Original article

Mechanical properties of new self-adhesive resin-based cement

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Abstract

Purpose: The aim of this study was to compare the bonding strength, flexural strength, elastic modulus, water absorption and the expansion after water storage of new self-adhesive resin cements to commercially available dental cements.

Methods: Two types (hand-mix and auto-mix) of new self-adhesive resin cements (SAC-H and SAC-A, Kuraray Medical), one conventional resin cement (Panavia F2.0), three self-adhesive resin cements (Relyx Unicem, Maxcem and G-Cem), and two resin-modified glass-ionomer cements (Fuji Luting S and Vitremer) were used. Shear bond strengths, flexural strengths and elastic moduli (ISO 4049), water absorption (ISO 4049), and the expansion rate after water storage were investigated.

Results: Both SAC-H and SAC-A provided adhesion to enamel and dentin, and had the same bond strength to gold alloy and zirconia as conventional resin cements. SAC-H and SAC-A had greater flexural strengths (86.4–93.5 MPa) than commercial self-adhesive resin cements or glass-ionomer cements. The elastic moduli of self-adhesive and glass-ionomer cements were 5.2–7.4 GPa and 2.3–3.4 GPa, respectively. The water absorption of SAC-H and SAC-A (26.3–27.7 mg/mm³) were significantly lower than commercial self-adhesive resin cements. SAC-H and SAC-A showed significantly lower expansion rates (0.17–0.26%) than commercial self-adhesive cements and glass-ionomer cements after 4 weeks water storage.

Conclusions: It is suggested that the new self-adhesive resin cements exhibited a favorable bonding capability and mechanical properties.

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Keywords: Self-adhesive cement; Bond strength; Flexural strength; Water absorption; Expansion rate

1. Introduction

Resin-based cements are widely used for luting inlays, crowns and veneers because they adhere to metal and ceramics. However, teeth and restorations require surface treatments, such as etching and bonding, when conventional resin-based cements are used [1,2], making the luting operation using resin-based cements technique-sensitive. Thus, if the surface treatment is insufficient, the bond strength will be impaired. Resin-based self-adhesive cements have recently been made available to deal with this problem. Self-adhesive cements do not require any surface treatment of the teeth or restorations, so they are said to be easier to handle and have clinically effective bond strength. In fact, it is reported that self-adhesive resin cements provide the equivalent bond strength of conventional resin cements to dentin [3,4], gold alloy and glass ceramics [5], and zirconia [6,7]. On the other hand, resin-based cements have greater flexural strength [8], lower water sorption [9], and better mechanical properties than resin-modified glass-ionomer cements.

The newly developed resin-based self-adhesive cements examined in this study contain a great deal of adhesive acidic monomer to enhance bonding to the adherent surface, along with catalysts that provide improved curing properties. It is therefore expected that they will offer excellent adhesion and great mechanical strength as well as low water sorption and expansion. The purpose of this study is to confirm the bond strength of the newly developed resin-based self-adhesive cements and evaluate their mechanical properties by comparing with other resin cements and resin-modified glass-ionomer cements.

The null-hypothesis is that the flexural strength, water sorption, and changes in dimension of new self-adhesive cements will be inferior to those of conventional resin cements.
2. Materials and methods

2.1. Bond strength

The following five materials were used to evaluate shear bond strength. SAC-H (the hand-mixed type, Clearyl SA Luting) and SAC-A (the auto-mixed type, Clearyl SA Cement), manufactured by Kuraray Medical (Tokyo, Japan), were used to represent the new type of resin-based self-adhesive cement. Panavia F2.0 (Kuraray Medical) was the conventional resin cement. Maxcem (Kerr, Orange, CA, USA) was the conventional resin-based self-adhesive cement. Fuji Luting S (GC, Tokyo, Japan) was the resin-modified glass-ionomer cement (Table 1). The adherents used were human tooth enamel and dentin, gold alloy and zirconia.

Extracted sound human teeth without caries or filling restoration were stored in 4 °C water and used as human tooth specimens. For our experiment, upper and lower premolars and molars that allowed a necessary bonding area were used.

Each adherent was embedded in a ring using composite resin and the surface of the adherent was polished. The enamel and dentin of extracted human teeth were polished with #80 and #1000 water-resistant polishing paper by using a rotary polishing machine (Ecomet 4, Buehler, Lake Bluff, IL, USA) and then cleaned by ultrasonic vibration for 2 min. The gold alloy (Casting Gold Type4, Ishifuku, Tokyo, Japan) was cast in the usual manner. The zirconia was milled using the CAD/CAM system (Cercon, DeguDent, Hanau, Germany), sintered according to the manufacturer’s instructions, and then polished with #80 and #1000 water-resistant polishing paper. Both the gold alloy and the zirconia were thereafter sandblasted with 50 μm alumina powder under a pressure of 0.4 MPa until the surface lost its luster, and then cleaned by ultrasonic vibration for 2 min. In case of the Panavia, the surfaces of the enamel and dentin adherents were treated using a self-etching primer (ED Primer II, Kuraray Medical), and the surfaces of the gold alloy adherents were treated using a primer (Alloy Primer, Kuraray Medical).

