development of a standard laboratory test to accurately predict the relative resistance to degradation of various cements *in vivo*. The water sorption by the luting agent may have detrimental effect such as filler/matrix debonding and breakdown of the resin matrix and restoration fracture by hygroscopic expansion, which causes internal stresses and degradation. Some studies found that hygroscopic expansion of a material resulting from the sorption of water as a factor for improved marginal adaptation yet on the other side clinically beneficial consequences in compensating for initial polymerization shrinkage.

Flexural strength is a mechanical parameter for brittle material, is defined as a material’s ability to resist deformation under load. In a tooth-cement-restoration assembly, the cement should have adequate flexural strength to be able to transmit the stresses between the tooth and restoration without breaking. This will prevent breaking of the brittle restorative material. Hence, the elastic modulus of the luting cement should be closer to the dentin, so there will be less stress concentration at the cement tooth interface and will result a better durable bond. Resin cements are approximately 20× stronger and 130×...
tougher in flexure than conventional luting cements, which make them the material of choice in the cementation of all-ceramic restorations.\(^3\)

Therefore, in view of the clinical importance of dissolution of luting cements in the oral environment, an *in vitro* study was designed to compare the sorption, solubility and flexural strength of resin-based luting cements.

**Materials and Methods**

Disk- and rectangular-shaped plastic molds were prepared of dimensions (10 mm diameter and 2 mm depth) and (25 mm × 2 mm × 2 mm) according to ISO standard-4049, respectively, to fabricate specimens required for the study.

All the luting agents manipulated according to manufacturer’s instructions and loaded into plastic mold. To reduce irregularities and voids in the specimens, the mold was slightly overfilled with luting agent, and then it was pressed by microscopic slide under hand pressure to extrude any excess material. The specimens were allowed to set according to the recommended setting time. Light curing gun and composite light curing unit had been used to ensure complete photoactivation and uniform curing of the specimens. After the final set of luting cements [Figures 1 and 2], specimens were removed from mold and grounded on both sides with silicone carbide paper to remove irregularities.

Grouping of samples is listed below:

- **Group 1**: Composite Ionomer (A) - 30 disc-shaped specimens \(\{A1, A2, A3\}\) and 10 rectangular-shaped specimens \(\{A4\}\)
- **Group 2**: GC FUJI CEM (B) - 30 disc-shaped specimens \(\{B1, B2, B3\}\) and 10 rectangular-shaped specimens \(\{B4\}\)
- **Group 3**: Secure (C) - 40 samples (30 disc-shaped specimens \(\{C1, C2, C3\}\) and 10 rectangular-shaped specimens) \(\{C4\}\)
- **Group 4**: Panavia F (D) - 40 samples (30 disc-shaped specimens \(\{D1, D2, D3\}\) and 10 rectangular-shaped specimens) \(\{D4\}\).

- A1, B1, C1, D1 samples kept in distilled water
- A2, B2, C2, D2 samples kept in water: ethanol
- A3, B3, C3, D3 samples kept in artificial saliva
- A4, B4, C4, D4 samples subjected to 3 points bending test.

All specimens were weighed \(m_0\) using an analytical scale with accuracy up to 0.01 mg. Diameter and thickness were measured using a digital caliper with accuracy up to 0.01 mm. The diameter of each specimen was measured at two points at right angles to each other and the mean diameter calculated. The thickness of each specimen was measured at the center of the specimen and at four equally spaced points on the circumference and the mean thickness calculated. The volume \(V\) of each specimen was calculated as follows in cubic millimeters using the mean thickness and diameter: 

\[
V = \pi \times r^2 \times h
\]

Where \(r\) - Mean sample radius (diameter/2) and \(h\) - Mean sample thickness.

For each material, 10 specimens were placed in a glass vial containing 20 ml of distilled water, another 10 specimens placed in 50% ethanol: water solution (pH 5.4) and remaining 10 specimens in artificial saliva. The vials were wrapped in aluminum foil to exclude light and placed in an incubator at 37°C. The dimensions and weights of the specimens were recorded at 1 day, 3 day and then at 1, 2, 3, 4 and 5 weeks after the initial measurements. At each time interval, specimens were removed from their respective solutions, wiped gently with a soft paper towel to remove excess solution, weighed, measured and immediately returned to their respective solution. Following the 5-week immersion period, specimens were removed from the solutions and placed in a desiccator containing calcium sulfate for 5 weeks.

Weight and dimensional change were determined at 1 day, 3 day then at 1, 2, 3, 4 and 5 weeks. At each time interval, the volume was calculated as above.

The values of water sorption \(W_s\) and solubility \(W_r\) were calculated using the following equations (ISO 4049:2000): 

\[
W_s = \frac{m_1 - m_2}{V}
\]

\[
W_r = \frac{m_0 - m_0}{V}
\]

Where \(m_0\) is the specimen mass before immersion (mg), \(m_i\) is the specimen mass after immersion (mg),
m is the specimen mass after desiccation (mg), and V is the specimen volume before immersion (mm³).

