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ORIGINAL RESEARCH



Microtensile Bond Strength of Polyacid-modified Composite Resin to Irradiated Primary Molars

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ABSTRACT

Aim: This study evaluated the influence of various doses of radiotherapy on the microtensile bond strength (μ TBS) of compomer resin to dentin and enamel in primary molars.

Materials and methods: Thirty-five intact primary molars were collected and divided into seven groups. Teeth were irradiated with doses from 10 to 60 Gy, except for the control group. Compomer restorations were performed, and enamel–compomer resin beams and dentin–compomer resin beams were tested at a crosshead speed of 1 mm/min.

Results: No statistically significant difference was found between the irradiated tooth enamel and the control group (F = 1.1468; p = 0.194). However, statistically significant differences were evident among the dentin groups (F = 11.050; p < 0.001).

Conclusion: Radiation may not cause a significant difference in the μ TBS of compomer resin to primary tooth enamel, but appears to dose dependently decrease its bond strength to primary tooth dentin.

Clinical significance: Radiotherapy may affect the success rate of compomer fillings in primary teeth, especially in deeper cavities with exposed dentin.

Keywords: Compomers, Radiotherapy, Tensile strength.

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INTRODUCTION

Radiotherapy is part of the multidisciplinary approach to the treatment of head-and-neck cancers in children and adolescents. Head-and-neck radiotherapy involves regions, such as the tongue, nasopharynx, tonsils, tongue base, and the floor of the mouth.¹⁻³ Studies evaluating the long-term effects of head-and-neck radiotherapy in children have reported adverse effects, such as facial asymmetry, ophthalmological problems, neuroendocrine problems, hearing loss, and dental abnormalities in the first 10 years following the radiotherapy. Dental anomalies, including microdontia, trismus, mandibular hypoplasia, hypodontia, developmental abnormalities of roots, and radiation decay, are also not uncommon.⁴

Previously, studies evaluating the effects of radiotherapy in dentistry focused on radiation-related tooth decay. However, the focus has now started to shift toward evaluating the effects of radiotherapy on the bonding of adhesive restorative materials to teeth, albeit there are only a few studies on the subject thus far. All of these studies evaluated the bond strength of composite resin restorative materials to permanent teeth and revealed that radiotherapy reduces composite resin bond strength to both enamel and dentin in permanent molars.^{5,6} Soares et al⁶ have reported similar low bond strength between composite resin and the radicular dentin. Another study that evaluated permanent teeth reported that the application of a single radiotherapy dose of 60 Gy caused a reduction in the bond strength between glass ionomer cement and dentin.⁷ However, the effects of radiation on the bonding strengths between compomer resin restorative materials widely used in deciduous teeth and enamel and dentin hard tissue in deciduous teeth have not been studied.

The aim of this study was to evaluate the effects of various doses of radiation therapy (10–60 Gy) on the μ TBS of compomer resin restorative materials to the enamel and dentin of primary molars and also to compare the

mode of fracture by which these bonds fail. The null hypothesis was that radiotherapy in doses from 10 to 60 Gy has no effect on the bond strength of compomer resins on enamel and dentin hard tissue of primary molars and does not cause any changes in the mode of fracture failure.

MATERIALS AND METHODS

This study was approved by the Research Ethics Committee of our school (#2010.6.1/26). Thirty-five intact human primary molars—freshly extracted or exfoliated were used. The teeth were kept in 0.9% saline solution at room temperature for periods no longer than 1 month until they were used for experiments. After removing tissue debris and deposits on the teeth with a periodontal scaler, teeth were randomly divided into seven groups of five teeth. These groups were: G-1: nonirradiated (control group) and G-2, G-3, G-4, G-5, G-6, and G-7, which were irradiated with 10, 20, 30, 40, 50, and 60 Gy respectively. Radiation therapy was administered at 2 Gy per day, five times per week up to a maximum total dose of 60 Gy.

Sample Preparation

Quadrant upper jaw models were created from dental wax to keep the teeth stable and to allow the radiotherapy to be applied in a reproducible position at each treatment session. Teeth were placed in order on these models. Models were then placed in a Styrofoam box, and the simulation of radiotherapy application was performed. One wax jaw quadrant was removed from the box after every 10 Gy of radiation. The wax jaw models were incubated in pure water between radiation treatment sessions. Under the supervision of the radiation oncologist, a technician applied the radiotherapy to the teeth using a linear accelerator (Siemens Primus, Malvern, USA). The maximum total dose applied to the teeth was 60 Gy. This dose was applied to the teeth five times per week at 2 Gy per day in 30 fractions over 6 weeks. Radiotherapy applications were done at the same time every day.

