

Original Article

## Evaluation of Compressive Strength and Sorption/Solubility of Four Luting Cements

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### Abstract

**Statement of Problem:** Compressive strength (CS) and sorption/solubility of the luting cements are two associated factors. Searching a correlation between sorption/solubility and compressive strength of various luting cements is required.

**Objectives:** To measure the water sorption/solubility, and compressive strength of three resin-based and one conventional glass ionomer (CGI) luting cement after 1 and 24 h of immersion in distilled water and to determine if there is any correlation between those properties found.

**Materials and Methods:** Four luting cements were investigated. For each material, 10 disc shaped specimens were prepared for measuring the sorption/solubility. The specimens were cured according to the manufacturer's instructions, and the sorption/solubility were measured in accordance with the ISO 4049's. For testing the compression strength, for each material 16 cylindrical specimens were prepared by insertion of cements into a stainless steel split mould. The specimens were cured, divided into groups of 8, and then stored in distilled water at  $(37 \pm 1)^\circ\text{C}$  for 1 and 24 h. The test was performed using the universal testing machine, the maximum load was recorded and CS was calculated. The data were analysed using SPSS software version 18. One-way ANOVA, *post-hoc* Tukey's test and Pearson's correlation coefficient were performed.

**Results:** G-CEM had the highest mean CS ( $153.60 \pm 25.15$ ) and CGI luting had the lowest CS ( $21.36 \pm 5.37$ ) ( $p < 0.001$ ). After 24 h, mean CS values showed an increase for almost all materials except for RelyX<sup>TM</sup> U200 which showed a slight reduction. However, no statistically significant difference was founded (all  $p > 0.05$ ). The lowest mean sorption/solubility value was for RelyX<sup>TM</sup> U200 and Panavia F, and the highest for CGI luting (all  $p < 0.001$ ).

**Conclusions:** The compressive strength of all cements did not necessarily increase after 24 h and varied depending on the materials. There was a strong reverse correlation between sorption and CS values after both 1 and 24 h immersion. It may be practical for clinician to use those cements with the less sorption / solubility and more stable compression strength over time.

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## Introduction

Several categories of the luting cements are being used for cementation of direct or indirect restorations to the tooth structure [1]. Water based cements such as zinc phosphate and polycarboxylate cements have been used for a long time as the main luting agent for the cementation of restoration. Since 1972 [2] when it was introduced, glass ionomer cements have gained popularity due to their clinical advantages including fluoride release and their adhesion capabilities to the tooth structure [3]. However, poor early mechanical properties and moisture sensitivity are considered as disadvantages of these materials [4].

The resin luting cements are the latest type of luting agents for the cementation of indirect aesthetic restorations such as all-ceramic and porcelain veneers. Resin cements are classified into two types: conventional and self-adhesive resin luting cements. The self-adhesive resin luting cements (SARLC) have the ability to bond to the tooth structure and the internal surface of the restoration without using adhesive system. Resin luting cements are comprised of the same basic constituents as the composite restorative material but with lower concentration of filler particles with a variable amount of 55–70 W% [5]. Resin cements have advantages of colour stability, adhesion to dental structure and other materials, low water sorption and solubility, and better mechanical properties in comparison to traditional cements [6-8]. There are also major disadvantages, such as polymerization shrinkage [8].

Luting cements seal the interface of the indirect restoration and the prepared tooth surface; hence, the clinical success of the restoration depends on the mechanical and physical properties of the luting cements over the duration of the restoration [9]. These materials are subjected to forces of mastication and transferring the stresses from indirect restorations to the tooth structure. Therefore, it is necessary for luting cement to provide high strength in order to maintain the durability and success of the restorations [10,11].

In addition to the mechanical properties, other clinically related characteristics including dimensional stability and structural integrity should be considered for the selection of a durable luting agent [3]. To maintain the dimensional stability, luting cement should have adequate resistance to fracture and deterioration when exposed to the oral cavity fluids [1]. Water sorption and solubility may cause

degradation of the cement, leading to disintegration in margin of the restorations. Clinically, loss of marginal integrity can cause unwanted consequences such as marginal leakage and discoloration, secondary caries, hypersensitivity, releasing toxic substances, and finally may result in debonding or the fracture of the restoration [3,12].

