Influence of curing mode of resin luting cements on bond strength to dentin

Andreia Assis Carvalho¹, Anivaldo Stefan², Vladímir Franco de Sá Barbosa³, Lawrence Gonzaga Lopes⁴, Marcelo Giannini⁵

1Mestre em Clínica Odontológica, Universidade Federal de Goiás - UFG, Department of Prevention and Oral Rehabilitation, Goiânia, Goiás, Brazil, Address: Department of Prevention and Oral Rehabilitation - School of Dentistry, Federal University of Goiás, Praça Universitária, s/n, Faculdade de Odontologia, Setor Universitário, Goiânia-GO, Brazil, CEP 74605220
2Mestre em Prótese, São Leopoldo Mandic - SLMANDIC, Department of Prosthesis, Campinas, SP, Brazil, Address: Rua Borges Lagoa, 1065, cj 139, Vila Clementino, São Paulo, SP, Brazil. CEP 04038-032
3Mestre em Clínica Odontológica, Universidade do Estado do Amazonas - UEA, Department of Dentistry, Manaus, AM, Brazil, Address: State University of Amazonas, School of Health Sciences, Odontologia, Av. Carvalho Leal, 1777, Cachoeirinha, Manaus, AM, Brazil. CEP 69065-001
4Doutor e Mestre em Dentística, Universidade Federal de Goiás - UFG, Department of Prevention and Oral Rehabilitation, Goiânia, Goiás, Brazil, Address: Department of Prevention and Oral Rehabilitation - School of Dentistry, Federal University of Goiás - Praça Universitária, s/n, Faculdade de Odontologia, Setor Universitário, Goiânia-GO, Brazil, CEP 74605220
5Doutor e Mestre em Clínica Odontológica, Universidade Estadual de Campinas - UNICAMP, Associate Professor, Department of Restorative Dentistry, Piracicaba Dental School, Piracicaba, SP, Brazil, Address: Department of Restorative Dentistry - Piracicaba Dental School - Campinas State University - Av. Limeira, 901 - Piracicaba, SP, Brazil, CEP 13414-903018

Abstract

Self-adhesive, dual-polymerizing resin cements require no treatment to the prepared tooth surfaces before cementation. Aim: The aim of this study was to evaluate the influence of curing mode on bond strength (BS) of three cementing systems to bovine dentin. Methods: The buccal enamel surfaces of 50 bovine incisors were removed to expose dentin and to flat the surface. The teeth were divided into five groups (n=10), which consisted of two resin cements (Multilink and Clearfil SA Cement) that were tested in dual- (halogen light for 40 s) and self-cured modes, and a control (RelyX ARC). Two cylinders of resin cements (1.0 mm X 0.75 mm) were prepared on each bonded dentin surface. After 24h at 37oC, resin cylinders were subjected to micro-shear testing in a universal testing machine (4411/Instron - 0.5 mm/min). Data were statistically analyzed by two-way ANOVA, Tukey and Dunnett’s test (5%). Results: Multilink showed higher BS than those observed on Clearfil SA. Light-curing resulted in higher BS for both Multilink and Clearfil SA. When Multilink was light-cured, no significant difference on BS was demonstrated between it and RelyX ARC. Conclusions: The highest BS values were obtained in control group and light-cured Multilink resin cement.

Keywords: Tensile Strength. Adhesives. Dentin.

Introduction

Resin cements (RC) are low viscous composite resins used to fix indirect restorations and to promote sealing between restoration and tooth substrates. The longevity of indirect restorations is directly connected with the effectiveness of the bond between dental tissues and RC. RC can be classified as light-cured, self/auto-cured or dual-cured based on polymerization mode, without considering the dentin pretreatment method. RC are produced in dual-polymerized formulations, which are indicated for restorations with...
high opacity or sufficient thickness to inhibit light energy from tip of curing unit to the RC or other clinical scenarios when the light is significantly attenuated.

According to the treatment of dental tissue, the RC are classified: total-etch, self-etch or self-adhesive. At present, all RC are based on the use of either an etch-and-rise or of a self-adhesive, however, application in stages technology is complex and considerably technology sensitive and thus may affect bonding efficiency. The new self-adhesive, dual-polymerizing RC require no separate acid etching, priming or bonding resin applications to the prepared tooth surfaces before. However, little is known about the effectiveness of these new cements regarding the bonding to mineralized dental tissues when light-cured or with no curing light exposure. The purpose of this study was to evaluate the influence of curing mode on bond strength of RC to bovine dentin, using two resin cements that were either light polymerized or allowed to autopolymerize in the absence of light. Furthermore, the site morphology of bond failures was analyzed to classify them according to the fracture mode. The null hypotheses tested were that light-curing has no effect in bond strength values and no experimental group would reach the BS of control group.