Flexural strength was determined using A4, B4, C4 and D4 specimens (25 mm × 2 mm × 2 mm) according to ISO standard – 4049. After final setting, specimens were removed from the mold and maintained at room temperature for 24 h in incubator. The specimens were subjected to 3 points bending test in universal testing machine.

**Results**

The means of sorption and solubility values are shown in Tables 1 and 2, respectively.

Graph 1 showing the flexural strength of 4 different luting cements. The result of the study clearly depicted that highest water sorption was observed for GC Fuji Cem in distilled water and artificial saliva media. For Composite Ionomer highest sorption was observed in ethanol:water medium. Panavia F showed least sorption in all three media. GC Fuji Cem exhibited more solubility in artificial saliva and ethanol:water (1:1) media. Composite Ionomer showed more solubility in distilled water medium. Secure luting agent showed least solubility in all three media. Highest flexural strength was observed by Panavia F and least by Tokeyoma Ionomer.

**Discussion**

The clinical importance of dental luting agents exhibiting properties such as water sorption, solubility, and flexural strength cannot be neglected. Previously, studies used water, acids, and other solvents to simulate the contaminating environment of mouth to test behavior of luting agents. For *in vitro* tests, the chemical structure of the solutions should be used as it stimulates the complexity of the oral environment. In this study, to maintain standardization of the test conditions, samples were kept in incubator at 37°C during immersion period and to maintain pH of storage media, it was changed every week.

The result of this study showed that highest water sorption was observed with GC Fuji Cem followed in decreasing order by Composite Ionotite, Secure and Panavia F in artificial saliva and distilled water media. But in ethanol:water (1:1) medium, the highest sorption was found for Composite Ionotite. The least sorption found for Panavia F in all three media.

The results of Yoruc and Karaaslan showed the water sorption was seen to be maximum on the first day of immersion, followed by gradual intake over a period of time until an equilibrium state was attained. They stated that water absorption of glass ionomer cement (GIC) is mainly dependent on material composition.

High water sorption of resin-modified GIC was noticed compared to other resin luting agents in this study. The main reason for hygroscopic expansion was believed due to the presence of hydroxyl-ethyl methacrylate, a significant monomer component of resin-modified GIC. Hygroscopic expansion may be considered as a favorable property, as its initial effect may contribute to compensate any polymerization shrinkage. Increase amount of water sorption in GC Fuji Cem results may result in higher hygroscopic expansion, which in turn could lead to unfavorable forces acting on the tooth structure and the restoration. Scientific evidence shows that absorbed water works as a plasticizer for the cement, creating unsupported areas.

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**Table 1: Mean water sorption values**

<table>
<thead>
<tr>
<th>Group</th>
<th>M2</th>
<th>M3</th>
<th>W' (%)</th>
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<tbody>
<tr>
<td>A1</td>
<td>589.53</td>
<td>565.41</td>
<td>15.36</td>
</tr>
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<td>B1</td>
<td>589.24</td>
<td>557.01</td>
<td>20.53</td>
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<tr>
<td>C1</td>
<td>691.69</td>
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<td>5.57</td>
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<tr>
<td>D1</td>
<td>529.22</td>
<td>523.18</td>
<td>5.26</td>
</tr>
<tr>
<td>A2</td>
<td>618.79</td>
<td>564.31</td>
<td>34.70</td>
</tr>
<tr>
<td>B2</td>
<td>596.63</td>
<td>552.22</td>
<td>28.29</td>
</tr>
<tr>
<td>C2</td>
<td>701.42</td>
<td>681.64</td>
<td>12.6</td>
</tr>
<tr>
<td>D2</td>
<td>539.85</td>
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<td>9.79</td>
</tr>
<tr>
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<td>591.53</td>
<td>563.69</td>
<td>17.73</td>
</tr>
<tr>
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<td>585.42</td>
<td>556.26</td>
<td>18.57</td>
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<tr>
<td>C3</td>
<td>691.83</td>
<td>679.83</td>
<td>7.64</td>
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<tr>
<td>D4</td>
<td>531.65</td>
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</table>

**Table 2: Mean water solubility values**

<table>
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<th>Group</th>
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<th>M3</th>
<th>W' (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>A1</td>
<td>571.66</td>
<td>565.41</td>
<td>5.68</td>
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<tr>
<td>B1</td>
<td>564.47</td>
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<td>D1</td>
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<td>1.81</td>
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<td>564.31</td>
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<td>B2</td>
<td>562.83</td>
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<td>6.76</td>
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<tr>
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<td>685.74</td>
<td>681.64</td>
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<tr>
<td>D4</td>
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<td>524.48</td>
<td>2.15</td>
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underneath restoration, and consequently, increasing the chance of fracture of restoration under masticatory forces.\textsuperscript{[11,12]}

Highest water solubility was observed with GC Fuji Cem followed by Composite Ionotite, Panavia F and Secure in ethanol:water (1:1) and artificial saliva medium. But in distilled water medium, highest solubility was found for Composite Ionotite. The least solubility found for Secure in all three mediums.