Previous studies reported comparable moisture sensitivity for the resin modified (RMGIC) with conventional glass ionomer cements [10]. Knobloch *et al.* [3] investigated sorption and solubility of 3 RMGICs and 3 resin cements reported that water sorption was significantly higher in all RMGICs as compared to resin cements, attributing it to the hydrophilic nature of HEMA in the composition of RMGICs [3]. Similarly, by evaluating the sorption and solubility of eight luting agents (two RMGICs and six resin cements) in distilled water and ethanol, Mese *et al.* found that RMGICs exhibited higher solubility and sorption as compared to resin cements [12].

Although the resin luting cement showed lower water sensitivity, the weakening of their mechanical properties has been reported after water storage [13]. Water is mainly absorbed by the polymer matrix, leading to hydrolytic degradation, debonding of the fillers, and matrix softening. When the resin matrix starts swelling because of water sorption, unreacted monomers and deboned fillers may leach out from the matrix. Consequently, moist conditions can decrease the final strength and increase the creep [12]. Örtengren *et al.* [14] studied the effect of 60 days of water sorption on the flexural strengths of two resin cements. Their findings showed that after water storage, resin cement revealed significantly lower flexural strength and higher deflection which may be explained by the plasticizing effect of water on the polymers.

Several studies have compared the mechanical properties, and water sorption / solubility of different classes of luting cements [11,12]. Yet, the correlation between water sorption and solubility of various categories of luting cements and their compressive strength has not been widely studied.

Therefore, the present study aimed to place various luting cements in distilled water and determine: 1) the effect of 1 and 24 h immersion on the compressive strength; 2) water sorption/solubility of those cements in accordance with the ISO 4049's; 3) if a correlation exists between the sorption/solubility and the compressive strength of the luting cements.

The null hypothesis is that the storage time does not affect compressive strength, or there is no correlation between the compressive strength and water sorption/solubility of the cements.

## Materials and Methods

Four luting cements were investigated in this study, as shown in Table 1.

right angles to each other were made and the area (in square millimetres) was calculated by using the mean diameter. By measuring the thickness of the specimen at the centre of the specimen and at four equally spaced points on the circumference, the volume,  $V$ , was calculated in cubic millimetres as follows:  $V = \pi \times (d/2)^2 \times h$ , where  $\pi = 3.14$ ,  $d$  is the mean diameter and  $h$  is the mean thickness of the specimen. The specimens of each material were immersed in distilled

**Table 1:** Description of the materials

Cement	Manufacturer	Type	LOT Number	Expiry date
RelyX™ U200 Automix syringe	3MESPE, St. Paul, MN, USA	SARLC	635906	2017-12
Panavia F 2 pastes	Kuraray Noritake Dental Inc., Okayama, Japan	CRLC	000035	2016-09-05
G-CEM Encapsulated	GC corporation, Tokyo Japan	SARLC	1307041	2016-07
CGI luting & lining cement Powder and liquid	GC corporation, Tokyo Japan	CGIC	1407171	2017-07

SARLC= Self Adhesive Resin Luting Cement, SGIC= Conventional Glass Ionomer luting Cement, CRLC= Conventional Resin Luting Cement.

### Water sorption and Solubility test

For each material, 10 disc-shaped specimens of  $10 \pm 0.1$  mm of diameter and  $1.0 \pm 0.1$  mm of thickness were prepared. A polyethelene mould was filled with the material according to the manufacturer's instructions. Then, the mould was sandwiched between two pieces of Mylar strip and pressed by two glass plates under hand pressure to remove the excess material. The light cured specimen were eradicated for the recommended exposure time through Mylar strip using LED curing unit at a wavelength range of 440-480 nm and an emitting light intensity of 1500 mW/cm<sup>2</sup> (Radii plus LED; SDI, Melbourne, Victoria, Australia). In accordance with the ISO 4049's instructions [15], the specimens were transferred to a desiccator (Labx Company, Ontario, Canada) containing freshly dried silica gel (SIGMA-ALDRICH, Taufkirchen, Germany) maintained at  $(37 \pm 1)^\circ\text{C}$ . After 22 h, the specimens were removed and transferred to a second desiccator maintained at  $(23 \pm 1)^\circ\text{C}$  for another 2 h.