Materials and methods

The materials (cementing systems: resin cement and bonding agent) used in this study and their compositions are described in Table 1. The RelyX ARC (3M ESPE, St. Paul, MN, USA) that is dual-cured RC was used in combination with a three-steps etch-and-rise adhesive (Scotchbond Multipurpose, 3M ESPE, St. Paul, MN, USA) and considered the control in this study. Multilink is a dual-cured RC used in combination with a self-etching primer (mixed Multilink Primer A/B) (Ivoclar Vivadent, Schaan, Liechtenstein). Clearfil SA Cement (Kuraray Noritake Dental Inc., Kurashiki, Japan) is dual-cured self-adhesive RC, which does not require a previous bonding agent application.

Fifty freshly extracted bovine incisors stored at 6oC were used in this study. The roots and labial enamel were removed using a diamond disk (Isomet, Buehler, Evanstone, IL, USA) under water-cooling. The dentin surfaces were wet-abraded with 600-grit SiC paper (Carborandum, Vinhedo, SP, Brazil) to expose a flat dentin. Teeth were randomly divided into five groups (n = 10):

- Group 1: Primer A and B / Multilink (dual-cured mode / light- and self-curing)
- Group 2: Primer A and B / Multilink (only self-cured mode)
- Group 3: Clearfil SA Cement (dual-cured mode / light- and self-curing)
- Group 4: Clearfil SA Cement (only self-cured mode)
- Control: Scotchbond Multipurpose / RelyX ARC (regular use with light-activation of adhesive and resin cement).

The RC and bonding agents were applied according to the manufacturer’s instructions and all light-curing procedures were performed with the Optilux 500 (Demetron, Kerr, Orange, CA, USA) light-curing unit (650 mW/cm²). The methodology developed by Shimada et al. (2002) was used to prepare specimens for the micro-shear test. Two cylindrical translucent molds (Tytong tubing, TYG-030, Saint-Gobain Performance Plastics, Aurora, OH, USA) were positioned over dentin and freshly mixed dual-cure RC (Multilink, Clearfil SA Cement or RelyX ARC) were placed in the molds to fill their internal volume and light activated for 40 s or not, depending on the experimental group.

Teeth were stored in 100% humid environment at 37°C for 24h. The tube molds were removed to expose the RC cylinders (1.0 mm high by 0.75 mm diameter) bonded to the dentin surface, thus, two bonded small RC cylinders were obtained for each dental fragment. Prior to testing, all resin cements were checked under stereomicroscope (30x, Meiji Techno Co. LTD., Saitama, Japan) for bonding defects and RC flashing.

Each tooth was attached to the testing device with cyanoacrylate glue (Zapit, DVA, Corona, CA, USA) and tested in a universal testing machine (EZ-test 500, Shimadzu Co, Tokyo, Japan). A shear load was applied at the base of the RC cylinder with a thin wire (0.20 mm diameter) at a crosshead speed of 1.0 mm/min until failure. The shear bond strength were calculated and expressed in MPa. A bond strength value was then calculated for each tooth by averaging the values of two cylinders from that tooth. Exploratory analysis of data regarding normality was
performed by Kolmogorov-Smirnov test. Bond strength data were analyzed statistically by two-way (cement X activation mode) analysis of variance (ANOVA), Tukey and Dunnett’s tests at the 5% level of significance.

After testing, the teeth were mounted on aluminum stubs, gold-sputter coated (SCD 050, Baltec, Vaduz, Liechtenstein) and observed under high vacuum with a scanning electron microscope (JSM-5600LV, Jeol, Tokyo, Japan). Photomicrographs of representative areas of the fractured surfaces were taken at 100X magnification for fracture pattern evaluation, which was classified as (1) adhesive along the dentin, (2) cohesive within the RC and (3) mixed when simultaneously exhibiting dentin surface, remnants of RC and/or adhesive and cohesive failure within the dentin.

