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Bond Strength of Dental Adhesive Systems Irradiated with Ionizing Radiation

Adriana Dibo da Cruz²/Luciano de Souza Gonçalves³/Alessandra Nara de Souza Rastelli⁴/Lorenço Correr-Sobrinho⁵/Vanderlei Salvador Bagnato⁶/Frab Norberto Bóscolo⁷

Purpose: The aim of the present paper was to determine the effect of different types of ionizing radiation on the bond strength of three different dentin adhesive systems.

Materials and Methods: One hundred twenty specimens of 60 human teeth (protocol number: 032/2007) sectioned mesiodistally were divided into 3 groups according to the adhesives systems used: SB (Adper Single Bond Plus), CB (Clearfil SE Bond) and AP (Adper Prompt Self-Etch). The adhesives were applied on dentin and photo-activated using LED (Lec 1000, MMoptics, 1000 mW/cm²). Customized elastomer molds (0.5 mm thickness) with three orifices of 1.2 mm diameter were placed onto the bonding areas and filled with composite resin (Filtek Z-250), which was photo-activated for 20 s. Each group was subdivided into 4 subgroups for application of the different types of ionizing radiation: ultraviolet radiation (UV), diagnostic x-ray radiation (DX), therapeutic x-ray radiation (TX) and without irradiation (control group, CG). Microshear tests were carried out (Instron, model 4411), and afterwards the modes of failure were evaluated by optical and scanning electron microscope and classified using 5 scores: adhesive failure, mixed failures with 3 significance levels, and cohesive failure. The results of the shear bond strength test were submitted to ANOVA with Tukey’s test and Dunnett’s test, and the data from the failure pattern evaluation were analyzed with the Mann Whitney test (p = 0.05).

Results: No change in bond strength of CB and AP was observed after application of the different radiation types, only SB showed increase in bond strength after UV (p = 0.0267) irradiation. The UV also changed the failure patterns of SB (p = 0.0001).

Conclusion: The radio-induced changes did not cause degradation of the restorations, which means that they can be exposed to these types of ionizing radiation without weakening the bond strength.

Keywords: adhesion, dentin, dentin adhesives, irradiation, degradation.


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The light-curing composite resins are widely used in restorative dentistry. One of the established factors of clinical success for dental restorations is the reliable bond strength between the restorative material and dental tissues.¹,³,⁵,⁹,¹⁶-¹⁸ that it is obtained by dental adhesives.

The improvement of the adhesive systems caused an increase in their clinical applications in adhesive dentistry, including pit and fissure sealants, orthodontic brackets, adhesive bridges, laminate veneers, and direct resin restorations.¹ However, in spite of the improvements, undesirable consequences such as recurrent caries or marginal discoloration are often found in resin restorations following long-term clinical use, due to exposition to several external agents.⁷

Restorations in the oral cavity are challenged by different external agents, including ionizing radiation from radiographic diagnosis, radiotherapy, or environmental sunlight. These restorations may be negatively influenced by the interaction of radiation with their atoms and molecules through electrostatic and electromagnetic forces. The interaction occurs with any atoms and molecules exposed to the radiation source.⁴,¹⁹ i.e., tissue, vital organs, or biomaterials.
Although some studies have reported on the direct effects of the exposure of ionizing radiation on dental materials and tissues, the results of such exposure are still unclear.

Some studies used high-energy ionizing radiation, such as gamma or electron radiation, on dental materials or tooth/restoration complexes. In the case where such radiation was directly applied to dental materials, it was observed that microhardness, fracture toughness, and others mechanical properties changed proportionally to the increase of the applied radiation dose. When radiation was applied to restored teeth to simulate radiotherapy, some changes in bond strength were observed.

Other studies used low-energy ionizing radiation, such as ultraviolet radiation (UV), and in these cases color changes were observed (among other alterations). To our knowledge, nothing exists in the literature about effects of the x-ray exposure used for diagnosis on dental materials or on dental tissues.

Both high- and low-energy ionizing radiation have also been used to induce accelerated aging by exposure to light to assess the resistance of materials to the degradation process. Considering that intraoral adhesive restorations can be exposed to different types of ionizing radiation as they have great energy and capacity of penetration, any negative change in the adhesive capacity of the restoration caused by irradiation will be a contraindication for these adhesive systems. Thus, the aim of the present paper was to determine the effect of different types of ionizing radiation on the bond strength of three different dentin adhesive systems.

