Effect of X-ray Radiation Dose on the Bond Strength of Different Adhesive Systems to Dentin

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\textbf{Purpose:} To investigate the influence of different x-ray radiation doses on the bond strength of adhesive restorations to dentin using different bonding strategies.

\textbf{Materials and Methods:} Flat dentin surfaces on human molars were obtained and cylinder-shaped specimens for the microshear bond test were built up with a composite (Z250, 3M ESPE), using three adhesive systems: a two-step etch-and-rinse (Single Bond 2 – SB2, 3M ESPE), a two-step self-etching (Clearfil SE Bond – CSE, Kuraray), or a single-step self-etching (Adper Prompt – ADP, 3M ESPE). The specimens were assigned to 4 groups (\(n = 10\)), according to the x-ray dose: 0 (control), 5, 35, or 70 Gy. Radiation was directed to the surface of the resin cylinders. Microshear testing was conducted after 24 h, and the failure modes classified under magnification (200X). Data were submitted to two-way ANOVA and Holm-Sidak’s test (\(p \leq 0.05\)). A nonlinear regression analysis was carried out with bond strength as dependent variable.

\textbf{Results:} Bond strength results were dose and material dependent. SB2: control > 5 = 35 > 70; CSE: control = 5 > 35 = 70; ADP: control = 5 = 35 = 70. Generally, SB2 > CSE > ADP. The nonlinear regression plots showed that in general, an increase in radiation dose may predict a decrease in bond strength (\(R^2 \geq 0.905\)). Failure modes were dependent on the bonding system, generally with no significant influence of radiation.

\textbf{Conclusion:} X-ray radiation might present a dose-dependent detrimental effect on the bond strength of resin composite restorations to dentin.

\textbf{Keywords:} adhesive systems, shear bond strength, dentin, radiation, radiotherapy, x-ray.

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primer and adhesive resin, resulting in micromechanical interlocking with the exposed collagen mesh. On the other hand, self-etching systems use nonrinse acidic monomers that simultaneously condition and prime dentin, presenting either two application steps, where the priming step is followed by application of free-solvent adhesive resin, or a single-step, where primer and adhesive resin are combined into one solution. For self-etching systems, adhesion is obtained through shallow hybridization with residual hydroxyapatite.14

Previous studies have evaluated the influence of radiation on bond strength of adhesive systems using irradiated dentin.1,6 In these studies, the restorative procedures were performed after irradiation, but bonding may be performed in tumor patients either before or after irradiation. It has been suggested that restorations with composite resins might show reduced life expectancy in irradiated patients.6 However, little is known about the effects of x-ray radiation on composite restorations, for instance, whether they have a shorter clinical service life than nonirradiated restorations. Moreover, the literature lacks studies which address the combined effect of different radiation doses and adhesion strategies on the bond strength of composite restorations.

The aims of this study were to (1) investigate the effect of different x-ray radiation doses on the bond strength of resin composite restorations mediated by distinct bonding strategies and/or application steps, and (2) examine the relationship between radiation dose and bond strength to dentin. The hypothesis tested was that the x-ray radiation would interfere with the bonding to dentin, irrespective of the dose applied.

### Table 1  Adhesive systems used in the study

<table>
<thead>
<tr>
<th>Material</th>
<th>Manufacturer</th>
<th>Main components</th>
<th>Procedure</th>
</tr>
</thead>
<tbody>
<tr>
<td>Single Bond 2</td>
<td>3M ESPE</td>
<td>Bis-GMA, HEMA, UDMA, acrylic and itaconic acid copolymers, water, ethanol, colloidal silica</td>
<td>a, b, c, e, f, g</td>
</tr>
<tr>
<td>Clearfil SE Bond</td>
<td>Kuraray</td>
<td>Primer: MDP, HEMA, water</td>
<td>d, f, e, g</td>
</tr>
<tr>
<td>Adper Prompt</td>
<td>3M ESPE</td>
<td>Bond: Bis-GMA, MDP, HEMA, colloidal silica</td>
<td>d+e, f, g</td>
</tr>
</tbody>
</table>

a: acid etch surface with 35% H3PO4 for 15 s; b: rinse with water; c: remove excess dentin moisture with absorbent paper; d: application of self-etching primer to dry dentin; e: application of bonding agent; f: air thin for 20 s; g: positioning of the elastomer mold and light activation for 20 s.

