

## Bond strength of self-adhesive resin cements to dry and moist dentin

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**Abstract:** This study evaluated the effects of humidity conditions and evaluation times on the dentin bond strength (DBS) of two self-adhesive resin cements (RC). The RC used were: RelyX Unicem (3M ESPE) and Clearfil SA Cement (Kuraray Med.). One hundred and twenty coronal portions of bovine incisors ( $n = 10$ ) were used. Buccal surfaces were abraded in order to expose a flat dentinal surface (180-grit SiC) and to standardize the smear layer formation (600-grit SiC). The humidity conditions tested were: dry (air-dried for 10 s), slightly moist (water application with disposable applicator on dried dentin and water excess removed with absorbent paper), and moist (same application without water removal). The RC were used according to the manufacturers' recommendations and were applied to prepolymerized resin discs (2 mm thick; Sinfony, 3M ESPE), which were subsequently bonded to the dentin surfaces. After 24 h, half of the teeth were prepared for the microtensile bond strength test, while the other half were stored in water for 6 months and tested in tension (0.5 mm/min) until failure. A 3-way analysis of variance and the Tukey test were performed (preset alpha of 0.05). No RC showed any reduction of DBS after 6 months, and no significant difference was observed between them. The moist dentin increased the bond strength of Clearfil SA Cement for both periods of time. Humidity conditions can change the DBS; however, the study's results were product-dependent.

**Descriptors:** Dentin; Tensile Strength; Resin Cements; Humidity.

### Introduction

The self-adhesive resin cements do not require acid conditioning of the tooth structure and application of the bonding agent before cementation. These luting materials simplify the cementation procedures, because they save time and reduce the number of steps for cementation of indirect restorations and posts, when compared to conventional resin cements.<sup>1-3</sup>

The bonding to tooth structures is promoted by specific functional monomers, which differ among different commercial products. According to the manufacturers, the functional monomers are able to bond chemically the calcium from hydroxyapatite, which is one of the bonding mechanisms responsible for the retention of the restoration.<sup>4-6</sup> However, little information is available about these chemical reactions, and the durability of this bonding requires further investigation, since clinicians have switched from conventional resin cement to these new self-adhesive resin cements.<sup>7-9</sup>

**Declaration of Interests:** The authors certify that they have no commercial or associative interest that represents a conflict of interest in connection with the manuscript.

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Submitted: Apr 07, 2013  
Accepted for publication: Jun 18, 2013  
Last revision: Jul 12, 2013

The manufacturers' instructions provide information regarding the proper use of these materials; however, some of these are unclear regarding the humidity conditions of mineralized tissues (enamel and dentin) before cementation. The purpose of this *in vitro* study was to measure the bond strength of pre-polymerized composite discs to underlying dentin, using two self-adhesive resin-based cements under three humidity conditions. In addition, the bond failure site morphology was analyzed and compared among material types and humidity conditions. The research hypothesis tested was that the bond strength would be significantly higher when the resin cement is applied to wet dentin than when it is used on dried surfaces.

## Methodology

Two self-adhesive resin cements were selected:

- RelyX Unicem (3M ESPE, St. Paul, USA) and
- Clearfil SA Cement (Kuraray Noritake Dental Inc., Kurashiki, Japan).

The composition and the batch number are presented in Table 1. One hundred and twenty freshly extracted incisor bovine teeth (stored in a 0.05% thymol [LabSynth, Diadema, Brazil] solution at 5°C) were used and randomly divided into four groups (n = 10; Institutional Review Board protocol #089/2009). Their buccal surfaces were wet and abraded with 180-grit silicon carbide paper (Carborundum Abrasivos, Vinhedo, Brazil) using a machine (APL-4, Arotec Ind. e Com. Ltda., Cotia, Brazil) to remove the enamel and to expose a flat dentin surface with a remaining dentin thickness ranging from 1.0 to 1.5 mm. Afterwards, the teeth were abraded with 600-grit silicon carbide paper (Carborundum Abrasivos, Vinhedo, Brazil) for 10 s to standardize the smear layer formation. The humidity conditions tested were:

