Challenging the Resin-Zirconia Interface by Thermal Cycling or Mechanical Load Cycling or Their Combinations

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Abstract: The purpose of this in vitro study was to evaluate the influence of mechanical load cycling (MLC), which simulated mastication, alone or combined with thermal cycling (TC), on the resin shear bond strength (SBS) to zirconia. Two resin cements (Panavia F2.0 and RelyX U200) were bonded (bonding area: 2.38 mm) to air-abraded zirconia (Everest ZS-Ronde). The specimens were subjected to SBS test before and after TC (5000 cycles), MLC (5000 cycles in 37°C water), TC/MLC, or MLC/TC aging (n = 15). Before SBS test, the mechanical and physical properties of the two resin cements were studied (n = 5). For both resins, unlike TC (p > 0.05), the three MLC-containing aging conditions significantly decreased the SBS values when compared to the non-aged condition (p < 0.05). In the case of MLC-only aging, RelyX U200, with significantly higher hydrophobicity (p = 0.004), showed a significantly higher SBS value than Panavia F2.0 (p = 0.035). The MLC aging-containing groups showed increased occurrence of mixed failure. The application of MLC combined with TC may more closely simulate intraoral conditions.

Keywords: bonding durability; mechanical load cycling; thermal cycling; zirconia

1. Introduction

The increasing demand for all-ceramic restorations has led to the development of yttria-stabilized tetragonal zirconia polycrystal (Y-TZP) with enhanced mechanical properties such as high flexural strength and high fracture toughness [1,2]. Due to their superior mechanical performance, zirconia-based restorations not requiring high retention may be cemented using non-adhesive luting agents [3]. In many clinical situations, however, a strong, durable adhesion between the zirconia framework and the luting cement is a prerequisite for the long-term success of zirconia-based restorations [4–6]. Therefore, adhesive resin cements containing acidic functional monomers have been reported as the most appropriate materials, the monomers being capable of interacting chemically with the hydroxyl groups of the zirconia surface [6].

Although the initial bonding of the resin-zirconia interface is of importance, the natural aging of the interface needs to be simulated to evaluate long-term adhesion durability and to identify more effective bonding techniques and adhesive materials [7,8]. There are various artificial aging techniques for estimating the gradual degradation of the bonding interface. Among them, long-term water storage at a constant temperature or thermal cycling (TC) are most often used to simulate natural
aging of resin bonds [9,10]. However, the TC method remains controversial and there is no definite evidence that failure in practice occurs by thermal stress [10].

In TC tests, differential thermal changes can induce mechanical stresses and, as a result, cause crack propagation through bonded interfaces [11]. In mechanical load cycling (MLC), such crack formation and propagation seem to take place more directly, particularly when the bonded interface is brittle. Unlike TC, MLC has not been commonly used in dental research that challenges various bonded interfaces for the evaluation of bonding durability [12–14]. At present, however, there are no standardized MLC procedures and few studies using MLC to evaluate the adhesion durability between zirconia and resin.

The purpose of this study was to compare the influence of four aging methods (TC or MLC or their combinations) on the shear bond strength (SBS) of resin bonded to zirconia. Two adhesive luting cements with the potential to bond chemically to zirconia ceramic due to their functional monomers were used [3], and their mechanical/physical properties were investigated prior to bonding. The null hypothesis tested was that the aging methods do not differently decrease the SBS of the resins to zirconia.

2. Materials and Methods

2.1. Zirconia Specimens

Rectangular-shaped zirconia ceramic plates (10 mm × 10 mm × 1 mm; Everest ZS-Ronde, Kaltenbach & Voigt, Biberach, Germany) were sintered following the manufacturer’s recommendation and embedded using an acrylic resin. Each zirconia surface was air-abraded with 50 µm Al2O3 (pressure: 0.25 MPa; angle: perpendicular to the surface; distance: 10 mm; time: 10 s) [15], ultrasonically cleaned, and finally air-dried.