After 24 h, the specimens were removed and weighed on an electronic balance (GR-3000, A & D CL Toshiba, Tokyo, Japan) to an accuracy of  $\pm 0.1$  mg. This procedure was repeated every 24 h until a constant mass,  $m_1$ , was obtained. After the final drying, two measurements of the diameter at the

water at  $(37 \pm 1)^\circ\text{C}$  for 7 d. After this time period, the specimens were removed, washed with distilled water, air dried and then weighed in one minute and recorded as  $m_2$ .

The specimens were reconditioned to a constant mass in the desiccator. The constant mass was recorded as  $m_3$ . The values for water sorption,  $W_{sp}$ , and solubility,  $W_{sl}$ , in  $\mu\text{g}/\text{mm}^3$ , was calculated using the following equations:  $W_{sl} = (m_1 - m_3)/V$  and  $W_{sp} = (m_2 - m_3) / V$  where  $m_1$  is the conditioned mass prior to immersion in water;  $m_2$  is the mass of the specimen after immersion in water for 7 d;  $m_3$  is the mass of the reconditioned specimen (all in micrograms); and  $V$  is the volume of the specimen, in cubic millimetres.

### Compression test

For each material, 16 cylindrical specimens of 4.0 mm in diameter and 8.0 mm long were prepared by insertion of cements into a stainless steel split mould. The light cure materials were cured according to the manufacturer's instruction by the same LED-curing unit mentioned above. The specimens were removed from the split mould and cured with the same exposure time on the opposite side and each lateral face. The specimens were divided into groups of 8

**Table 2:** Mean  $\pm$  SD (MPa) of compressive strength for all luting cements analysed using Tukey's test

Luting cement	1 hour	24 hours	p value*
RelyX™ U200	138.50 $\pm$ 22.8 <sup>A</sup>	129.20 $\pm$ 19.92 <sup>A</sup>	0.59
Panavia F	110.07 $\pm$ 47.5 <sup>A</sup>	162.33 $\pm$ 22.00 <sup>A</sup>	0.31
G-CEM	153.60 $\pm$ 25.15 <sup>A</sup>	162.60 $\pm$ 18.48 <sup>A</sup>	0.65
CGI Luting	21.36 $\pm$ 5.37 <sup>B</sup>	30.50 $\pm$ 22.78 <sup>B</sup>	0.63

- In each column, mean values with different upper case letters show a significant difference ( $p$  value  $< 0.05$ ).

\*shows significance level of student's  $t$  test between two storage time in each material.

( $n=8$ ) and then stored in distilled water at ( $37 \pm 1$ )°C for one and 24 h.

Following the storage, the compression strength test was performed using the universal testing machine (Zwick/Roll Z020, Zwick GmbH & Co, Germany) loaded at a crosshead speed of 0.5mm/min. The maximum load at specimen failure was recorded and compressive strength was calculated using the following formula:  $\delta=F/S$  where  $F$  is the maximum load at the fracture point (N) and  $S$  is the surface area of the specimen (mm<sup>2</sup>).

#### Data Analysis

The data were analysed using SPSS software version 18 (SPSS Inc., Chicago, IL, USA). One-way ANOVA was used to compare different variables between the materials, and *post-hoc* Tukey's test was performed to show significant differences in subgroup comparisons. Student's  $t$  test was used to show significant differences between the two storage times for compressive strength of each material. Correlations between sorption/solubility and compressive strength were assessed using Pearson's correlation coefficient.  $p$  value of  $< 0.05$  was considered to be statistically

significant.