Restorative Procedures

After radiotherapy, all teeth were removed from the jaw models. The roots were then embedded in acrylic blocks up to 1 mm below the cementoenamel junction for the microsectioning process. Teeth were sectioned horizontally from one-third of the crown length so that dentin and enamel surfaces could be obtained from the same tooth.

The enamel surfaces of the five teeth in each group were sectioned using an Isomet device (1000 Isomet; Buehler Ltd., Lake Bluff, Illinois, USA). Smear layer was removed from the open enamel and dentin surfaces by applying 600-grit silicon carbide sandpaper for 30 seconds. The enamel and dentin surfaces were then treated with 34.5% phosphoric acid (Vococid; Voco, Cuxhaven, Germany) for 30 seconds, washed with water for 20 seconds, and dried with polyurethane foam (Voco, Cuxhaven, Germany). A self-etch bonding agent (SEA; Futurabond M; Voco, Cuxhaven, Germany) was applied to the prepared enamel surface according to the manufacturer's instructions for 20 seconds, air-thinned for 5 seconds, and cured with a standard light-emitting diode device for 10 seconds. A total of 6 mm of compomer restorative resin material (Glossiosit; Voco, Cuxhaven, Germany) was placed incrementally in layers of no more than 2 mm; each layer was cured for 20 seconds. The compomer-restored teeth were then embedded in acrylic blocks up to 1 mm below the compomer restoration for the microcutting process. Enamel-compomer resin beams and dentin-compomer resin beams with a cross-sectional area of $1 \times 1 \text{ mm}^2$ were obtained from each sample using a precision-cutting device (1000 Isomet; Buehler Ltd., Lake Bluff, Illinois, USA).

Microtensile Test

Samples were incubated at 37°C for 24 hours and subjected to 500 thermal cycles of 5 to 55°C at 20-second intervals. Damaged beams or beams that were incompatible with the microtensile testing device were excluded from the study. The ends of each beam were glued to the test block of the microtensile test device (Micro Tensile Tester; Bisco, Schaumburg, Illinois, USA) using a Cyanoacrylate adhesive (Zapit Dental Ventures of America, Corona, California, USA). The microtensile stress test was performed at a crosshead speed of 1 mm/min. The load at failure was recorded in Newton (N) values and then converted into megaPascal (MPa). After failure, beam surfaces were evaluated under a stereomicroscope at ×40. The failure mode of each tested sample was categorized as "adhesive", "cohesive", or "mixed (adhesive and cohesive)".

Scanning Electron Microscope Evaluation

The failed surfaces of beams were also examined using a scanning electron microscope (SEM) (JMS-5600; Jeol Ltd., Tokyo, Japan). The sample cross-sections were coated with Au–Pd on a coating unit (Polaron E500; Comercial Assens-Llofrin SA, Barcelona, Spain) before SEM imaging.

Statistical Evaluation

One-way analysis of variance with Duncan *post hoc* multiple comparisons test was used to compare the µTBS of compomer resin to primary enamel and dentin following radiotherapy. The Mann–Whitney U-test was used to analyze the failure mode of enamel–compomer and dentin–compomer beams. All analyses were performed



with Statistical Package for the Social Sciences version 20.0 software (SPSS Inc., Chicago, Illinois, USA), with p < 0.05 considered as a statistically significant difference.

RESULTS

The average μ TBS values of enamel–compomer resin beams ranged from 36.4 MPa (G-4) to 44.0 MPa (G-5). This intergroup difference was not statistically significant (*F* = 1.1468; p = 0.194). Table 1 summarizes these average μ TBS values and statistical analyses.

The average μ TBS values of dentin–compomer resin beams ranged from 25.6 MPa (G-2) to 46.8 MPa (G-4). In contrast to the results from the enamel–compomer beams, there were statistically significant differences between the groups (*F* = 11.050; p = 0.000). Table 1 summarizes these differences (calculated by *post hoc* Duncan's multiple comparison test). The rank order of groups according to their average μ TBS value was (from low to high) G-1=G-4>G-5=G-6=G-3>G-2=G-7.