2.2. Characterization of Resin Cements

One adhesive resin cement (Panavia F2.0, Kuraray Medical Inc., Okayama, Japan) and a self-adhesive resin cement (RelyX U200, 3M ESPE, Neuss, Germany) were selected for this study (Table 1).

| Materials       | Manufacturer       | Component                                                                 | Lot No.       |
|-----------------|--------------------|---------------------------------------------------------------------------|---------------|
| Panavia F2.0    | Kuraray Medical Inc., Okayama, Japan | A paste: dimethacrylates, 10-methacryloyloxydecyl dihydrogenphosphate, camphorquinone, silica; B paste: dimethacrylates, barium glass, sodium fluoride | A paste: 00599C; B paste: 00122A |
| RelyX U200      | 3M ESPE, Neuss, Germany | Substituted dimethacrylate, 1-benzyl-5-phenyl-barbic-acid, calcium salt, 1,12-dodecanedimethacrylate, sodium p-toluene sulfinate, glass, silica, calcium hydroxide, titanium dioxide, methacrylated aliphatic amine | 553439 |

As the MLC test was performed in a wet environment, the flexural properties and surface energy parameters of the two resin cements were studied. To evaluate the flexural properties of the resins, the mixed resin paste was syringed into a stainless steel split mold with a dimension of 2 mm × 2 mm × 25 mm and covered by a polyester film; a glass slide was used to expel extra resin material on top of the mold [16,17]. The specimen was then light-cured for 40 s by placing the light guide tip of a dental light-curing unit (LCU, Bluephase® 20i, Ivoclar Vivadent, Schaan, Liechtenstein) against the slide in five overlapping sections [17]. All the resin specimens were stored in water for
24 h at 37 °C before testing. Dimensions of the specimens were determined by a digital caliper. The specimens (n = 5) were then subjected to a three-point bending test under a universal testing machine (UTS, Model 3343, Instron Inc., Canton, MA, USA; crosshead speed: 0.5 mm/min). The flexural strength (FS) in MPa was obtained with the equation [16,17]: 
\[ FS = \frac{3Fl}{2bh^2} \]
where F is the load at fracture (N), l is the support span (20 mm), b is the specimen width (mm), and h is the specimen depth (mm). The flexural modulus (FM) in GPa was also calculated [17]: 
\[ FS = \left[ \frac{Fl}{(6bh^3)} \right] \times 10^{-3} \]
in which F/\delta is the slope of the load-displacement curve (N/mm).

To evaluate the surface energy of the resin cements, contact angle (CA) measurements were performed. A split mold was used to prepare disc-shaped resins (diameter: 8 mm; thickness: 1 mm). Five resin discs were made for each material in a similar way as for flexural testing. The CAs (θ) of a liquid triplet (water, glycerol, and methylene iodide) with known surface energy parameters were determined on the resin surfaces using a CA goniometer (OCA 15 plus, DataPhysics, Filderstadt, Germany) [3,18,19]. The calculation was based on the Young-Dupré equation combined with the Lifshitz-van der Waals/Lewis acid-base (LW/AB) theory: 
\[ \gamma(I + \cos\Theta) = 2(\gamma_{s}^{LW} - \gamma_{s}^{LW})^{1/2} + (\gamma_{s}^{LW} - \gamma_{s}^{LW})^{1/2} + (\gamma_{s}^{LW} - \gamma_{s}^{LW})^{1/2} \]
in which \( \gamma_{s} \) and \( \gamma_{s} \) are the surface tensions of the liquid (l) and solid (s) surfaces, respectively; the superscripts + and - indicate the acid and base components, respectively. The total surface energy \( \gamma_{s} \) was calculated from: 
\[ \gamma_{s} = \gamma_{s}^{LW} + 2(\gamma_{s}^{LW} - \gamma_{s}^{LW})^{1/2} \]
In addition, the \( \gamma_{s}^{LW} \) and the \( \gamma_{s}^{AB} \) were calculated using the equations, respectively [20]: 
\[ \gamma_{s}^{LW} = \frac{2(\gamma_{s}^{LW} - \gamma_{s}^{LW})^{1/2} + (\gamma_{w}^{LW} - \gamma_{w}^{LW})^{1/2} - (\gamma_{w}^{LW} - \gamma_{w}^{LW})^{1/2} - (\gamma_{w}^{LW} + \gamma_{w}^{LW})^{1/2}}{2} \]
and 
\[ \gamma_{s}^{AB} = 2(\gamma_{s}^{LW} + \gamma_{s}^{LW})^{1/2} + (\gamma_{w}^{LW} + \gamma_{w}^{LW})^{1/2} - (\gamma_{w}^{LW} + \gamma_{w}^{LW})^{1/2} - (\gamma_{w}^{LW} - \gamma_{w}^{LW})^{1/2} \]
in which w refers to water.

The degree of hydrophobicity/hydrophilicity of each resin was expressed as the magnitude of \( \Delta G_{sws} = -2\gamma_{sw} \), in which G is the free energy and \( \gamma_{sw} = \gamma_{sw}^{LW} + \gamma_{sw}^{AB} \).