Results

The bond strength means (and standard deviation) of the five study groups are shown in Table 2. The Tukey’s test for multiple comparisons among four experimental groups (excluding the RelyX ARC control group) showed that the highest dentin bond strength was obtained when Multilink was light-cured ($p < 0.05$). The bond strengths of Multilink RC were always higher than those obtained with Clearfil SA Cement, independent on the cure mode tested ($p < 0.05$). The light activation improved the dentin bond strength for both RC ($p < 0.05$) (Table 3). Dunnett’s test compared control and experimental groups and indicated that only the light-activated Multilink group did not differ statistically from the control ($p = 0.836$) (Table 4).

### Table 2 - The bond strengths means (and standard deviation) in MPa for the resin cements tested.

<table>
<thead>
<tr>
<th>Group</th>
<th>mean</th>
<th>sd</th>
</tr>
</thead>
<tbody>
<tr>
<td>Multilink Light-cured</td>
<td>29.5 A</td>
<td>7.9</td>
</tr>
<tr>
<td>Multilink Self-cured</td>
<td>21.3 B</td>
<td>6.4</td>
</tr>
<tr>
<td>Clearfil Light-cured</td>
<td>10.8 C</td>
<td>3.1</td>
</tr>
<tr>
<td>Clearfil Self-cured</td>
<td>3.0 D</td>
<td>3.9</td>
</tr>
<tr>
<td>Rely X F Light-cured (control)</td>
<td>31.5</td>
<td>5.5</td>
</tr>
</tbody>
</table>

* Significant difference from the control by Dunnett’s test ($p < 0.05$).

### Table 3 - Tukey’s test for multiple comparisons among experimental groups, except for RelyX ARC.

<table>
<thead>
<tr>
<th>Comparison</th>
<th>$p$</th>
</tr>
</thead>
<tbody>
<tr>
<td>Multilink Light-cured x Multilink Self-cured</td>
<td>0.014 *</td>
</tr>
<tr>
<td>Multilink Light-cured x Clearfil Light-cured</td>
<td>&lt;0.001 *</td>
</tr>
<tr>
<td>Multilink Light-cured x Clearfil Self-cured</td>
<td>&lt;0.001 *</td>
</tr>
<tr>
<td>Multilink Self-cured x Clearfil Light-cured</td>
<td>0.001 *</td>
</tr>
<tr>
<td>Multilink Self-cured x Clearfil Self-cured</td>
<td>&lt;0.001 *</td>
</tr>
<tr>
<td>Clearfil Light-cured x Clearfil Self-cured</td>
<td>0.021 *</td>
</tr>
</tbody>
</table>

* statistically significant difference ($p < 0.05$).

Scanning electron microscopy (SEM) examination of fractured interfaces showed variations among groups. Multilink produced mixed fractures, which involved adhesive failure along dentin surface, remnants of RC and/or adhesive and cohesive failure within the dentin (Figures 1 and 2). For Clearfil SA Cement, cohesive fractures within RC were predominant when the materials were not light exposed (Figure 3), while the adhesive failures along the dentin were seen when light-cured (Figure 4). Adhesive failures were predominant for RelyX ARC (Figure 5).
Influence of curing mode of resin luting cements on bond strength to dentin

Discussion

Cementation procedure plays a significant role in the clinical success of indirect restorations\textsuperscript{5}. The clinical longevity of these restorations is dependent on the cavity design, the indirect restorative material, the properties of the RC and cementation technique selected\textsuperscript{6}. The most common tests for resin-ceramic bonding measurements are shear and tensile bond strength tests\textsuperscript{3}. Twenty years ago, shear and tensile BS tests were performed in specimens with relatively large bonded areas, approximately 7-28 mm\textsuperscript{2}, what was questioned due to the heterogeneity of the bonded surfaces and the stress distribution at the bonded interface during the testing\textsuperscript{10,11}. To overcome the non-uniform stress created in conventional shear and tensile BS tests, microshear and microtensile bond tests were developed, and these tests used specimens with a 1 mm\textsuperscript{2} cross-sectional area of approximately\textsuperscript{3}. The micro-tensile and micro-shear bond strength tests were developed by Sano et al.\textsuperscript{12} (1994) and Shimada et al.\textsuperscript{7} (2002), respectively, in attempt to improve the quality of the BS measurements.