Each type of cement was mixed according to the manufacturer’s instructions. For shear bond strength tests, stainless steel chips, 3 mm in diameter and 3 mm in height, were used for the human enamel and dentin adherent samples, and stainless steel chips, 5 mm in diameter and 3.6 mm in height, were used for the gold alloy and zirconia adherent samples. The adherent surface of each stainless steel chip was sandblasted and then surface-treated with a primer (Alloy Primer, Kuraray Medical). Each of the cements, mixed according to the manufacturer’s instructions, was applied onto one of the stainless steel chips and then the chips were placed under pressure onto each of the embedded adherent samples, by applying a pressure of 500 g onto the stainless steel chip from above with a jig. The cement samples, other than Fuji Luting S, were light cured from a position as close to the surface of the chip and the cement sample as possible at an angle of 45 degrees, from two different directions, for 20 s.

For light-curing the cement samples, a light-curing unit (JET Light 3000, Morita, Osaka, Japan) which uses a halogen lamp as the light source with an effective wavelength of 400–520 nm, was used.

The load was applied for 10 min and the test item was allowed to stand for 1 h. The specimens thus prepared were stored in water at 37 °C for 24 h before being subjected to a shear bond strength test at a cross-head speed of 1 mm/min using a universal testing machine (Autograph AGI, Shimadzu, Kyoto, Japan). Seven specimens were prepared for each test condition. The results of the shear bond tests were analyzed using one-way ANOVA and a Scheffe’s multiple comparisons test as a significant level of 0.05.

2.2. Mechanical properties

The following eight cements were used to evaluate mechanical properties: the resin-based self-adhesive cements were SAC-H, SAC-A, Relyx Unicem (3 M ESPE, Seefeld, Germany), Maxcem, and G-Cem (GC); the conventional resin cement was Panavia F2.0; and the resin-modified glass-ionomer cements were Fuji Luting S and Vitremer (3 M ESPE) (Table 1).

The three-point bending test and water sorption test were performed according to ISO4049 [10]. In addition, the expansion due to water sorption of each product was examined by using disc-shaped specimens of each product. Rectangular specimens (2 mm × 2 mm × 25 mm) were prepared for three-point bending test, using a stainless steel mold according to ISO4049 [10]. In addition, the following eight cements were used to evaluate mechanical properties: the resin-based self-adhesive cements were SAC-H, SAC-A, Relyx Unicem (3 M ESPE, Seefeld, Germany), Maxcem, and G-Cem (GC); the conventional resin cement was Panavia F2.0; and the resin-modified glass-ionomer cements were Fuji Luting S and Vitrmer (3 M ESPE) (Table 1).

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Finally, additional disc-shaped specimens (20 mm in diameter and 1 mm in thickness) were prepared using eight types of cements in the same manner as described for the above water sorption test. Two straight lines were drawn passing through the center of the specimen and crossing each other, and their length (the diameter of the sample) was measured using a micrometer to obtain the average diameter ($A$). The specimens were immersed in physiological saline solution at 37 °C and the length of the two lines was measured at 1 week, 2 weeks, 3 weeks and 4 weeks after the start of water immersion, to obtain the average diameter ($B$). Using the two average diameter values, the rate of expansion due to water sorption was calculated ($\frac{B}{A - 1} \times 100$). Five specimens were prepared for each test condition.

The statistical comparison of the specimens to the experiment was performed in the same manner as that for the bonding strength test.

### 3. Results

Panavia demonstrated the strongest bond strength to enamel (27.0 MPa), followed by SAC-H (19.8 MPa), SAC-A (18.3 MPa), Maxcem (10.9 MPa), and Fuji Luting S (10.1 MPa). Panavia also had the strongest bond strength to dentin at 19.1 MPa, followed by SAC-H (12.5 MPa), SAC-A (12.3 MPa), Maxcem (8.1 MPa), and Fuji Luting S (3.8 MPa). Both SAC-H and SAC-A had significantly stronger bond strengths than Maxcem and Fuji Luting S, with no significant difference found between SAC-H and SAC-A (Fig. 1). There was no significant difference in the bond strength to gold alloy among the following four types of cement: SAC-H (27.4 MPa), SAC-A (25.0 MPa), Maxcem (20.5 MPa) and Panavia (25.2 MPa). Fuji Luting S had significantly weaker bond strength (4.6 MPa) to gold alloy than the other four types of cement. Strength of bond to zirconia was 37.9 MPa for SAC-H, 40.3 MPa for SAC-H, and 40.7 MPa for Panavia; i.e., there was no significant difference among the three. But Fuji Luting S had significantly weaker bond strength (21.5 MPa) to zirconia than the other four types of cement (Fig. 1).