Table 2 presents the frequency of different modes of bond failure in the enamel–compomer and dentin– compomer beams. Overall, failure in the enamel– compomer resin beams was predominantly (69%) adhesive failure, with mixed and cohesive failure rates being 21 and 10% respectively. However, there were statistically significant differences between the groups in terms of failure mode notably between the G-2 group and G-5 (MWU = 117.00; p = 0.014), G-6 (MWU = 85.50; p = 0.013), and G-7 (MWU = 112.50; p = 0.015). Failure in the dentin–compomer resin beams was also mainly adhesive failure (80%), with mixed failure and cohesive failure accounting for 18 and 2% respectively. However, there was no significant difference in the predominant mode of failure between the groups.

The SEM micrograph images of enamel beams are shown in Figure 1. Figures 1A, B and D illustrate the characteristics of mixed failure at ×40, ×45, and ×43 magnification respectively. Figure 1C shows adhesive failure between hard dental tissue and restorative material at ×60 magnification. Figure 2 shows similar images for dentin beams and demonstrates cohesive failure (Figs 2A, B and D, at ×65, ×75 and ×47 magnification respectively) and mixed failure (Fig. 2C, at ×50).

Figure 3 shows high magnification images of the interface between the restorative material and the dental hard tissue. Dentin surfaces were partially exposed (*white arrow*). Figure 3C shows the partial failure of the bond between the restorative material and the dentin surface. Figure 3D shows tearing in the interface between the enamel rods and the compomer. Similarly, Figures 3A and C depict the breakdown of the bond between the restorative material and the dentin constraint and the dentin rod surface (*black arrow*).

DISCUSSION

Although compomer resins have lower mechanical and esthetic properties when compared with composite resins, they are widely preferred in pediatric patients due to their low technical accuracy requirements (ease of placement), fluoride release properties, biocompatibility, shade options, and acceptable esthetics.⁸ We thus chose

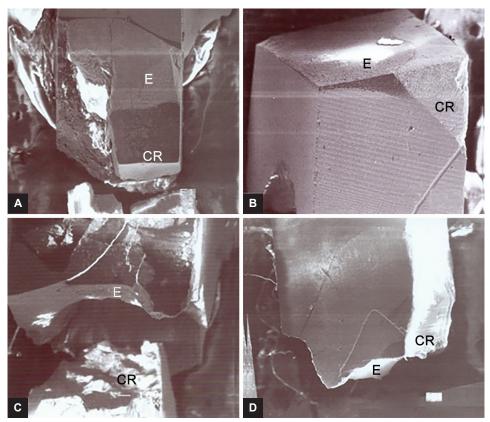
Groups	Sample number (n)		Microtensile (mean ± SD) and statistical significance (F/P)					
	Enamel	Dentin	Enamel	F/P	Dentin	F/P		
G-1	21	21	41.5 ± 9.2 ^a	F = 1.468/p = 0.194	45.4 ± 10.5 ^a	F = 11.050/p = 0.000		
G-2	18	17	42.9 ± 12.3 ^a		25.6 ± 11.6 ^b			
G-3	22	16	38.4 ± 9.6 ^a		29.2 ± 8.1 ^{b,c}			
G-4	23	21	36.4 ± 12 ^a		46.8 ± 10.5 ^a			
G-5	20	17	44.0 ± 8.7 ^a		34.8 ± 11.9 ^c			
G-6	15	17	38.9 ± 11.4 ^a		34.5 ± 14.7 ^c			
G-7	18	15	38.3 ± 8.9 ^a		26.1 ± 10.4 ^b			

Table 1: Mean µTBS (MPa) results of enamel and dentin

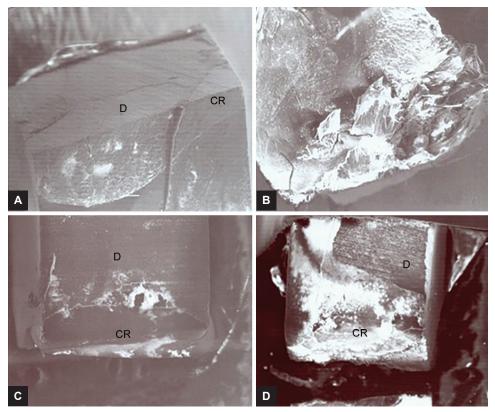
The different small letter superscripts in each column indicates the statistical significant difference between groups; SD: Standard deviation