MATERIALS AND METHODS

After approval of this project by the Ethics Committee for Human Research of the School of Dentistry, Piracicaba, State University of Campinas, protocol number 032/2007, 60 noncarious human permanent third molars were selected. The teeth were cleaned and stored in distilled water at 4°C for no more than 6 months post-extraction. The crowns were cut off of the roots and then sectioned mesiodistally using a diamond saw (#7020 flexible diamond disk, KG Sorensen, Barueri, SP, Brazil) in an Isomet low-speed saw (Buehler, Lake Bluff, IL, USA) with water cooling.

Each half crown was embedded in PVC cylinder rings with epoxy resin to maintain the outside dentin surface level with the ring base. The dentin surfaces were wet ground with 180-, 220-, 400- and 600-grit SiC abrasive papers, in order to create a smooth, flat surface in the medium dentin. After this, 120 specimens were divided randomly into 3 groups (n = 40). Each group used one of the adhesive systems described in Table 1.

The adhesive systems were applied on dentin as described in Table 2. Absorbent paper was used to remove the excess dentin moisture, and all adhesives systems were photopolymerized with an LED light-curing unit (Lec 1000 MMoptics, São Carlos, SP, Brazil) at 1000 mW/cm².

In order to obtain cylinders for the microshear bond strength test, customized 0.5-mm-thick elastomer molds, each with three cylinder-shaped orifices (1.2 mm in diameter), were placed on the tooth surfaces, allowing delimitation of the bonding area. Afterwards, the cylinder-shaped orifices were filled with composite resin (Filtek Z-250 Universal Restorative, 3M ESPE, St Paul, MN, USA), and a mylar strip was placed over the filled orifices. Prior to the composite resin photo-activation procedures, a constant and uniform 250 g cementation load was applied for 2 min, using a custom-made device. The composite resins were photo-activated for 20 s with the same LED light-curing unit mentioned above. The specimens were stored at 37°C (± 1°C) and 95% (± 5%) relative humidity during the entire experiment.

Each group (n = 40) was subdivided randomly into 4 subgroups (n = 10 specimens with n = 30 cylinders) according to the following experimental treatments:

1. Ultraviolet radiation (UV), in light box with relative humidity at 50% (± 5%) using a mercury light HN ZN, 15 W (Huaning, Nanjing, China), 253.7 nm, 15 W, for 48 h, resulting in 1157.76 J/cm².
2. Diagnostic x-ray radiation (DX) from a Poli-Tecnica 300/125 x-ray unit (Engelcin, São Paulo, SP, Brazil), using 30-s exposure times, 80 kV tube voltage, 200 mA tube current, 100 cm focus-object distance and 10 cm² radiation field.
3. Therapeutic x-ray radiation (TX), from Clinac 600 Linear Accelerator (Varian Medical Systems, Palo Alto, CA, USA), with a 6 MV x-ray beam, 70 Gy exposition dose, 100 cm focus-object distance, 10 cm² radiation field.

Thus, with the end of the experimental treatment application (48 h after specimen photo-activation), the customized molds were removed from the specimens. All resin cylinders were checked by stereomicroscope (Carl Zeiss do Brasil, São Paulo, Brazil) under 40X magnification. Twenty-two cylinders (6.1% of the sample) presenting flaws, irregularities or bonding defects were eliminated from the test. Specimens with 2 cylinders were accepted as samples, which presented only 1 cylinder would be eliminated, but none were found. Therefore, the sample maintained all 120 specimens.

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 test was conducted in a universal testing machine (Instron, Canton, MA, USA, model 4411), at a crosshead speed of 0.5 mm/min until failure. Bond strength values were calculated in MPa. The average bond strength value of the cylinders was taken as a reference value of the specimens, which then was used for statistical analysis. Bond strength data were submitted to ANOVA by Tukey's test (p = 0.05) for comparison among the radiation types and among the adhesives; each group was independently compared with the control group by Dunnett’s test (p = 0.05).
Table 1 Adhesive systems used in the present study and its groups