1. Dry: air-dried for 10 s, 70 psi and a distance of 10 mm between the dentin surface and the tip of the air-syringe (Dabi Atlante, Ribeirão Preto, Brazil).
2. Slightly moist: water application with a Microbrush disposable applicator (Microbrush International, Grafton, USA) on dried dentin, and water

**Table 1** - Composition and lot number of the materials tested in this study.

Resin cement	Composition (lot number)
Clearfil SA Cement	Bis-GMA, TEGDMA, sodium fluoride, 10-methacryloyloxydecyl dihydrogen phosphate, hydrophobic aromatic dimethacrylate, hydrophobic aliphatic dimethacrylate, silanated barium glass filler, silanated colloidal silica, dl-camphorquinone, initiators, accelerators, catalysts, pigments (filler = 66 wt%; 45 vol%; avg. 2.5 µm) (0004AB)
RelyX Unicem	Methacrylated phosphoric acid esters, triethylene glycol dimethacrylate, substituted dimethacrylate, silanized glass powder, silane treated silica, sodium persulfate, substituted pyrimidine, calcium hydroxide (filler = 72 wt%; avg. < 9.5 µm) (365945)

excess removed with absorbent paper (Kleenex, Kimberly-Clark, Mogi das Cruzes, Brazil).

3. Moist: the same application of condition 2 without the removal of water.

One hundred and twenty pre-polymerized (B2D shade; Sinfony, 3M ESPE, St. Paul, USA), light-cured composite resin discs, 2 mm thick and 10 mm in diameter, were prepared to simulate overlying laboratory-processed composite resin restorations. The surface of each disc to be bonded to the prepared tooth was airborne-particle abraded with 50 µm aluminum oxide (Danville Engineering Inc., San Ramon, USA) for 10 s (air pressure: 0.552 MPa; distance from the tip: 1.5 cm), and silanated using coupling agents (Ceramic Primer, 3M ESPE, St. Paul, USA or Clearfil Ceramic Primer, Kuraray Noritake Dental Inc., Kurashiki, Japan), according to the manufacturer's directions.<sup>10</sup>

All cements were manipulated and applied according to the manufacturers' instructions. The mixed resin cement pastes were applied to the sandblasted and silanated surface of the pre-polymerized composite resin disc, after which the disc was placed on the dentin surface. A 500 g load was applied for 5 min. For light-polymerized groups, the load was removed and the light-activating tip (XL 3000, 3M ESPE, St. Paul, USA) was positioned against the composite resin disc after loading, and the unit was activated for 40 s. To facilitate the specimen-gripping length while bond testing was being performed,

**Table 2** - Mean (standard deviation) bond strength (MPa) of resin cements to dentin.

Resin cement	Evaluation time	Humidity conditions		
		Dry	Slightly moist	Moist
Clearfil SA Cement	24 hours	12.7 (6.0) <sup>B</sup>	15.7 (6.3) <sup>AB</sup>	19.0 (8.4) <sup>A</sup>
RelyX Unicem		18.9 (5.8) <sup>A</sup>	19.3 (3.6) <sup>A</sup>	19.7 (2.8) <sup>A</sup>
Clearfil SA Cement	6 months	14.2 (6.2) <sup>B</sup>	17.8 (7.0) <sup>AB</sup>	22.1 (4.6) <sup>A</sup>
RelyX Unicem		18.3 (6.7) <sup>A</sup>	15.7 (2.9) <sup>A</sup>	16.5 (3.6) <sup>A</sup>

No significant difference ( $p > 0.05$ ) in bond strength was noted between resin cements and evaluation times (uppercase letters = row).

a 3-mm-thick block of autopolymerizing composite resin (Concise, 3M of Brazil, Sumaré, Brazil) was then added to the untreated, pre-polymerized composite resin surface.