![Fig 1](image) Experimental set-up of the study. (1) adhesive system applied to ground dentin; (2) elastomer mold with cylinder-shaped orifices positioned onto the surface, and orifices filled with photoactivated resin composite; (3) polyester strip placed between the mold and the curing unit guide, and light activation of the composite; (4) different x-ray radiation doses applied to the specimens; (5) microshear bond strength testing.
MATERIALS AND METHODS

Sixty posterior human maxillary/mandibular molars with no caries or fillings, extracted over the course of three months, were obtained under a protocol approved by the institutional review board of Piracicaba Dental School, Brazil. The teeth were stored in 0.5% chloramine-T solution for 7 days for disinfection, and then in distilled water at 4°C until testing. The specimens were sectioned perpendicular to the long axis of the tooth with a diamond saw under water cooling, and their buccal and lingual surfaces were separately embedded in epoxy resin yielding 120 tooth specimens. In order to create a flat surface in medium dentin, these surfaces were wet ground with 220-, 400- and 600-grit SiC abrasive papers. The embedded tooth specimens were stored in distilled water, and assigned to 3 groups of 40 tooth specimens according to three adhesive systems: a two-step etch-and-rinse (Single Bond 2 – SB2; 3M ESPE; St Paul, MN, USA), a two-step self-etching (Clearfil SE Bond – CSE; Kuraray; Osaka, Japan) and a single-step self-etching (Adper Prompt – ADP; 3M ESPE) system. Material compositions are shown in Table 1.

In order to obtain specimens for the microshear bond strength test, the experimental design shown in Fig 1 was carried out.12 The adhesive systems were applied on dentin in accordance with the manufacturers’ instructions, following the procedures described in Table 1. Before the bonding agent was light activated, customized 1-mm-thick elastomer molds, each with three cylinder-shaped orifices (1 mm in diameter), were placed on the tooth surfaces, allowing the bonding area to be delimited. The adhesive was light activated for 20 s with the light-guide tip (XL2500; 3M ESPE, 700 mW/cm²) placed directly onto the elastomer mold. Thereafter, the orifices were filled with a photoactivated composite (Filtek Z250, shade A3; 3M ESPE). A transparent polyester strip was placed over the filled orifices and light activation was performed for 20 s. Forty specimens were obtained for each adhesive system.

The tooth specimens of each material were then randomly assigned to four subgroups, and each of them was submitted to one of 4 x-ray radiation doses: 0, 5, 35, or 70 Gy, with doses being applied using just one shot. Control specimens (dose = 0) remained stored in distilled water, at 37°C. Specimens were placed in individual containers during irradiation. To provide dose homogeneity, the container was filled with distilled water up to 5 mm above the resin blocks. Irradiation was performed using a linear accelerator (Clinac 6/100; Varian Medical Systems; Palo Alto, CA, USA) with 6 MeV and a 10 x 10 cm area. Radiation was applied perpendicular to the surface of the resin cylinders, at a 100 cm distance from the source.

After irradiation, tooth specimens were stored in distilled water at 37°C for 24 h, and all resin cylinders were examined at 40X magnification. Those presenting flaws, irregularities, or bonding defects were eliminated. For the microshear test, a thin steel wire (0.2 mm in diameter) was looped around each cylinder and aligned with the bonding interface. The bond strength test was conducted in a mechanical testing machine (model 4411, Instron; Canton, MA, USA), at a crosshead speed of 0.5 mm/min until failure. Bond strength values were calculated in MPa. For each adhesive system/radiation dose combination, ten tooth specimens were tested, and the average value of two to three resin cylinders was recorded as the bond strength value for each sample.

Fractured specimens were examined under an optical microscope (200X magnification). Failure modes were classified as follows: adhesive failure (mode 1), cohesive failure within the bonding agent (mode 2), or mixed failure involving bonding agent, composite, and dentin (mode 3). Bond strength data were submitted to two-way ANOVA followed by Holm-Sidak’s pairwise multiple comparison procedure, at a significance level of p < 0.05. Additionally, a nonlinear regression model was used to analyze the relationship between radiation dose (independent variable) and bond strength to dentin (dependent variable) for each material.