The new self-adhesives, dual-polymerizing RC were designed to simplify the cementation procedures\textsuperscript{13}. These cements have appeared on the market with a proposal to simplify the adhesive bonding technique, with the use of cement in one step\textsuperscript{14}, eliminating the demand for pretreatment of the tooth and the restoration\textsuperscript{5}. Clearfil SA Cement and Multilink resin cements are considered simplified RC, since the Clearfil SA Cement is one-step self-adhesive cement and the Multilink is applied in an association with a self-etching adhesive. Also, they are less technique-sensitive and time consuming when compared with multi-step RC\textsuperscript{15}. The bonding effectiveness to dentin of these RL is dependent on quality of functional monomers from resin monomer for Clearfil SA Cement or self-etching adhesive for Multilink. The functional adhesive monomer of Clearfil SA Cement is the 10-methacryloyloxydecyl dihydrogen phosphate (10-MDP). MDP can modify the smear layer and result in demineralization of dentin\textsuperscript{16}, however, in this study the self-adhesive resin monomer containing 10-MDP showed lower bond strength than Multilink and RelyX ARC cementing systems. Most of experimental groups using self-adhesive systems presented bond strength values significantly lower than those observed in the control. Other studies have also shown superior bond strength measurements for the conventional RC in comparison to the self-adhesive resin cements\textsuperscript{4,6,17}.

Multilink resin cement was placed over primed dentin (Multilink Primer A + B), which contains co-initiators that can react with the acidic resin monomers to produce free radicals, initiating the reaction of self-cured RC and primers\textsuperscript{18}. The Primer A is an aqueous solution of initiators, while the Primer B contains HEMA (2-hydroxyethyl methacrylates) and phosphoric acid acrylate monomers. It is possible to speculate that the presentation mode of co-initiators and the quality of bonding promoted by self-etching adhesive from Multilink cementing system were the main reasons for no differences in bond strength for the dual-polymerizing (self + light-cured) group of Multilink when compared to the control group.

The two autopolymerizing groups exhibited lower bond strength values than the dual-polymerizing groups, demonstrating that the curing mode significantly affected the bond strength. The
overall properties of RC using the dual-cure system seem to be superior when compared to the single-cure system\textsuperscript{19}. Noronha Filho et al.\textsuperscript{9} (2010) observed that RC present low degree of conversion when the indirect restoration material attenuates the light activation. The dual curing RC has been developed to overthrive these conditions\textsuperscript{19}. The dual-cure process is due to both external light and chemical agent in RC curing. In this process occurs first the polymerization induced by light and the polymerization induced by chemical agent is performed later. This technique satisfies incomplete polymerization due to insufficient light transmittance at the bottom of the sample\textsuperscript{19}. Light activation changed the failure mode on Clearfil SA Cement. Cohesive failures within RC (Figure 3) were observed for Clearfil SA Cement without light exposure, while light activation resulted in adhesive failures (Figure 4). The low degree of conversion of this RC in self-cure mode reduced the bond strength to dentin and the mechanical properties, such as fracture toughness, diametrical tensile strength and hardness\textsuperscript{21}.

The combination of bonding agent (Scotchbond Multipurpose) and RC (RelyX ARC) was used as control in this study. De Munck et al.\textsuperscript{22} (2005) assure that a comparison of contemporary adhesives reveals that the three-step, ethanol-water-based etch-and-rinse adhesives remain the ‘gold standard’ in terms of adhesion durability. Scotchbond Multipurpose belongs to this category of the best adhesives, which has primer and bonding agents (hydrophobic resin) in separate bottles. These categories of adhesive systems always involve the use of phosphoric acid, which permits demineralization of the dental tissues and, after rinsing, a complete elimination of the smear layer\textsuperscript{23}. This category of adhesive systems is capable of reaching high degree of conversion and consequently bond strength to dentin, as showed in this study\textsuperscript{22}.

Further studies will be carried out at additional times because the 24 hours may not correspond to the maximum polymerization rate, especially for the self-curing mode materials. Also, the long-term study of this subject would be important to learn more about the adhesive properties to dentin of the cementing system tested. This study used bovine teeth instead of humans, because bovine dentin morphology and adhesion are similar to human dentin and bond strength values were compatible with results published previously\textsuperscript{24}.

In conclusion, light activation increased the bond strength to dentin for Clearfil SA Cement and Multilink. No significant difference in bond strength was noted between control and light-cured Multilink group. Other experimental groups showed lower bond strength to dentin than that obtained with the control.

Acknowledgements

The authors would like to thank the Capes / Brazil (Coordenação de Aperfeiçoamento de Pessoal de Nível Superior - PROCAD) for the financial support. The authors state that they have no conflicts of interest.

References


