Original Article

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# Bond strength of a universal adhesive with different etching strategies to irradiated dentin

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**Objectives:** To evaluate the effects of irradiation and etching strategies on the microtensile bond strength (μTBS) of a universal adhesive to dentin.

**Materials and methods:** Extracted human molars (n = 10) were divided into irradiated and non-irradiated groups. Five teeth received a single dose of 60 Gy, while the others served as controls. Mid-coronal dentin surfaces were prepared and bonded with a universal adhesive using etch-and-rinse (ER) and self-etch (SE) strategies. Resin composite build-ups were sectioned to obtain resin–dentin sticks. Three sticks per tooth were tested for μTBS at a crosshead speed of 1 mm/min. The mean value per tooth was used as the statistical unit. Data were analyzed using two-way ANOVA and Duncan test ($p < 0.05$). Failure modes were examined using scanning electron microscopy (SEM).

**Results:** Two-way ANOVA showed no significant effect of irradiation ($p = 0.529$) and no significant interaction between irradiation and etching strategy ($p = 0.884$). A significant effect of etching strategy was found, with higher μTBS in the ER groups compared with the SE groups ($p = 0.021$). However, one-way ANOVA with Duncan test showed no significant differences among the four groups ($p > 0.05$). The control and ER groups showed slightly higher mean values. Mixed failure predominated, followed by adhesive and cohesive failure within the resin composite, whereas cohesive failure in dentin was rare.

**Conclusion:** The etch-and-rinse approach provided higher bond strength than the self-etch approach regardless of irradiation.

**Keywords:** irradiation, microtensile bond strength, universal adhesive

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## Introduction

Head and neck cancer is a major global health problem that includes malignancies of the oral cavity, pharynx, larynx, nasal cavity, and salivary glands [1, 2]. Radiotherapy is a commonly used treatment modality and plays an important role in both early and advanced stages of the disease [3]. However, this treatment can affect the structure of dental tissues and lead to changes in both enamel and dentin.

Irradiation induces microstructural and chemical alterations in dental hard tissues [4]. Enamel may exhibit increased porosity, mineral loss, and structural defects. Dentin is more susceptible due to its higher organic content [5-7]. Reported changes include collagen degradation, obliteration of dentinal tubules, and reduced mechanical properties [7, 8]. These alterations may compromise the bonding performance of adhesive systems and reduce the longevity of

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restorative treatments. Radiotherapy is also associated with adverse oral effects, including mucositis, xerostomia, microbial imbalance, and radiation-related caries [9].

Radiation-related caries is one of the most clinically significant complications in irradiated patients and mainly affects cervical and root dentin. These changes, together with salivary dysfunction, contribute to rapid caries progression. As a result, dentin becomes the primary substrate for restoration, and durable adhesive bonding becomes essential [10]. Despite these challenges, there is no clear recommendation regarding the most appropriate adhesive strategy for irradiated teeth. The choice between etch-and-rinse (ER) and self-etch (SE) techniques remains unclear [11, 12].

Clinical studies in irradiated patients are limited due to difficulties in specimen collection and limited experimental control. For this reason, *in vitro* models are commonly used to simulate the effects of irradiation under controlled conditions [13-15]. Previous studies have suggested that a single-dose irradiation protocol may be used as a substitute for fractionated irradiation in laboratory settings.

Universal adhesives are widely used due to their versatility and simplified application. Palfique Universal Bond (Tokuyama Dental, Japan) is of particular interest because of its favorable properties. It requires fewer clinical steps and can reduce chair time, which may be beneficial for patients with limited tolerance for dental procedures [16]. Its self-curing capability allows polymerization in areas with limited light penetration, which may improve bonding performance in compromised conditions such as irradiated teeth. However, the bonding performance of this material under irradiation conditions remains unclear.

Therefore, this study aimed to evaluate the effect of irradiation and etching strategies on the microtensile bond strength of a universal adhesive under *in vitro* conditions. The null hypotheses of this study were that (1) there would be no significant difference in bond strength between irradiated and nonirradiated teeth and (2) there would be no significant difference between etch-and-rinse and self-etch strategies.

## Materials and Methods

### Tooth selection and grouping

This study was conducted following ethical approval from the Ethics Committee (No. MU-DT/PY-IRB 2025/DT163). Extracted human molars (n = 10) were collected from patients for whom extraction was clinically indicated. Only sound teeth without caries, restorations, cracks, or structural defects were included. The teeth were divided into two groups. Five teeth were assigned to the irradiated group and exposed to a single dose of 60 Gy (VT). The remaining five teeth served as the nonirradiated control group (CT). All specimens were stored in 0.5% chloramine-T solution at 4°C and used within six months.