 Table 2: Frequency of different modes of bond failure in the enamel–compomer and dentin–compomer beams

	Enamel			Dentin						
Groups	Adhesive	Cohesive	Mixed	Adhesive	Cohesive	Mixed				
G-1	12	2	7	13	8	-				
G-2	8	1	9	14	3	_				
G-3	13	4	5	14	2	_				
G-4	15	2	6	16	4	2				
G-5	16	3	1	15	2	_				
G-6	14	1	-	14	3	-				
G-7	16	-	2	15	-	_				

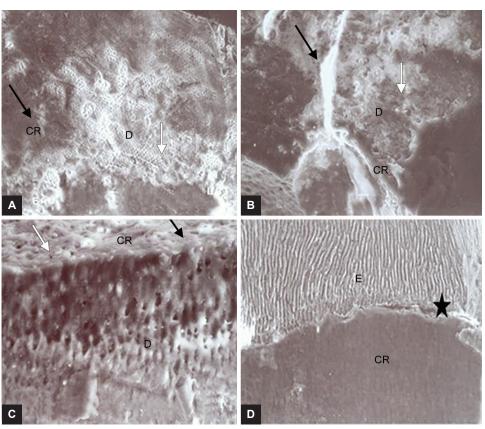


Figs 1A to D: Scanning electron microscopy images of the enamel rods (E: enamel; C: compomer resin). (A) ×40, indicator 100 μ m; (B) ×45, indicator 100 μ m; (C) ×60, indicator 100 μ m; and (D) ×43, indicator 100 μ m



Figs 2A to D: Scanning electron microscopy images of dentin rods (D: dentin; C: compomer resin). (A) ×65, indicator 100 μ m; (B) ×75, indicator 100 μ m; (C) ×50, indicator 100 μ m; and (D) ×47, indicator 100 μ m

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Figs 3A to D: Interfaces of dental hard tissue restorative materials (D: dentin; C: compomer resin). (A) ×270 indicator 100 μ m (black arrow: exposed dentin tissue, white arrows: compomer resin); (B) ×500, indicator 10 μ m (white arrow: compomer resin); (C) ×850, indicator 10 μ m; and (D) ×350, indicator 100 μ m (black star: tearing area)

this material to investigate the effects of radiation on the bond strength between this and the dental hard tissues.

Deterioration in the mechanical properties of enamel due to exposure to radiation is well recognized in adult teeth and depends on many variables.⁹ Studies investigating the effects of different doses of radiotherapy on restorative material bond strength in permanent teeth produced inconclusive results, with some researchers suggesting that radiotherapy increases bond strength, while others conclude that there is either a decrease or no change.⁵⁻⁷ There are currently no published studies evaluating similar effects of radiotherapy on the bonding strength of restorative materials to deciduous teeth. In this study, we investigated the effect of radiation on the µTBS between compomer resin and primary tooth enamel and dentin. We observed no significant difference in the µTBS of the compomer to enamel between the control group and any of the radiation groups, for which we believe that there are a number of possible contributory factors.

Presence and Radiation Resistance of Aprismatic Enamel Surfaces in Deciduous Teeth

Ripa et al¹⁰ showed the presence of aprismatic enamel in all primary teeth of children aged between 6 and

12 years. It has been reported that this aprismatic enamel surface varies based on the tooth type, age, ethnic background, and geographic region. We, therefore, attempted to neutralize this variability using the same tooth type (primary molars) from similarly aged children (9-12 years) living in the same geographical region. Grötz et al¹¹ applied lactic acid (pH 5) to radiation-exposed permanent teeth, found significant differences in the sensitivity of enamel to demineralization, and concluded that the enamel was less resistant to acid attack after exposure to radiation. In our study, irradiated and nonirradiated primary molars were demineralized with 34.5% phosphoric acid (with pH measured under laboratory conditions) before the application of a dentin-bonding agent. However, we did not detect any changes in bond strength, indicating that the radiation and control groups have similar resistance to acid attack. This might be due to the protective effect of the aprismatic enamel surface on primary teeth, which Kuhar et al¹² have reported to confound the demineralization process and that it theoretically protects the teeth against environmental damage. This is consistent with our study. Others have reported that aprismatic enamel can be dissolved in acid in 15 seconds, but we noted no difference between any of our groups even after

a 30-second "total-etch" step, and thus, hypothesize that radiation has no effect on the aprismatic enamel surface.¹³ Our finding that the enamel–compomer bond fails predominantly (69%) due to adhesive failure supports this hypothesis.