The teeth were stored in distilled, deionized water at 37°C for 24 h, and were then vertically, serially sectioned into several 1.0-mm-thick slabs using a cutting instrument (Isomet 1000, Buehler Ltd., Lake Bluff, USA). Each slab was further sectioned perpendicularly to produce bonded sticks approximately 1.0 mm<sup>2</sup> in cross-section. Each bonded stick was attached to the grips of a microtensile testing device (Cometa, Piracicaba, Brazil) with cyanoacrylate glue (Super Bonder, Henkel/Loctite, Diadema, Brazil), and tested in tension in a universal testing machine (EZ Test, Shimazu, Kyoto, Japan) at a crosshead speed of 0.5 mm/min until failure. After testing, the specimens were removed carefully, and the cross-sectional area at the site of fracture was measured to the nearest 0.01 mm with a digital caliper (727-6/150, Starret Ind. e Com. Ltda., Itu, Brazil). The specimen's cross-sectional area was divided by the peak tensile load at failure to calculate the stress at fracture (MPa). A single failure stress value was then calculated for each tooth by averaging the values of 6 sticks from that tooth. A 3-way analysis of variance (ANOVA) (factors: material, humidity and time) was performed to determine the effect of these major factors on and their interaction with tensile strength. The Tukey-Kramer test was used to detect multiple comparisons among the experimental groups. All statistical testing (SAS Institute Inc., Cary, USA) was performed at a preset alpha of 0.05.

Fractured surfaces of tested specimens were allowed to air-dry (Marconi Equip. Lab., Piracicaba, Brazil) overnight at 37°C, after which they were

sputter-coated with gold (MED 010, Balzers, Balzer, Liechtenstein) and examined by a single individual using a scanning electron microscope (voltage 15 kV; VP 435, Leo, Cambridge, UK). Failure patterns were classified as:

1. cohesive within the resin cement;
2. adhesive along the pre-polymerized composite overlay–resin cement interface;
3. adhesive along the dentin surface;
4. mixed when simultaneously exhibiting the dentin surface and remnants of the resin cement; and
5. cohesive within the dentin.

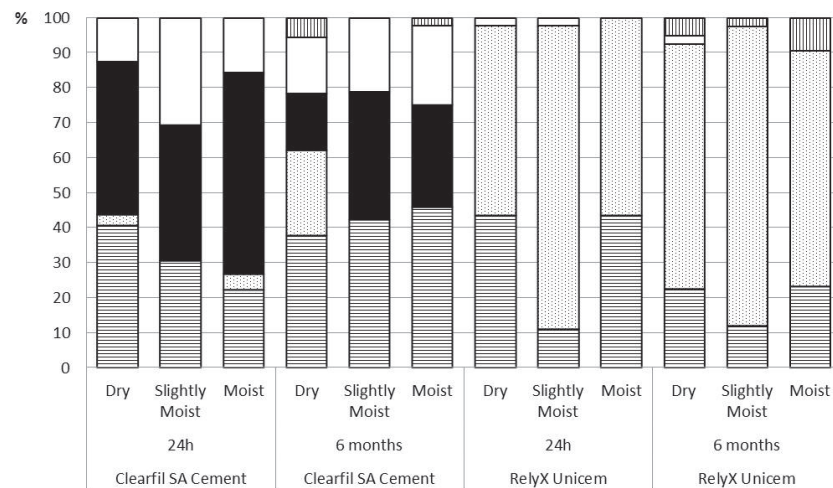
Representative areas of the failure patterns were photographed at 400×

## Results

The mean bond strength values are presented in Table 2. Three-way ANOVA revealed statistically significant differences only for the factor, “humidity” ( $p = 0.0475$ ). Conversely, the statistical analysis revealed no significant differences for the “material” ( $p = 0.3309$ ) and “time” ( $p = 0.8859$ ) factors. The double interactions between “material and humidity” and “material and time” factors were significant ( $p = 0.0384$  and  $p = 0.0072$ , respectively). The triple interaction was not statistically significant ( $p = 0.5440$ ).