#### Results

As shown in Table 2, after 1 h of immersion in distilled water, the highest mean CS value was observed for G-CEM (153.6  $\pm$  25.15), followed by RelyX™ U200 (138.5  $\pm$  22.8) and Panavia F (110.07  $\pm$  47.5). CGI luting showed the lowest mean CS (21.36  $\pm$  5.37) ( $p < 0.001$ ). After 24 h, mean CS values were numerically but not statistically raised for all the cements except for RelyX™ U200 which showed a slight reduction. After 24 h, CS values for Panavia F increased as high as G-CEM with the following order: G-CEM = Panavia F > RelyX™ U200 > CGI luting.

Table 3 compares the water sorption and solubility of all tested cements. The lowest mean sorption value was for RelyX™ U200 (23.6  $\pm$  1.59) and Panavia F (24.10  $\pm$  6.87). The highest mean sorption value was for CGI luting (76.20  $\pm$  6.17) ( $p < 0.001$ ). The lowest mean solubility value was found to be for RelyX™ U200 and Panavia F followed by G-CEM, and CGI luting, respectively ( $p < 0.001$ ).

There was a strong reverse correlation between sorption and CS values after both 1 and 24 h of immersion ( $r = -0.702$ ,  $p = 0.002$ ; and  $r = -0.775$ ,  $p < 0.001$ , respectively). Solubility showed a moderate reverse correlation with CS values after 1 h ( $r = -0.530$ ,  $p = 0.035$ ) and no significant correlation after 24 h ( $r = -0.409$ ,  $p = 0.116$ ).

#### Discussion

Compressive strength and sorption/solubility are correlated factors which enhance the longevity of bonded restorations when they are in an optimum level. A high compressive strength of the luting cements enables them to withstand masticatory forces in the mouth and increases the fracture resistance of

**Table 3:** Mean  $\pm$  SD ( $\mu\text{g}/\text{mm}^3$ ) of sorption and solubility for all luting cements analysed using Tukey's test

Luting cements	Sorption	Solubility
RelyX™ U200	23.61 $\pm$ 1.59 <sup>A</sup>	1.23 $\pm$ 0.83 <sup>A</sup>
Panavia F	24.10 $\pm$ 6.87 <sup>A</sup>	8.40 $\pm$ 4.28 <sup>AB</sup>
G-CEM	38.90 $\pm$ 4.59 <sup>B</sup>	17.29 $\pm$ 9.55 <sup>BC</sup>
CGI Luting	76.20 $\pm$ 6.17 <sup>C</sup>	27.97 $\pm$ 2.11 <sup>C</sup>

- In each column, mean values with different upper case letters show a significant difference ( $p$  value  $< 0.05$ ).

( $p < 0.05$ ).



the restoration, especially in brittle materials such as ceramics [15].

In this experiment, compressive strength and water sorption/solubility of two self-adhesive resin luting cements (SARLC), one conventional resin luting cement (CRLC), and one conventional glass ionomer (CGI) luting were examined and compared. In general, CGI luting exhibited significantly lower compressive strength and higher water sorption / solubility than both SARLC and CRLC. Comparison of the resin cements showed that CRLC had a lower strength than SARLC but it showed a comparable or less water sorption / solubility.

As shown in Table 2, compressive strength of all materials showed an increase after 24 h of immersion in distilled water except for RelyX™ U200 which revealed a slight decrease after 24 h. The increase in the materials' strength after 24 h can be related to their setting reaction. For glass ionomer cements, after mixing, the calcium polycarboxylate is formed in the first few minutes while the aluminum polycarboxylate, which improves the mechanical properties of the cement, takes at least 24 hours or even longer to be formed [16-18].

Likewise, for resin-based materials, polymerization and maturation take 24 h to be completed. Therefore, the mechanical properties of these materials are expected to be improved significantly after 24 hours. In the present study, one of the SARLC (RelyX™ U200) had a slight reduction from  $\approx 138$  to  $\approx 129$  MPa while the other one (G-CEM) had a slight increase from  $\approx 153$  to  $\approx 162$  MPa. The CRLC, Panavia F, showed the lowest compressive strength ( $\approx 110$  MPa) among resin-based cements after 1 h of setting while after 24 h of setting, it had a remarkable increase ( $\approx 162$  MPa) that was equal to the highest value for G-CEM.