### 2.3. Shear Bond Strength (SBS) Test

The bonding of the resin cements to the air-abraded zirconia specimens was performed using the Ultradent jig method (Ultradent Products Inc., South Jordan, UT, USA) [18]. One bonded resin cylinder was made on one zirconia surface by packing the resin into plastic matrices (internal diameter: 2.38 mm) and then light-cured for 40 s using the LCU. All the specimens were immersed in water at 37 °C for 24 h before aging. Depending on the subsequent aging method, the specimens were divided into five groups each (including the non-aged (NA) group) for each resin cement. The specimens of the TC group were thermocycled 5000 times between 5 °C and 55 °C water baths (dwelling time: 30 s; exchange time: 5 s). MLC was used for the MLC group, the specimens being cycled 5000 times with a force of 50 N at a frequency of 1.66 Hz in 37 °C water (Figure 1) [21]. The TC then MLC (TC/MLC group) and MLC then TC (MLC/TC group) conditions were also studied.

![Figure 1. Illustration of mechanical load cycling simulator and specimen design. Loading device was allowed free movement transverse to loading direction.](image)

For debonding, the specimens were engaged at their cylinder bases with a shear blade in the UTS (crosshead speed: 1.0 mm/min) until bonding fracture took place [22]. SBS values (MPa) were calculated from the peak load divided by the bonding area. To determine the failure mode, the fractured zirconia
surfaces were observed under an optical microscope (SZ61, Olympus, Tokyo, Japan). Failures were classified as adhesive (A) when the fracture occurred completely between the resin and the zirconia, mixed (M) when the fracture continued into the resin and cohesive (C) if the fracture site was located exclusively within the resin. In addition, the debonded zirconia surfaces of the RelyX U200 specimens subjected to MLC were examined using scanning electron microscopy (SEM, JSM-6700F, Jeol, Tokyo, Japan) at 30× and 2000× magnification under a voltage of 10 kV.

2.4. Statistical Analysis

The flexural properties and surface energy parameters of the two resin cements were statistically analyzed using Student’s $t$ test ($\alpha = 0.05$). The SBS results were statistically analyzed using two-way analysis of variance (ANOVA) and post hoc analysis (Tukey or Student’s $t$ test). Weibull analysis was also applied to the SBS data to determine the shape and scale parameters of Weibull distribution.

3. Results

The flexural properties and surface energy parameters of the two resin cements are summarized in Table 2. Neither the flexural strengths nor moduli differed significantly between the two materials ($p > 0.05$). Their total surface energies were statistically similar ($p = 0.718$). On the other hand, the base component of Panavia F2.0 was significantly higher than that of RelyX U200 ($p = 0.006$). RelyX U200 was significantly more hydrophobic than Panavia F2.0 ($p = 0.004$).

Table 2. Flexural properties and surface energy parameters of the two resins ($n = 5$).

| Resins       | Flexural Properties | Surface Energy Parameters (mJm$^{-2}$) $^2$ |
|--------------|---------------------|--------------------------------------------|
|              | Flexural Strength (MPa) | Flexural Modulus (GPa) | $\gamma_s$ | $\gamma_s^{\text{LW}}$ | $\gamma_s^a$ | $\gamma_s^-\gamma_s^{\text{AB}}$ | $\Delta G_{\text{sws}}$ |
| Panavia F2.0 | 77.5 (9.4) $^1$ | 6.6 (0.3) $^a$ | 42.6 (2.1) $^a$ | 41.0 (1.7) $^a$ | 0.03 (0.01) $^a$ | 21.1 (1.9) $^a$ | 1.7 (0.4) $^a$ | -14.9 (3.3) $^a$ |
| RelyX U200   | 79.1 (7.5) $^a$ | 6.4 (0.4) $^a$ | 42.1 (2.7) $^a$ | 41.0 (2.8) $^a$ | 0.02 (0.01) $^a$ | 17.2 (0.9) $^b$ | 1.1 (0.3) $^b$ | -23.7 (3.7) $^b$ |

$^1$ Within the same column, the same lowercase superscript letters (a and b) show no significant difference between the two resin cements ($p > 0.05$). $^2 \gamma_s$: total surface energy, $\gamma_s^{\text{LW}}$: Lifshitz-van der Waals component, $\gamma_s^a$: acid component, $\gamma_s^-\gamma_s^{\text{AB}}$: base component, $\Delta G$: degree of hydrophobicity/hydrophilicity.