RESULTS

Table 2 shows the results of the microshear bond strength test. The factors “adhesive system” (p < 0.001) and “radiation dose” (p < 0.001) were significant, as was their interaction (p = 0.007). For SB2, all doses significantly reduced the bond strength to dentin (p < 0.01); similar findings were observed for 5 and 35 Gy (p = 0.278), whereas 70

<table>
<thead>
<tr>
<th>Table 2</th>
<th>Means (standard deviations) for microshear bond strength (MPa)</th>
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<tbody>
<tr>
<td></td>
<td>Control</td>
</tr>
<tr>
<td>Single Bond 2</td>
<td>15.7 (1.9)^A,a</td>
</tr>
<tr>
<td>Clearfil SE Bond</td>
<td>12.2 (2.2)^A,b</td>
</tr>
<tr>
<td>Adper Prompt</td>
<td>6.7 (2.1)^A,c</td>
</tr>
</tbody>
</table>

Means followed by different capital letters in the same line, and small letters in the same column, indicate significant differences at p < 0.05.
Gy presented a significantly higher effect (p < 0.0001). For CSE, the application of 5 Gy did not affect the bond strength (p = 0.260), but the application of 35 and 70 Gy significantly reduced the bond strength to dentin (p < 0.0001), with similar findings between these two doses (p = 0.398).

On the other hand, the adhesive system ADP presented low bond strength values throughout the study, with no significant effect of radiation, irrespective of the dose applied (p > 0.05). In addition, SB2 presented significantly higher bond strengths than both CSE and ADP (p < 0.05), except for irradiation with 70 Gy, when SB2 was similar to CSE (p = 0.294). Correspondingly, CSE showed a significantly higher bond strength than ADP (p < 0.05), irrespective of the radiation dose.

The results of the nonlinear regression analyses are shown in Fig 2. All models fitted the regression plots well (SB2: R² = 0.905; CSE: R² = 0.999; ADP: R² = 0.912), showing a strong relationship between radiation dose and bond strength to dentin. This relationship was found to be statistically significant for SB2 (p = 0.049) and CSE (p = 0.011), but not for ADP (p = 0.297). With regard to the failure analysis, each adhesive system presented a characteristic outcome: SB2 presented predominantly mixed failures, CSE predominantly adhesive failures, and ADP predominantly cohesive failures within the bonding layer. In general, radiation had no significant influence on the failure modes, except after application of 70 Gy in SB2, for which an increase in cohesive failures within the bonding layer was detected.

DISCUSSION

The results of this study showed a significant detrimental effect of radiotherapy on the bond strength to dentin, confirming the tested hypothesis. This finding is probably re-
lated to x-ray-induced alteration of the substrate, as previous studies showed decreased mechanical properties and damage to collagen fibrils after radiation.\textsuperscript{3,5,9} Moreover, it has been reported that the apatite crystals of dental hard tissues incorporate some sodium, carbonate, and magnesium by entrapment during their formation.\textsuperscript{8} When irradiated, these point defects could be mobilized from the surface layer of the crystals, removing the entrapped ions and modifying the structure of the crystals, thus potentially interfering with the adhesion.

The alteration in bond strength was dose and material dependent. Generally, the higher the x-ray dose, the lower the bond strength. Correspondingly, the regression analysis showed a strong relationship between the radiation dose and the bond strength to dentin for all materials (Fig 2); while a 5 Gy dose only slightly influenced the adhesion, 35 and 70 Gy doses strongly affected it. Moreover, the different bonding systems presented distinct behaviors. SB2 showed the highest bond strength values throughout the investigation, except after irradiation with 70 Gy. This is probably a result of the acid-etching step of this system, which may increase the irregularities for retention on the dentin surface. In addition, CSE showed higher bond strength than ADP, and this might be related to the slightly higher pH of the former,\textsuperscript{6} leading to formation of a more homogeneous hybrid layer when compared with the strong self-etching ADP.\textsuperscript{4} Indeed, ADP showed low bond strength values throughout the study, probably because of its single application step, which might hinder the proper etching of the substrate and simultaneous infiltration of the bonding resin. Although the ADP system showed no statistical difference in bond strength among the x-ray doses, probably because of the constant low bond strength values, a strong relationship between radiation dose and bond strength was also detected for this system (Fig 2).