In this study, resin modified GIC showed a remarkable increased solubility in comparison with the other resin luting agents used. The water absorption by GC Fuji Cem is a diffusion controlled process, and the water uptake occurs largely in the resin matrix. The water absorbed by the polymer matrix could cause filler matrix debonding or even hydrolytic degradation of the fillers and may affect composite materials by reducing their mechanical properties. The hydrolytic degradation is a result of either the breaking of chemical bonds in the resin or softening through the plasticizing action of water. When resin samples immersed in water, some of the components such as unreacted monomers dissolve and are leached out of the samples.\textsuperscript{[13-15]}

Dual cure self-adhesive resin luting agents had lower solubility compared to self-cure self-adhesive because they might have a higher degree of polymerization due to two initiation system.\textsuperscript{[16]}

When sorption and solubility of luting agents were compared with values in distilled water, artificial saliva and ethanol: water, a very highly significant difference was found. From this study, it is clear that luting cements were more soluble in ethanol: water (6.76%) than in distilled water (4.75%) and artificial saliva (4.31%) which showed concurrence with the study done by Masaki Iwaku for resin reinforced GIC.\textsuperscript{[17]}

Flexural strength describes the amount of force required to bend and break the material when a test piece of specific thickness is loaded. This is also called as “transverse strength.”\textsuperscript{[18]}

In the present study flexural strength was significantly high in case of Panavia F (81 MPa) and relatively low in case of Composite Ionotite (18MPa). Panavia F > Secure > GC Fuji Cem > Composite Ionotite.

Huan Lu et al.\textsuperscript{[19]} showed that the form of initiation system had a considerable effect on flexural strength of the resin luting agents. When two initiation systems are used better polymerization is achieved for the dual-polymerized study samples. Their results are similar to this study. Thus, it is recommended that the dual cure luting agents should be light initiated to achieve better mechanical properties like higher strength and modulus of elasticity.

Cattani Lorente et al.\textsuperscript{[20]} reported that water absorption of luting agents after a long-term immersion in an aqueous solution could result in compromised mechanical properties. Fractions of this absorbed water could reduce the strength of the dental luting agents due to the erosive and plasticizing effect of water.\textsuperscript{[21-23]}

Therefore using these luting agents clinically for cementation of all-ceramic crowns is questionable.\textsuperscript{[24,25]}

Alcohol-containing solution appears to have some effect on cement sorption and solubility. Toledano et al. showed alcohol-containing solutions have increased the solubility of self-cured resin composite.\textsuperscript{[26]} Likewise, Moraes Porto et al. showed that Listerine mouthwash could increase the solubility in two tested composite resins.\textsuperscript{[27]} This was attributed to the efficiency of ethanol as the solvent of resin cross-linking networks. Ethanol can easily penetrate the resin matrix and cause swelling and release of unreacted monomers.\textsuperscript{[28,29]} As unreacted monomer is likely to remain within the cement mass, it is vulnerable to be dissolved by the solvent. Hand-mixing of resin material may incorporate air voids that can induce inhibition of resin polymerization; thus, increase the amount of monomer and subsequent solubility.\textsuperscript{[30]}

Furthermore, the porosity enhances the transportation of fluid through the cement, the subsequent swelling and cross-linking dissolution.\textsuperscript{[31]}

The limitation of this study is that the tests do not simulate the exact clinical situations. The longevity of any luting cement depends on various other factors such as the clinical conditions, amount of moisture contamination, powder-liquid ratio, mixing technique, manufacturer and the batch of the luting cement. In this study, only static forces are considered but not the complex dynamic forces that act on restoration in the oral cavity. However, these \textit{in vitro} results are important for screening the sorption, solubility and flexural strength qualities of the different luting cement types. For further evaluation, additional studies are required.

**Conclusion**

Within the limitation of this study, it can be concluded that:

1. The resin-based luting agents demonstrated the least amount of water sorption and solubility among all specimens. Of these, the dual cure resin luting agents demonstrated the least water sorption and solubility than self-cure resin luting agent.

2. The result of this study showed that highest water sorption was observed with GC FUJI CEM followed in decreasing order by Composite Ionomer, Secure and Panavia F in artificial saliva and distilled water media. But in ethanol:water medium, highest sorption was found for Composite Ionomer. Highest water solubility was observed with GC Fuji Cem followed by Composite Ionomer, Panavia F and SECURE in ethanol:water (1:1) and artificial saliva media, But in distilled water medium, highest solubility was found for Composite Ionomite.

3. All the luting cements showed highly significant increase in sorption and solubility in ethanol:water medium when compared to the sorption and solubility in artificial saliva and distilled water media.

4. Panavia F luting agent showed least water sorption in all three media.

5. Secure luting agent showed least solubility in all three media.

6. Highest flexural strength was observed by Panvia F and least by Tokeyoma Ionomer.

**References**

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