<table>
<thead>
<tr>
<th>Groups</th>
<th>Commercial name</th>
<th>Manufacturer</th>
<th>Composition*</th>
</tr>
</thead>
<tbody>
<tr>
<td>SB</td>
<td>Adper Single Bond Plus Adhesive</td>
<td>3M/ESPE, Dental Products, St Paul, MN, USA</td>
<td>Bis-GMA, HEMA, dimethacrylates, ethanol, water, copolymer of polyacrylic, polyitaconic acids and silica nanofiller</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>Primer: MDP, HEMA, dimethacrylate monomer, water, catalyst</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>Bond: MDP, HEMA, dimethacrylate monomer, microfiller, catalyst</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>Liquid 1 (red blister): Methacrylated phosphoric esters, bis-GMA, initiators based on camphorquinone, stabilizers</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>Liquid 2 (yellow blister): water, HEMA, polyalkenoic acid, stabilizers</td>
</tr>
<tr>
<td>CB</td>
<td>Clearfil SE Bond</td>
<td>Kururaray, Kurashiki, Japan</td>
<td></td>
</tr>
<tr>
<td>AP</td>
<td>Adper Prompt Self-Etch Adhesive</td>
<td>3M/ESPE, Dental Products, St Paul, MN, USA</td>
<td></td>
</tr>
</tbody>
</table>

* Information available in the technical product profile provided by the manufacturer.

Table 2 Protocol of application of the adhesive systems used in the present study

<table>
<thead>
<tr>
<th>Groups</th>
<th>Procedure*</th>
</tr>
</thead>
<tbody>
<tr>
<td>SB</td>
<td>Application of 35% phosphoric acid (Scotchbond Etchant, 3M/ESPE, Dental Products, St. Paul, MN, USA) on dentin for 15 s. Abundant rinse with distilled water for 10 s, and then dried the water excess with absorbent paper. Application of 3 coats of adhesive using a microbrush applicator (Microbrush International, Grafton, USA) with gentle rubbing for 15 s, and then dried with a soft air flow for 5 s. Photo-activation for 10 s.</td>
</tr>
<tr>
<td>CB</td>
<td>Application of primer using a microbrush applicator for 20 s and then dried thoroughly with a soft airflow. Application of 1 coat of bond using a microbrush applicator with a gentle rubbing and dried soft airflow for 3 s. Photo-activation for 10 s.</td>
</tr>
<tr>
<td>AP</td>
<td>Rapid mix of 1 drop of liquid 1 with 1 drop of liquid 2 for 5 s. Application of the mixture with hard rubbing over dentin using microbrush applicator for 15 s and then dried thoroughly with a soft air flow. Photo-activation for 10 s.</td>
</tr>
</tbody>
</table>

* Before starting the described procedures, the polished surface was cleaned with distilled water and dried with absorbent paper.

The fractured specimens were examined under an optical microscope (HMV-2, Shimadzu, Nakagyo-ku, Kyoto, Japan) at 200X magnification by the same appraiser. Modes of failure were classified as follows:

1. Adhesive failure, between dentin and adhesive.
2. Mixed failure, between dentin and adhesive with 75% dentin exposed.
3. Mixed failure, between dentin and adhesive with 50% dentin exposed.
4. Mixed failure, between dentin and adhesive with 25% dentin exposed.
5. Cohesive failure, within adhesive or composite resin.

Additionally, representative fractured specimens were sputter coated with gold and re-examined using SEM (JSM5600LV; JEOL, Peabody, MA, USA) for validation of optical microscopic assessment. Failure pattern scores were submitted to the Mann Whitney test (p = 0.05).

RESULTS

The results of the microshear bond strength test are shown in Fig 1. There were no differences in microshear bond strength due to exposure to different radiation types. No interaction processes occurred between radiation and adhesive systems. Statistically significant differences were only found in the UV-irradiated SB group when compared with the control group (Dunnett's test, p = 0.0267).

Figure 2 shows the failure pattern distributions according to the scores given in Fig 3. In Table 3, the SB adhesive showed a statistically significant change (p = 0.0000) of failure pattern with exposure to different types of radiation.
**DISCUSSION**

In the present short-term laboratory study, the tooth/restoration complex was submitted to different types of ionizing radiation to evaluate their influence on adhesive bond strength. Different adhesive systems were also used to evaluate the possibility of alteration on bond strength after irradiation due to the differences in the mode of application of the adhesive systems. The SB adhesive is a two-step system, in which the first step consists of preparing the tooth surface with etching gel followed by rinsing, and the second step of applying the primer/bonding agent. The CB adhesive is also a two-step system, but the first step consists of a self-etching primer system without rinsing to prepare the tooth surface, and the bonding agent is applied in the second step. The AP adhesive is a one-step system where etching, primer and bonding agents are applied together.