Table 3 summarizes the SBS results of the two resin cements bonded to the zirconia surfaces, together with the Weibull parameters and failure modes for each group. The Weibull cumulative failure probability curves for the SBS values are shown in Figure 2. In addition, Figure 3 shows the SEM images of the zirconia surfaces showing the mixed failure pattern after debonding. Both the aging method and resin cement used significantly affected the SBS, the two-way ANOVA also showing a significant interaction between the two variables ($p < 0.05$). Therefore, the data were analyzed with one-way ANOVA and the Student’s $t$ test. In this study, no significant difference in SBS was detected between the two materials before aging ($p = 0.133$). The SBS values for both resin cements slightly decreased after TC, but with no significant differences in the NA condition ($p > 0.05$). For both resins, the three MLC-containing aging conditions (MLC alone, TC/MLC, and MLC/TC groups) significantly decreased the SBS values when compared to the NA ($p < 0.05$). For Panavia F2.0, there were no significant differences among the MLC, TC/MLC, and MLC/TC groups ($p > 0.05$). Meanwhile, MLC aging of the RelyX U200 exhibited a significantly higher SBS value than did the TC/MLC and MLC/TC conditions ($p < 0.05$). However, the combinations of the two aging methods (either TC/MLC or MLC/TC groups) did not produce significantly different SBS values for either resin ($p > 0.05$). For both resins, the Weibull moduli of the MLC-containing aging groups significantly decreased when compared with the NA groups. For both cements, the failure modes were exclusively adhesive in the NA and TC groups. In contrast, the MLC aging-containing groups (MLC, TC/MLC, and MLC/TC groups) showed increased occurrence of mixed failure.
Table 3. Shear bond strength (in MPa) of the two resin cements to the zirconia surfaces, together with Weibull parameters and failure modes for each group (n = 15).

| Resins       | Aging 1 | Shear Bond Strength | Weibull Parameters 3 | Failure Mode 4 |
|--------------|---------|---------------------|----------------------|---------------|
|              | Mean (SD) | m (95% CI) | σ₀ | r² | σ₀,05 | A | M | C |
| Panavia F2.0 | NA      | 19.7 (4.3) aA²    | 4.9 (4.5–5.3) aA² | 21.4 | 0.98 | 11.7 | 15 | 0 | 0 |
|              | TC      | 17.2 (5.0) abA²   | 3.7 (3.3–4.0) bA² | 19.0 | 0.97 | 8.5 | 15 | 0 | 0 |
|              | MLC     | 13.4 (4.1) bCA²   | 3.3 (3.2–3.5) bA² | 14.9 | 0.99 | 6.1 | 11 | 4 | 0 |
|              | TC/MLC  | 12.7 (3.9) cA²    | 3.4 (3.2–3.7) bA² | 14.2 | 0.99 | 5.9 | 6  | 9 | 0 |
|              | MLC/TC  | 11.4 (3.3) cA²    | 3.6 (3.3–3.9) bA² | 12.7 | 0.98 | 5.5 | 7  | 8 | 0 |
| RelyX U200   | NA      | 22.0 (4.2) aA²    | 5.7 (5.3–6.1) aA² | 23.8 | 0.99 | 14.1 | 15 | 0 | 0 |
|              | TC      | 20.5 (4.5) aA²    | 4.9 (4.4–5.4) abB | 22.4 | 0.97 | 12.2 | 15 | 0 | 0 |
|              | MLC     | 16.6 (3.8) bB²    | 4.7 (4.4–5.0) bB² | 18.1 | 0.99 | 9.6  | 13 | 2 | 0 |
|              | TC/MLC  | 11.7 (3.1) cA²    | 4.1 (3.8–4.4) bcB | 12.8 | 0.98 | 6.2  | 9  | 6 | 0 |
|              | MLC/TC  | 10.2 (2.9) cA²    | 3.9 (3.4–3.4) cA² | 11.3 | 0.97 | 5.3  | 8  | 7 | 0 |

1 NA: non-aged; TC: thermal cycling; MLC: mechanical load cycling; TC/MLC: TC then MLC; MLC/TC: MLC then TC. For each cement, the same lowercase superscript letters (a, b, and c) within the same column show no significant differences among the aging conditions (p > 0.05). For each aging method, the same uppercase superscript letters (A and B) within the same column show no significant differences between the two resin cements used (p > 0.05). 3 m: shape parameter was considered to be significantly different when the CI (confidence interval) values did not overlap. σ₀: scale parameter (in MPa); r²: correlation coefficient; σ₀,05: probability of failure at 5% (in MPa). 4 A: adhesive failure; M: mixed failure; C: cohesive failure.

Figure 2. Weibull cumulative failure probability curves for the shear bond strength (SBS) values: (a) Panavia F2.0 and (b) RelyX U200.