Sample size was calculated using G*Power software (version 3.1.9.7) based on a fixed-effects two-way ANOVA ($\alpha = 0.05$, power = 0.80). In the absence of prior pilot data, a large effect size ($f = 0.40$) according to Cohen's conventions [17] was assumed. The analysis indicated a minimum sample size of five teeth per group. The tooth was considered the experimental unit. This study was conducted as an exploratory in vitro investigation.

### Specimen preparation

The specimen preparation process is

illustrated in Figure 1. Each tooth was sectioned at the mid-coronal dentin using a low-speed diamond saw (IsoMet 1000, Buehler, USA). The exposed dentin surfaces were polished with 600-grit silicon carbide paper under running water for 60 seconds to obtain a standardized smear layer [18]. Each specimen was divided into two halves and then treated using two etching strategies: etch-and-rinse (ER) and self-etch (SE) modes. Palfique Universal Bond (Tokuyama Dental, Japan) was applied according to the manufacturer's instructions Table 1. Resin composite build-ups were constructed using CLEARFIL™ AP-X ES-2 (Kuraray Noritake Dental Inc., Japan) in two 2-mm increments and light-cured for 20 seconds using a light-curing unit (Bluephase G2, Ivoclar Vivadent, Liechtenstein). The specimens were stored in distilled water at 37°C for 24 hours prior to testing.

## Microtensile bond strength (μTBS) testing

Each bonded tooth was sectioned to obtain resin–dentin sticks with a cross-sectional area of approximately 1 × 1 $mm^2$, as illustrated in Figure 1. Three sticks from each tooth were selected for μTBS testing. Each stick was attached to a testing jig using cyanoacrylate adhesive and subjected to tensile load in a universal testing machine at a crosshead speed of 1 mm/min until failure. The μTBS value was calculated by dividing the maximum load at failure by the bonded area. The mean bond strength per tooth was calculated and used as the statistical unit to avoid pseudo-replication [18, 19].

## Failure mode analysis

Following the μTBS testing, all fractured specimens were carefully removed from the jig to