In the present study, the teeth were stored in chloramine-T solution for a week, and then in distilled water prior to the bonding procedures. Lee et al.,\textsuperscript{10} evaluating different storage conditions, found similar results in bond strength tests for distilled water or chloramine-T solution. In addition, the dentin smear layer for all bonding strategies was standardized by wet polishing the tooth surfaces with SiC abrasive papers. Although it has been reported that the effect of surface abrasive methods might be material dependent,\textsuperscript{15} this is a well-known, commonly used procedure for preparation of dentin samples for bonding tests.\textsuperscript{1,12,18} Moreover, as previously reported,\textsuperscript{12} each tooth was considered an experimental unit in this study (n = 10 per group). The three resin cylinders tested for each tooth were averaged, and their mean value computed as the bond strength for each specimen. This was carried out to reduce the intratooth variability associated with bonding tests,\textsuperscript{11} where different regions of a same tooth surface are tested. Where the resin cylinders presented defects before testing (typically one or two per group), these cylinders were eliminated.

Distinct failure characteristics were detected among the bonding materials, and this observation is probably related to the different composition or application steps of the systems. SB2 presented predominantly mixed failures, which might be explained by the in-depth demineralization of dentin by the phosphoric acid. The etching procedure leaves nonencapsulated collagen fibrils after bonding, because of the inability of this material to fully infiltrate the exposed collagen mesh.\textsuperscript{18} These unprotected areas may serve as spots for stress concentration during the shear testing, generating failures involving not only the bonding layer, but also the dentin tissue. Conversely, CSE showed predominantly adhesive failures, probably because of its lower ability to create micromechanical retention compared to the phosphoric acid, leading to failures at the dentin/adhesive interface. On the other hand, ADP showed predominantly cohesive failures within the bonding layer, which could be related to a lower ultimate strength of the polymer. As ADP is applied in a single step, with primer and bonding agents mixed together, the lower cohesive strength of the polymer could be a result of the inhibitory effect of the solvent on the polymerization mechanism.\textsuperscript{19}

The radiation generally presented no significant effect on the failure modes, except for SB2, for which an increase in cohesive failures within the bonding layer was detected after application of 70 Gy. This could be related to a radiation effect on the polymer structure, altering the network and reducing the frictional forces between the polymer chains. As a result, the ultimate strength of the material would be reduced. For the other systems, no radiation effect on the failure patterns was evident. For ADP, this is probably because of the low bond strength values observed throughout the entire study, while for CSE this is likely related to its two-step mode of application. For CSE, the final application of bonding resin over the priming solution may enhance the properties of the polymer, since this resin has a higher amount of the stiff bis-GMA monomer, increasing the mechanical strength of the network. Therefore, a more heterogeneous polymer, with lower resistance to radiation, could be formed in SB2, due to the presence of solvent, which is really very difficult to remove completely during air-drying procedures and can interfere with the properties of the polymer.\textsuperscript{19} Indeed, for SB2, a 5 Gy dose significantly affected its bond strength, while for CSE, this effect was observed only for higher doses.

Head and neck malignancies are often in close proximity to normal tissues, such as the spinal cord, brain stem, salivary glands, and optic structures. Therefore, in addition to a decrease in bond strength of adhesive restorations, other alterations might occur after radiotherapy, such as changes in salivary function and mucosal structures, radiation caries, or osteoradionecrosis.\textsuperscript{13,17} Moreover, properties of the restorative material might also be influenced by x-ray radiation; damage in composites could include polymer cracking or fracture, delamination, interphase cracking, filler dislodge-ment, and debonding. The damage mechanisms operating and the rate of damage accumulation would probably be related to the dose applied. However, the literature lacks investigations regarding x-ray-induced changes in dental composites.

The present results raise a question about the harmful influence of x-ray radiation on the bond strength to dentin, and the two-step self-etching system tested seems to be less affected by a low-radiation dose than the etch-and-rinse system. Nonetheless, in clinical situations, it is difficult to pre-
dict whether adhesive restorations performed with the materials tested here would require replacement after irradiation. A conclusive answer based on the present results is not possible, since this in vitro study could not take into account the effects that alteration in salivary flow or oral flora may have after irradiation. Therefore, clinicians should be aware of possible implications that radiotherapy may have on the longevity of adhesive fillings, and a careful follow-up of the performance of these restorations is advisable.

**CONCLUSION**

X-ray radiation might present a dose-dependent detrimental effect on the bond strength of resin composite restorations to dentin.

**REFERENCES**


**Clinical relevance**: Under clinical conditions, if x-ray radiotherapy is necessary for patients with adhesive fillings, a careful follow-up of the performance of these restorations is advisable.