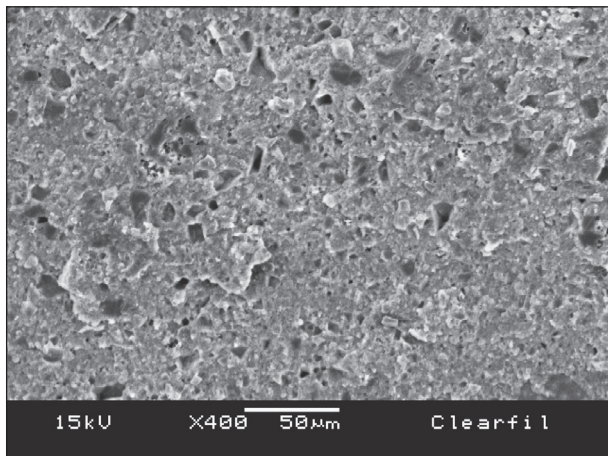
The Tukey-Kramer test revealed significant differences among humidity conditions only for Clearfil SA Cement ( $p < 0.05$ ). Analysis of data with respect to these differences in the humidity condition showed that the Clearfil SA Cement had a significantly lower bond strength when applied to the dried dentin surface than when applied to moist dentin ( $p < 0.05$ ). However, the dry and slightly moist

**Figure 1** - Distribution of failure modes among experimental groups.

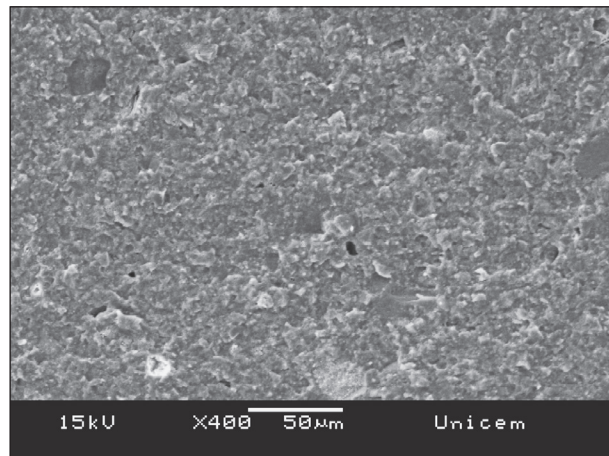


Legends:

- Type 1: Cohesive failure within the cement.
- Type 2: Adhesive failure along the pre-polymerized composite overlay-resin cement interface.
- Type 3: Adhesive failure along the dentin surface.
- Type 4: Mixed failure.
- Type 5: Cohesive failure within the dentin.



**Figure 2** - Cohesive failure within the Clearfil SA Cement (type 1; original magnification 400X).

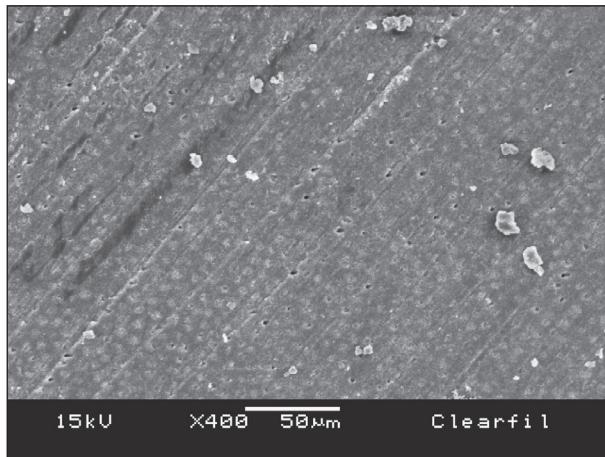


**Figure 3** - Adhesive failure along the pre-polymerized composite overlay interface showing little signs of the RelyX Unicem cement (type 2; original magnification 400X).