SAC-H and SAC-A had greater flexural strength (86.4–93.5 MPa) than commercial self-adhesive resin cements (29.7–74.7 MPa) or glass-ionomer cements (26.0–27.2 MPa) (Fig. 2). The elastic moduli of the self-adhesive and glass-ionomer cements were 5.2–7.4 GPa and 2.3–3.4 GPa, respectively (Fig. 3).

The water sorption of SAC-H, SAC-A and Panavia (26.3, 27.7 and 25.3 μg/mm²) were significantly lower than those of Maxcem and G-Cem (59.5 and 68.9 μg/mm²) (Fig. 4).

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**Table 1**

List of materials used in this study.

<table>
<thead>
<tr>
<th>Type</th>
<th>Product</th>
<th>Manufacturer</th>
<th>Shade</th>
<th>Lot No.</th>
</tr>
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<tbody>
<tr>
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<td>SAC-A</td>
<td>Kuraray Medical</td>
<td>Universal</td>
<td>A: SACA-A-2-061211 B: SACA-U-1-070529</td>
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<tr>
<td>Conventional resin</td>
<td>Panavia F2.0</td>
<td>Kuraray Medical</td>
<td>Brown</td>
<td>A: F3A-302 B: F3B-159</td>
</tr>
<tr>
<td>Self-adhesive resin</td>
<td>Relyx Unicem</td>
<td>3M ESPE</td>
<td>A2 Universal</td>
<td>56818</td>
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<tr>
<td></td>
<td>Maxcem</td>
<td>Kerr</td>
<td>Clear</td>
<td>2794357</td>
</tr>
<tr>
<td>Self-adhesive resin</td>
<td>G-Cem</td>
<td>GC</td>
<td>A2</td>
<td>Powder/Liquid: 0704051</td>
</tr>
<tr>
<td>Resin-modified glass-ionomer</td>
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<td>Yellow</td>
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</tr>
<tr>
<td></td>
<td>Viteremer paste</td>
<td>3M ESPE</td>
<td>White</td>
<td>HY7HY</td>
</tr>
</tbody>
</table>

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Fig. 1. Shear bond strength results (MPa). Vertical lines represent standard deviations.

Fig. 2. Flexural strength results (MPa). Vertical lines represent standard deviations.

Fig. 3. Elastic modulus results (GPa). Vertical lines represent standard deviations.
The expansion due to water sorption tended to be greater as time passes. Both SAC-H (0.17%) and SAC-A (0.26%) had significantly lower rates of expansion than the commercial self-adhesive cements (0.72–0.97%) and the glass-ionomer cements (1.23–2.33%) when measured at 4 weeks after the start of water immersion (Fig. 5).

4. Discussion

The newly developed self-adhesive cements, SAC-H and SAC-A, have almost the same chemical composition; the major difference between them is their mixing method. In our experiments, it was found that SAC-H and SAC-A had almost the same strength of bond and similar mechanical properties, and that these are not affected by the difference in mixing method.

Our experiments also showed that the bond strengths of commercial resin-based self-adhesive cement (Maxcem) to enamel and dentin were weaker than those of conventional resin cement (Panavia). This agreed with the result of a study [11] performed in the past which examined the use of Maxcem and Panavia in composite onlays. The bond strength of SAC-H and SAC-A to enamel and dentin fell short of that of Panavia, but was greater than that of Maxcem and resin-modified glass-ionomer cement (Fuji Luting S), suggesting that SAC-H and SAC-A are capable of providing adhesion to tooth structure without requiring the performance of surface treatment.