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**Fig 1** Results of the microshear bond strength test. * The value differed from control according to the Dunnett test (p<0.05). Groups with same letter (capital letter in the columns with same treatment comparing adhesive systems and lower case letters in the joined columns comparing different treatments) are not significantly different according to ANOVA and Tukey's test (p>0.05).

**Fig 2** Distribution of the failure patterns (median values) in specimens (from 1 to 10) with scores used (from 1 to 5). A= Results of the SB adhesive; B= Results of the CB adhesive; C= Results of the AP adhesive. The fractioning of median values can occur in specimens with two cylinders.
Table 3 Median values of the failure patterns (minimum – maximum)

<table>
<thead>
<tr>
<th>Experimental treatment</th>
<th>Material</th>
<th>SB adhesive</th>
<th>CB adhesive</th>
<th>AP adhesive</th>
</tr>
</thead>
<tbody>
<tr>
<td>Control non-experimental</td>
<td></td>
<td>3.0 (2.0 - 5.0) Aa</td>
<td>5.0 (1.0 - 5.0) Ba</td>
<td>5.00 (4.0 - 5.0) Ab</td>
</tr>
<tr>
<td>Diagnostic X-radiation</td>
<td></td>
<td>4.0 (2.0 - 5.0) Bb</td>
<td>2.0 (1.0 - 5.0) Aa</td>
<td>5.00 (3.0 - 5.0) Ac</td>
</tr>
<tr>
<td>Therapeutic X-radiation</td>
<td></td>
<td>5.0 (3.0 - 5.0) Cb</td>
<td>3.0 (3.0 - 5.0) ABa</td>
<td>5.00 (3.0 - 5.0) Ab</td>
</tr>
<tr>
<td>Ultraviolet radiation</td>
<td></td>
<td>5.0 (1.0 - 5.0) BCb</td>
<td>2.0 (2.0 - 5.0) Ba</td>
<td>5.00 (4.0 - 5.0) Ac</td>
</tr>
</tbody>
</table>

Values with the same letters (capital letters in the columns and lower case letters in the rows are not significantly different according to the Mann-Whitney test (p>0.05).

The results showed that the different types of radiation did not cause changes in the bond strength of the CB or AP adhesive; only the SB adhesive showed an increased bond strength after exposure to UV when compared with control group. The high deviation pattern values observed in Fig 1 could have occurred due to the manufacturing method of the cylinders, which could have internal micro-irregularities imperceptible under the stereomicroscope. The results of the present study agreed with the study of Bulucu et al,5 who also did not observe changes in bond strength after exposure to TX. However, they observed that the irradiation with TX changed the bond strength behavior of the different adhesives. In the present study, the same phenomenon was observed: the bond strength behavior of the CB adhesive was similar to the SB adhesive before exposure to radiation. However, after exposure to DX and TX, the CB adhesive behaved similar to the AP adhesive. The bond strength behavior of the AP adhesive was different from the SB and CB adhesives, but after exposure to UV, it was similar to them.

In the study by Ye et al,22 changes were observed in the degree of conversion of adhesives when light activated using different curing units. The adhesives were applied using a different method. These differences occur because the adhesives are dissimilar both in the amount of solvent and in the type of photoinitiator used, which induced dissimilar conversion behavior. However, this behavior also depends on the intensity and breadth of the radiation spectrum. In the present study, three different adhesive systems were selected and photo-activated with an LED unit, which has a narrower radiation spectrum than halogen bulbs. Due to its narrower spectrum, using an LED unit could cause insufficient conversion, which might lead to greater changes when later exposed to radiation – chiefly UV. For other radiation types, a possible change could only occur after interac-
tion with dental tissue, because absorbed and scattered radiation produces secondary radiation, with smaller wavelengths, and is then able to interact with the molecules of the adhesives.

The present study focused on effects of ionizing radiation on dental restoration by examining adhesive bond strength behavior, varying the adhesives and energy of the radiation. Other studies focused only on therapeutic-dose ionizing radiation in dental tissue. The study by Bulucu et al. focussed on the effects of dentin exposure to TX at a dose of 60 Gy, although their study had two distinct groups: one with radiation applied on teeth without restorations and the other with radiation applied on restored teeth. Gernhardt et al. evaluated the effects of restorations on irradiated dentin; however, in their study the restorations were not irradiated. In one study by Gernhardt et al., no differences were found in adhesive bond strength after radiotherapy, while Bulucu et al. did find differences. Thus, the present study agreed with Gernhardt et al. because here as well, no differences caused by TX radiation were found.