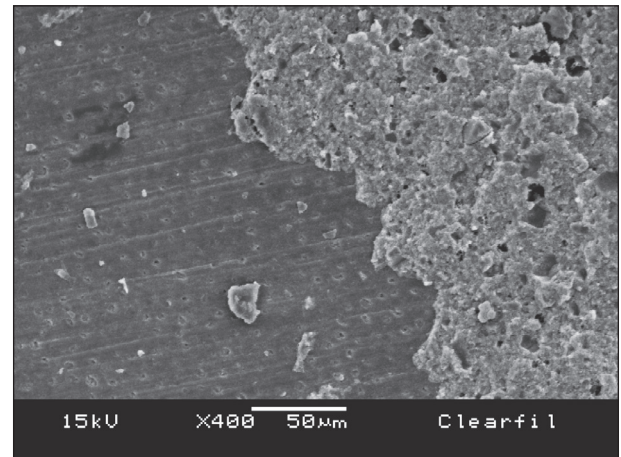
conditions did not differ from each other ( $p > 0.05$ ). The storage time did not influence the bond strength results ( $p > 0.05$ ).

Figure 1 shows the proportional prevalence (%) of the failure patterns in all experimental groups. Representative images depicting the failure classifications are presented in Figures 2–6. All groups showed cohesive failure within the resin cement (type 1; Figure 2). RelyX Unicem presented a high incidence of adhesive

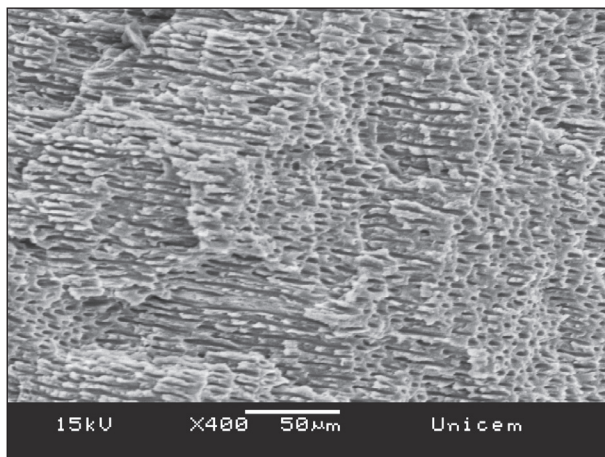
failure along the pre-polymerized composite overlay-resin cement interface (type 2; Figure 3), whereas adhesive failures along the dentin surface (type 3) and mixed fracture (type 4) were observed for Clearfil SA Cement (Figures 4 and 5, respectively) at both evaluation times. Cohesive failures within the dentin (type 5) were observed for both luting materials only after the storage of specimens for 6 months (Figure 6).



**Figure 4** - Adhesive failure along the dentin surface (type 3) with absence of the self-adhesive resin (Clearfil SA Cement) over the dentin surface (original magnification 400×).



**Figure 5** - Mixed failure mode (type 4) showing the dentin surface and remnants of Clearfil SA resin cement (original magnification 400×).



**Figure 6** - Cohesive failure within the dentin (type 5) after 6 months of water storage with RelyX Unicem (original magnification 400×).

## Discussion

The functional monomer of RelyX Unicem is methacrylated phosphoric ester, which reacts chemically with the hydroxyapatite, promoting the bonding to the dental structures and with filler particles that are the basic components of this self-adhesive resin cement. This second acid-base reaction is important in neutralizing the acidic characteristic of the resin cement and reducing the hydrophilicity after mixing of the catalyst and base pastes.<sup>3,11-13</sup> The bond strength of RelyX Unicem to dentin obtained in this study ranged from  $15.7 \pm 2.9$  MPa to

$19.7 \pm 2.8$  MPa. These values were higher than those obtained by Piwowarczyk *et al.*<sup>14</sup> (6.2 MPa), Yang *et al.*<sup>15</sup> (8.2 MPa), Goracci *et al.*<sup>16</sup> (6.8 MPa), Holderegger *et al.*<sup>17</sup> (9.2 MPa), and Egilmez *et al.*<sup>18</sup> (13 MPa). On the other hand, similar results were found by De Munck *et al.*,<sup>11</sup> Sarr *et al.*,<sup>19</sup> Luhrs *et al.*,<sup>20</sup> Mazzitelli *et al.*,<sup>21</sup> Ebert *et al.*,<sup>22</sup> and Inukai *et al.*<sup>23</sup>