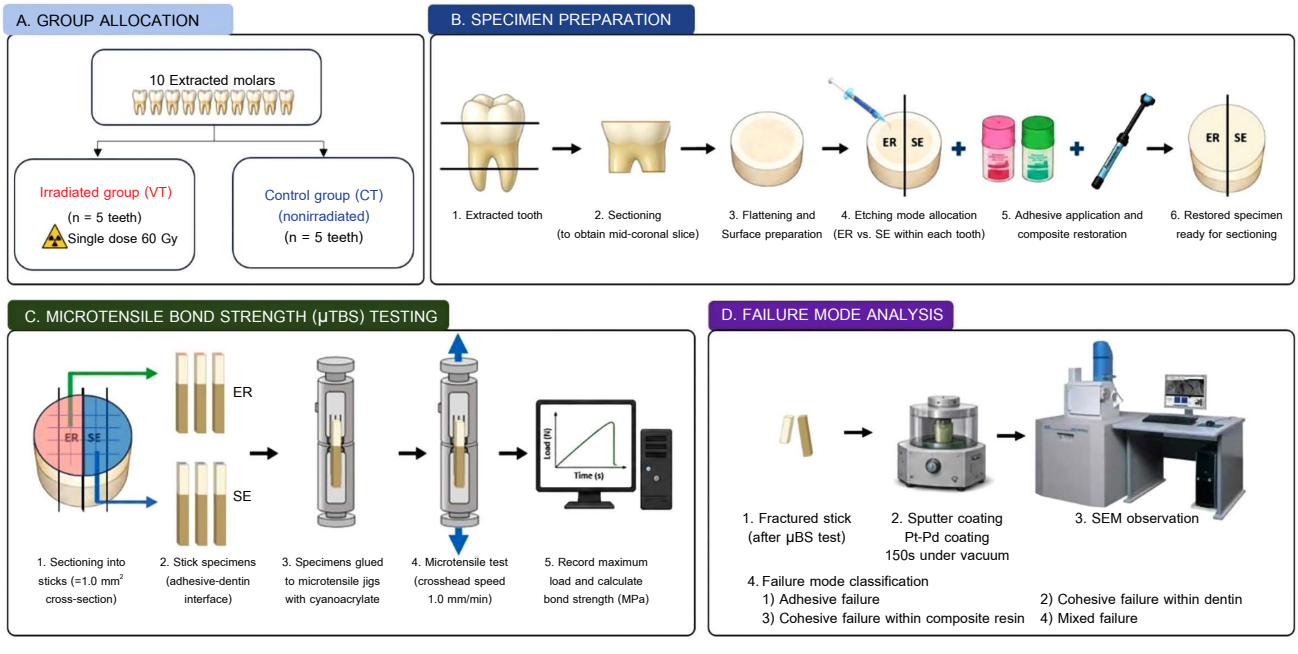



**Figure 1** Schematic illustration of the experimental design including (A) group allocation, (B) specimen preparation, (C) microtensile bond strength (μTBS) testing, and (D) failure mode analysis. Not: ER = Etch-and-Rinse; SE = Self-Etch; VT = Irradiated group; CT = Control group; μTBS = Microtensile bone strength; Gy = Gray (radiation dose).

Table 1 Adhesive components of Palfique Universal Bond and application procedures

| Adhesive Component | pH | Composition | Application procedure |
|---|---|---|---|
| Palfique Universal Bond (Tokuyama Dental Corporation, Tokyo, Japan; 492E75) | Mild 2.0 | **Bond A**<br>Phosphoric acid monomer (New 3D-SR monomer)<br>MTU-6<br>HEMA<br>Bis-GMA<br>TEGDMA<br>Acetone<br>**Bond B**<br>γ-MPTES<br>Borate<br>Peroxide<br>Acetone<br>Isopropyl alcohol<br>Water | **Self-etch mode**<br>– Mix component A and B according to manufacturer instructions<br>– Apply the adhesive to dentin<br>– Apply weak air continuously to the surface until the runny bond stays in the same position without any movement, then mild air to the surface<br>-Restore with a composite<br>**Etch-and-rinse mode**<br>– Apply phosphoric acid gel onto the prepared enamel first, and then the dentin(10-15seconds)<br>– Rinse thoroughly (at least 15 seconds) with water and dry with mild air or with cotton pellets; do not overdry<br>– Mix component A and B according to manufacturer instructions<br>– Apply the adhesive to dentin<br>– Apply weak air continuously to the surface until the runny bond stays in the same position without any movement, then mild air to the surface<br>-Restore with a composite |

MTU-6: 6-methacryloyloxyhexyl 2-thiouracil- 5-carboxylate, HEMA :2-Hydroxyethyl methacrylate, Bis-GMA: bisphenol A-glycidyl methacrylate, TEGDMA:triethylene glycol dimethacrylate, γ-MPTES: γ-methacryloxypropyl triethoxy silane

prevent mechanical damage unrelated to bond failure. Each specimen was mounted on aluminum stubs, sputter-coated with a platinum–palladium alloy for 150 seconds under vacuum, and subsequently examined using scanning electron microscopy (SEM; JSM-6610LV, JEOL Ltd., Tokyo, Japan). Failure modes were classified as adhesive, cohesive in dentin, cohesive in resin composite, or mixed [18].

## Statistical analysis

Data normality and homogeneity of variance were assessed. Data were then analyzed using two-way analysis of variance (ANOVA) followed by Duncan test to evaluate the effects of irradiation condition, etching strategy, and their interaction. The level of significance was set at $p < 0.05$. Statistical analyses were conducted using IBM SPSS Statistics for Windows, Version 31.0.1.0 (IBM Corp., Armonk, NY, USA).

## Results

The microtensile bond strength (μTBS) values for all experimental groups are presented in Table 2. Irradiation had no statistically significant effect on μTBS ($p = 0.529$). There was no significant interaction between irradiation and etching strategy ($p = 0.884$), indicating that the effect of adhesive strategy was consistent across irradiation conditions. However, etching strategy had a statistically significant effect on μTBS ($p = 0.021$). The ER groups demonstrated higher mean bond strength values than the SE groups under both irradiated and nonirradiated conditions.

Failure mode distribution is presented in Figure 2. Mixed failure was the predominant mode across all groups (55%), followed by adhesive failure (23.3%) and cohesive failure within the resin composite (20%). A small proportion of cohesive failure within dentin was also observed (1.7%).