It is reported that in the conventional resin cement Panavia, the acidic monomer (10-methacryloyloxydecyl dihydrogen phosphate, MDP) contained in the self-etching primer used for conditioning the tooth surface is effective for improving the adhesion of the cement to dentin and enamel [12]. Both SAC-H and SAC-A also contain MDP as a component and it was therefore suggested that MDP might help enhance the adhesion of these cements to enamel and dentin. In addition, the bond strength of SAC-H and SAC-A to gold alloy and zirconia, or materials used for dental restorations, was greater than that of Maxcem and glass-ionomer cement and not significantly different from that of Panavia. MDP is reported to be effective in bonding to gold alloy [13] and zirconia [14], as well as to tooth structure. In our experiments, it also seemed that the MDP contained in SAC-H and SAC-A worked effectively to enhance the strength of the bond to metal and ceramics. It is reported [5–7] that self-adhesive cements other than SAC-H and SAC-A also had almost the same bonding strength to metal and zirconia as conventional resin cements, indicating that self-adhesive cements are capable of providing adhesion to those adherents without any preliminary surface treatment. However, self-adhesive cement is a relatively new material, so reports on this material have so far been limited to initial bonding strength. Thus the bond strength of self-adhesive cement, used over a long span of time, is not yet known. It is therefore necessary to observe the performance of the material for a long span, in order to achieve a long-term prognosis and achieve high reliability in clinical settings.

Three-point bending and water sorption tests were performed to examine the mechanical properties of the various cements. In general, it is known that resin-based cements have greater flexural strength than glass-ionomer cements [8]. In this study, resin-based cements (SAC-H, SAC-A, Panavia, Unicem, and Maxcem) revealed greater flexural strength than Fuji Luting S and Vitremer.

However, the flexural strength of G-Cem, which the manufacturer claims to be a resin-based cement, was almost the same as that of the two types of glass-ionomer cements examined, and it was suggested that G-Cem has physical properties similar to those of the resin-modified glass-ionomer cement.

It is reported [15] that Panavia, a conventional resin cement, has greater flexural strength than the self-adhesive cement, Unicem, and the results of our experiment also supported this report.

It was reported that as a result of an experiment performed according to the same ISO standard as that used for our experiment, the conventional indirect restoration composite resin had a bending strength of about 121–221 MPa [16].

The flexural strengths (86–94 MPa) of SAC-H and SAC-A tended to be lower than the flexural strengths of conventional resin composites [16], but are at the same level as or higher than Panavia (78.9 MPa). Therefore, this indicates that SAC-H and SAC-A have the same level of flexural strength as conventional.
resin cements although both are the self-adhesive type. The MDP contained in SAC-H and SAC-H helps improve the strength of these cements’ bonds, but might impair the curing process if too much is used. It is said that the curing property of SAC-H and SAC-A has been improved by increasing the catalysts contained in them. This may have contributed to their attaining the same level of flexural strength as conventional resin cements. Likewise the flexural strength, the flexural modulus of elasticity tended to be higher in the resin-based cements than the glass-ionomer cement. Generally speaking, materials with high flexural strength tend to have a high flexural modulus of elasticity, but we did find a cement like G-Cem, which had a high flexural modulus of elasticity but was not so flexurally strong [17]. As a result of finite element analysis, it has been reported that the stress applied on cement by luting a crown becomes greater in proportion to the flexural modulus of elasticity of the cement used [18]. Among the resin cements used in this study, Panavia and Unicem had high moduli of elasticity (6.9–7.4 GPa). However, the moduli of elasticity of Panavia and Unicem were slightly lower than that (7.9–20.9 GPa) of resin composite materials used for crowns and bridges [19]. Although SAC-H and SAC-A had the same level of flexural strength as Panavia, their modulus of elasticity was lower than that of Panavia, suggesting that there would be a small likelihood that the stress applied to the cement layer would cause microfractures in the cement.

The resin-modified glass-ionomer cement used in this study fractured during the drying process, and thus it was not used to prepare specimens for the water sorption test. This fracturing seemed to occur because the resin-modified glass-ionomer cement had so high a water sorption rate [9] that the physical properties of the cement were quite seriously changed due to water sorption. That is, this finding suggests that the physical properties of even resin-based materials will be changed for the worse if they absorb too much water [20]. In our experiments, SAC-H and SAC-A underwent little water sorption (26.3–worse if they absorb too much [20]. In our experiments, properties of even resin-based materials will be changed for the cement had so high a water sorption rate [9] that the physical seemed to occur because the resin-modified glass-ionomer preparation for the water sorption test. This fracturing fractured during the drying process, and thus it was not used to

5. Conclusions

SAC-H and SAC-A, recently developed resin-based self-adhesive cements, provided adhesion to tooth structure and yet had the same strength of bond to gold alloy and zirconia as conventional resin cements, without requiring surface treatment.

In addition, SAC-H and SAC-A had equivalent flexural strength to conventional resin cements and underwent less expansion due to lower levels of water sorption or water immersion than conventional cements. All of these suggest that they are clinically useful as dental cements. The null hypothesis was rejected.

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References