Although the hydrated dentin substrate can facilitate ionization of the acid monomers, the results of this study suggested that the humidity conditions did not affect the bond strength of RelyX Unicem to dentin, i.e., no significant difference was observed when the self-adhesive was applied to surfaces with different humidity conditions. Conversely, when Guarda *et al.*<sup>24</sup> did not over-dry the dentin surface before cementation, a higher bond strength was observed for RelyX Unicem. Moreover, Mazzitelli *et al.*<sup>21</sup> found that RelyX Unicem performed better under simulated pulpar pressure, because the constant intrapulpar water perfusion changes the substrate wetness.

The functional monomer of Clearfil SA Cement is the 10-methacryloyloxydecyl dihydrogen phosphate (10-MDP), which is able to form a strong and stable ionic bond with calcium from hydroxyapatite.<sup>25</sup> Besides this chemical interaction,<sup>26</sup> Clearfil SA Cement can also provide micromechanical retention, since its monomer is capable of infiltrating the dentinal substrate.<sup>27,28</sup> The wetness of dentin was

important for increasing the bond strength of this resin cement. In the presence of water, the acidic monomer of Clearfil SA resin cement was ionized, acid-etched the dentin, and interacted with the dentinal substrate. Thus, the extrinsic dentinal wetness may have optimized these acid-base reactions, allowing for better setting and bonding.

In comparison with RelyX Unicem, few studies have investigated the bond strength of Clearfil SA Cement to dentin. In this study, the bond strength results for Clearfil SA Cement ranged from  $12.7 \pm 6.0$  MPa in dry dentin to  $22.1 \pm 4.6$  MPa in moist dentin, which were significantly different. The bond strength results for Clearfil SA Cement from Ebert *et al.*,<sup>22</sup> Inukai *et al.*,<sup>23</sup> and Ilday *et al.*<sup>29</sup> corroborated our data, while Egilmez *et al.*<sup>18</sup> showed lower bond strength values than those obtained in this study.

The self-adhesive resin cements (RelyX Unicem and Clearfil SA Cement) tested in this study showed no significant difference between materials in terms of bond strength. Although they present different compositions, these commercial formulations did not influence the bonding to dentin. In addition, the storage in water for 6 months did not alter the bond strength to dentin for any of the resin cements tested, independent of the humidity conditions of dentin. The chemical reaction between 10-MDP and calcium from hydroxyapatite is considerably stable,<sup>25-28</sup> which explains the absence of bond strength reduction for Clearfil SA Cement. The setting reaction of RelyX Unicem increases the pH value, changing the

monomer nature from hydrophilic to hydrophobic, and this neutralization reaction is important for the long-term stability of RelyX Unicem cement regarding bond strength to dentin.<sup>3,11-13</sup>

Although no change was observed in bond strength, the failure pattern was modified after storage of the bonded beam specimens for 6 months in water. The specimens stored in water for 6 months induced some cohesive failures within dentin, which did not occur for the specimens stored for 24 hours. The storage of bonded beams instead of restored teeth and without a peripheral composite-enamel bond<sup>30</sup> seemed to accelerate the degradation rate of the exposed dentin, resulting in cohesive failures within dentin during the tensile test.

## Conclusion

The storage in water for 6 months does not decrease the bond strength of self-adhesive resin cements to dentin. Conversely, the humidity conditions can change the bond strength to dentin; however, these results are product-dependent.

## Acknowledgements

This study was supported by grants from PIBIC/CNPq (*Programa Institucional de Bolsas de Iniciação Científica/Conselho Nacional de Desenvolvimento Científico e Tecnológico*) and FAPESP (*Fundação de Amparo à Pesquisa do Estado de São Paulo*), processes nos. 2009/51674-6 and 2010/13599-0, Brazil.

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