Lack of Effect of Radiation on Hydroxyapatite Crystals

Hydroxyapatite—an essential component of enamel—is reported to be unaffected by the application of radiation.¹⁴ The higher proportion of hydroxyapatite in enamel may explain why enamel is less affected by radiation than dentin.

Collagen, Which is affected by Radiation, is found at Much Lower Levels in the Interprismatic Area

Oliveira et al¹⁵ and Fosse have found that the density of enamel rods is higher in primary teeth than in permanent teeth. Conversely, rod diameter is smaller in primary teeth (3.22-3.47 µm for deciduous teeth vs 3.84-4.34 µm for permanent teeth), as is the interprismatic area.¹⁶ Organic matrix comprises 0.6% of the enamel in primary teeth, and the internal moisture appears to remain unchanged in teeth exposed to radiation in situ.¹⁷ The reactive OH and H ions that form water molecules are known to be initiators of radiation damage in organic material, particularly, in interprismatic enamel.¹⁸ Hence, with the greater proportion of organic material in deciduous teeth, it is logical that radiation will have a greater effect in these teeth, especially because, as described above, radiation has little effect on hydroxyapatite. Although deciduous teeth have more organic matrix and lower mineral content than permanent teeth, radiation did not cause any significant difference (p = 0.194) in µTBS between enamel and compomer.¹⁵ In contrast, there was a significant difference in μ TBS to dentin (p = 0.000). This difference might be due to variance in developmental origin: embryologically, the enamel develops from the ectoderm, while dentin develops from the mesenchyme, which means that dentin has more organic matrix than enamel.

Dentin collagens are highly hydrated due to their nature. Furthermore, acid etching degrades the dentin surface by disrupting inorganic mineral and some noncollagenous proteins. Consequently, the collagen fibers of demineralized dentin become exposed. This exposed matrix—if destabilized and/or denatured—is very soft and elastic so is susceptible to structural collapse. This is, especially, the case following air-drying, which dehydrates the collagen (micro) fibrils and deprives them of supportive water content.¹⁹ Collagen peptides form intermolecular H⁺ bonds with the closest collagen peptide, which may cause further collapse of the network, reducing the space between collagen fibers and limiting the penetration of resin monomer into these areas.²⁰ As a result, a uniform hybridized dentin layer cannot be achieved.²⁰ In our study, the etched dentin was rinsed with water and dried with polyurethane foam to eliminate these problems.

Dentin primers restore demineralized dentin. A hybrid layer—composed of resin polymers and collagen fibers—forms when monomers diffuse into the etched dentin surface. The physical properties of this layer are better than those of dentin; it is softer but tougher than normal dentin. Radiation applied after the resin restorative procedure affects this resin–dentin interface, whereas radiation applied before resin application affects the dentin collagen fibers. The radiation-induced changes in the dentin significantly reduce dentin hardness, wear resistance, and tensile strength; modify collagen and noncollagen structures; and alter the enamel–dentin composition.²¹⁻²³

Darchuk et al²³ have shown that higher doses of radiation lead to the destruction of dentin collagen chains, while Cheung et al²¹ characterized morphological and compositional changes in the intra- and intertubular dentinal collagen that modulated bonding to dentin.^{22,23} Free radicals that accumulate in irradiated dental tissues can affect the bonding mechanism.²⁴ Moreover, osteonectin (a specific noncollagenous glutamine- and phenylalanine-containing protein that links the hydroxyapatite and collagen phases) is also vulnerable to high doses of external radiation.²³ Together, these factors may explain the reduction in bonding strength between compomer resin and irradiated dentin and is consistent with the mainly adhesive type of failure observed in dentin in this study.

CONCLUSION

The application of different doses of radiotherapy to primary molar teeth has little effect on bond strength between compomer restorative material and enamel, but reduced the bond strength to dentin. It has been noted that radiotherapy application caused mainly adhesive failure in both enamel–compomer resin and dentin–compomer resin rods. In addition, there was a dose-dependent significant difference between the modes of failure for both enamel and dentin.

Clinical Significance

Radiotherapy may affect the success rate of compomer fillings in primary teeth, especially in deeper cavities with exposed dentin. Therefore, materials having a higher bond strength than compomer resin could be an alternative to restore the deeper cavities-exposed dentin in irradiated primary teeth.



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