These results suggest that the growth in the luting cements' strength is not directly dependent on the period of 24 h of setting. The effect of 24 h of immersion in distilled water on the compressive strength of the resin cements varied depending on the materials. Some materials showed a slight reduction and some others significant increase in strength after 24 h of immersion. This result is in agreement with that of a previous study [19].

A previous study [19] evaluating the shear punch strength of resin cements reported a reduction of strength for almost all SARLCs after one week, one month and even more after three months of immersion in distilled water. While Panavia F with HEMA

content in their composition showed an increase after one-week of immersion, it has been concluded that the HEMA could be a major factor in the reinforcement or stiffness of the polymer system [20].

It has been shown [21] that the water sorption of a SARLC (Maxcem) was significantly higher than a CRLC (Panavia F). It has also been hypothesized that the significant decrease in the strength of SARLC might be related to its greater water sorption than CRLC [19]. However, the results of our study did not prove this claim. The water sorption / solubility of the tested luting cements followed the following trend: RelyX™ U200 < Panavia F < G-CEM < CGI luting.

This result reveals that although G-CEM had the greatest water sorption/ solubility among other resin-based cements, did not drop the strength but also exhibited the highest strength value either after 1 or 24 h of immersion in water. On the other hands, RelyX™ U200 with the least sorption and solubility exhibited the lowest strength after 24 h of immersion. In other words, G-CEM with higher strength than RelyX™ U200 had higher water sorption and solubility. Hence, it is speculated that having a high water sorption/solubility does not necessarily decrease the compressive strength of resin luting cements. But, some factors other than sorption/solubility, such as polymerization, mode of curing (dual- or self-cure), and degree of conversion (%DC) might have a great influence on the strength [22] or sorption/ solubility [23] of the resin luting cements.

Factors that may affect polymerization include cement film thickness, opacity, and translucency of both the cement and restoration. Properly cured resin cement will exhibit high compressive and flexural strengths and less solubility in the oral fluids. The mixing method of the resin cement is also an important clinical factor that improves the performance of the resin cement [22]. Although resin cements are insoluble in oral fluids, being resins, they absorb water. Due to water sorption, the flexural strength of the resin cements is decreased [24]. The flexural strength affected by the thickness of the cement, the greater decrease occurred in the greater film thickness of the cement because it makes the cement unable to scatter stresses from masticatory function between the tooth and restoration. Therefore, keeping the resin cement layers to a thin layer minimizes the plasticizing effect in the resin cements [25].

In the present study, Panavia F (a dual-cure luting cement) showed significantly less sorption/solubility than G-CEM (a light cure luting cement) which can

be due to their different mode of curing. A recent study [23] investigated the effect of curing mode on the surface energy and sorption/solubility of SARLC and CRLC and found a greater sorption/solubility values for SARLC than the CRLC. The author concluded that the degree of conversion (%DC) was negatively correlated with the sorption/solubility values. Dual curing is shown to reduce the sorption and/or solubility in comparison with self-curing by increasing %DC [23].

The convention glass ionomer used in this study (CGI Lutin) exhibited the lowest strength and the highest sorption and solubility. It is well known that water has an important role in the glass ionomer cement. It contributes to the transportation of calcium and aluminum cations, reacting with the polyacid to form a polyacrylate matrix [26]. During the early stage of maturation, moisture contamination leads to loss of components, decrease of physical properties and loss of translucency [27]. After hardening, desiccation and loss of water results in the inadequacy of the reactions and surface crazing [28]. Therefore, it is expected to absorb and lose water easily.

## Conclusions

Within the limitations of this study, the following conclusions were drawn. In general, most of the cements showed an increase in the compressive strength after 24 h immersion in comparison with the 1 h immersion except for RelyX™ U200 that had a slight decrease. RelyX™ U200 and Panavia F, revealed the lowest sorption / solubility and CGI luting the highest. There was a strong reverse correlation between sorption and CS values after both 1 and 24 h immersion. Based on the results of this study, it is speculated that it may be practical for clinician to use those cements with the less sorption / solubility and more stable compression strength over time.

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**Conflict of Interest:** None declared.

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