The therapeutic radiation doses used in the previously cited studies and the present study were similar, and comparable with radiotherapy of the head and neck region. The exposure doses of UV and DX used in the previous study were based on others studies, but they did not have any clinical application. Due to great penetration, TX and DX can reach the dental tissue when applied to a restoration; however, the same did not occur with UV. UV radiation can break chemical bonds, to reactivate or to ionize molecules, but their photons have little energy and little penetration ability. Depending on the restoration thickness, UV cannot reach the dental tissue when applied to the restoration. Thus, the UV radiation can act in a more concentrated manner in the restorative material. There are no studies about the effects of DX or UV on dental tissues, only studies with therapeutic radiation doses. Concerning the radiation effects on dental tissues, there are two opposing views: one indicating that radiotherapy is able to change the dental structures and the other that it is not possible. However, in this study, a change of bond strength was only observed after exposing SB adhesive to UV (Dunnnett's test).

When dental materials alone, such as photoactivated composite resin, were irradiated with a therapeutic radiation dose, as in the study by von Fraunhofer et al., changes were observed in some mechanical properties of some materials. However, Curtis et al. did not observe any change after using the same restorative materials and the same radiation exposures, but they evaluated other mechanical properties that did von Fraunhofer et al. Thus, according to the cited authors, therapeutic radiation doses are able to cause some changes to dental materials; however, the response of each mechanical property is independent. Other previous studies corroborate the cited authors, affirming that the same exposure to radiation may induce different responses of different materials, due each material possessing either a larger or a smaller amount of radiosensitive chemical groups. The present study also corroborated these previously cited studies because each one of the three studied materials showed a different response to exposure to the same radiation. However, this may have occurred because of different application modes of each adhesive system.

The different types of radiation changed the failure patterns of SB adhesive, and UV induced greater changes when compared with the control group. Under the influence of the same radiation, the failure patterns also varied between the different adhesive systems. For SB adhesive, the application of x-ray radiation resulted in a slight increase in mixed failures, and UV radiation resulted in changes from adhesive failure to cohesive failure. The occurrence of cohesive failure could be an indication that the bond strength of the dentin to the adhesive equals or exceeds the resistance of the composite resin used. The reduction in incidence of adhesive failure due to exposure to radiation and the increased bond strength may indicate an adhesion improvement in the dentin/adhesive interface. Because in the present study, the results of failure patterns are linked with the increased bond strength of the SB adhesive, we may affirm that radiation improved this adhesive. When the increase of cohesive failure was added to the decreased bond strength, it suggests a degradation of the material, which did not occur in the present study. The results of the present study are in agreement with results of other studies which indicated that the therapeutic ionizing radiation dose does not cause detrimental effects on photoactivated composite materials, but it is able to improve the materials studied.

The use of different types of ionizing radiation to improve dental materials is known; however, the radiation doses applied in these cases in nonclinical situation are much higher. Regarding UV in the present study, a disagreement exists with previous studies which indicated that this radiation type has detrimental effects on dental materials. However, in these previous studies, the action of UV exposure on adhesive bond strength or failure pattern was not evaluated. The UV was used to cause aging in terms of color changes, microhardness, and degree of conversion, among others. Regarding DX, there are no studies in the literature about its interaction with dental materials. Therefore, further research should attempt to explain the interaction of different types of ionizing radiation with dental materials by use of other mechanical tests, in order to extend the lifetime of the restoration.

CONCLUSIONS

The types of ionizing radiation used in this study induced changes in bond strength and failure patterns of the studied adhesives. However, the changes depended on the radiation energy and type of adhesive materials evaluated. Considering the experimental model, it is possible to affirm that the radiation types applied do not cause degradation of adhesive materials by weakening the bond strength.
ACKNOWLEDGMENTS

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REFERENCES


Clinical relevance: Different external agents, including ionizing radiation from radiographic diagnosis, radiotherapy, or environmental sunlight, challenge restorations in the oral cavity. Thus, it is necessary to evaluate whether such radiation in different doses can induce changes in dental materials.