Figure 3. Representative SEM images of the fracture mode (RelyX U200) (magnification: upper: 30x; lower 2000x): (a) and (d): mechanical load cycling (MLC); (b) and (e): thermal cycling (TC)/MLC; (c) and (f): MLC/TC.
4. Discussion

Storage in water and TC of resin-bonded specimens are the most frequently used artificial aging methods [9]. Although TC is an effective accelerated aging protocol for evaluation of bonding durability [23], the extent to which it practically mimics the real clinical situation has been questioned [10]. TC tests cannot discriminate which type of failure causes the degradation of resin bond to ceramic [10]. While there is no definite evidence that thermal stresses cause failures, every TC test should be considered arbitrary [10]. This in vitro study thus evaluated the effectiveness of the MLC aging method, alone or combined with TC, in challenging the resin-zirconia interface.

As MLC was applied to the resin materials in a wet environment to simulate intraoral condition (Figure 1), the surface energy parameters, including degree of hydrophobicity/hydrophilicity, as well as the flexural properties of the resin cements bonded to zirconia were studied prior to the SBS test. The flexural property results suggest that the resin materials behave similarly when subjected to MLC in dry condition. The parameter \( \Delta G_{\text{sws}} \) indicates whether the polar attraction of solid to water is greater or smaller than the polar attraction of water molecules to each other and can thus serve as a more appropriate measure than water CA itself [24]. In the context of Lifshitz-van der Waals (van Oss and Good) theory, the base components of cements may significantly affect their bonding to zirconia [3]. In this study, notwithstanding a significant difference in the base components between the two resin cements (Table 2), no significant difference in SBS was found between the two materials before aging (Table 3). This may have been due to only a small difference in the base components of the materials.

During aging of a resin-bonded zirconia specimen in water, water may diffuse into the resin as well as into the adhesive interface [25]. Water sorption may cause hydrothermal degradation, such as the creation of swelling stresses, of a resin bonded to a zirconia [26]. Thus, the stability of the bonding interface may depend on the hydrophobic/hydrophilic nature of the resins used [25]. When more hydrophobic resins are used, greater stability is supposed, as water would not be able to penetrate the bonding interface so easily [25].

In TC, several factors such as temperature setting, dwell time, and number of cycles can affect the bond strength test results [23]. As regards the number of cycles, ISO 10477 prescribes 5000 cycles [27], which seem insufficient for estimating the long term bonding durability [10]. In this study, the SBS values after TC was not significantly different from those in the NA condition for both resins (Table 3), probably because the specimens were not water-saturated prior to TC and the number of TC was not high enough to age the resin bonds [8,28].

Unlike TC, the use of MLC, which simulates mastication, applies a direct load to a bonded specimen [29]. A force of 50 N was chosen to simulate an average of constant load found during mastication [21,29,30]. For both resin materials, the MLC-containing aging conditions significantly decreased the SBS values when compared to the NA (Table 3). However, the tendency was not the same between the two resin materials. In this study, the only significant difference in SBS between the two resins was found for the MLC-only aging condition. MLC aging of the RelyX U200 exhibited a significantly higher SBS than did the other MLC-containing aging conditions, probably due to the significantly higher hydrophobicity of the resin material (Table 2).

The SEM images of the zirconia surfaces after debonding (Figure 3) suggest the application of MLC to the resin-bonded specimens caused crack formation within the resin materials as well as at the resin-zirconia interface. For both cements, thus, the Weibull moduli of the three MLC groups significantly decreased when compared with the NA groups. When a resin-bonded specimen is subjected to aging in water, the resin may become less stiff over time due to water sorption and entrapped water droplets within the resin layer, making the material less stiff, resulting in impaired stress distribution across the luted interfaces to zirconia and a decrease in bond strength at the interface [26,31]. These degradation phenomena would be more effectively accelerated by the application of MLC rather than TC. Thus, the use of MLC combined with TC may more closely
simulate intraoral conditions, in which restored teeth are subjected to masticatory load as well as thermal changes.

5. Conclusions

The null hypothesis that the aging methods do not differently decrease the SBS of the resin cements to zirconia was partially rejected. For both resin cements, MLC significantly reduced the values by directly damaging the resin-zirconia interfaces. The SBS results for the MLC-only aging condition were also related to the hydrophobic/hydrophilic nature of the resin cements bonded to zirconia. However, the mechanism of the failure induced by MLC requires further investigation. In addition, a more standardized MLC testing protocol which more closely simulates loading on a material in the oral cavity should be established.

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