Table 2 Microtensile bond strength (MPa) (mean ± SD) of all groups

| Etching strategy | Microtensile Bond Strength (MPa) | |
|---|---|---|
| | CT | VT |
| Etch-and-rinse | 43.81±10.62[a] | 40.82±4.60[a] |
| Self-etch | 33.57±7.91[b] | 31.70±9.42[b] |

Note: Values with the same superscript lowercase letter indicate no statistically significant difference according to Duncan test ($p > 0.05$).

When analyzed by group, mixed failure was most frequent in all experimental conditions, with the highest percentage observed in the irradiated etch-and-rinse group (VT-ER, 66.7%). The control etch-and-rinse group (CT-ER) showed a relatively higher proportion of cohesive failure within the resin composite (40%), whereas the control self-etch group (CT-SE) exhibited a more distributed pattern among failure types. Cohesive failure within dentin was rare and observed only

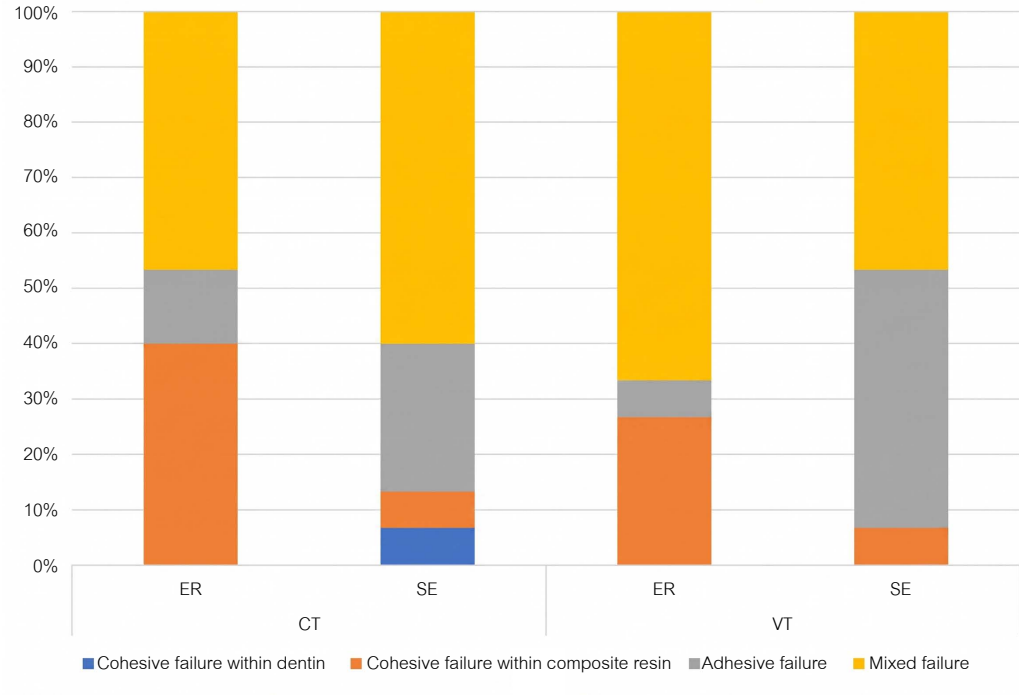



Figure 2 Analysis of the percentages of failure modes.

in the CT-SE group. Although mixed failure predominated, some variations in failure mode distribution were observed among groups.

## Discussion

This study evaluated the effect of irradiation and etching strategy on the microtensile bond strength of a universal adhesive system. The results demonstrated that irradiation did not significantly influence bond strength, whereas etching strategy showed a significant main effect. Therefore, the null hypothesis regarding irradiation was not rejected, while the null hypothesis for etching strategy was rejected.

Previous studies have reported that irradiation may reduce dentin bond strength due to structural and chemical changes, including collagen degradation, alterations in mineral composition, and increased dentin permeability. Collagen degradation may reduce resin infiltration and compromise hybrid layer integrity. Changes in mineral composition and tubular occlusion may reduce dentin permeability and limit adhesive diffusion. Increased brittleness may promote stress concentration at the adhesive interface [20]. Irradiation is also associated with radiation-related caries, particularly in cervical and root dentin, and with salivary dysfunction, which may further compromise bonding performance in clinical situations. However, irradiation did not significantly influence bond strength in the present study, and the findings should be interpreted with caution.

The bonding performance may be influenced by the adhesive system. The universal adhesive employed in this study contains a 3D SR monomer that can interact with calcium ions in hydroxyapatite and enhance micromechanical and chemical bonding within the hybrid layer. The borate-based initiator system may support polymerization under acidic conditions [21]. In addition, the adhesive shows self-curing capability, which may reduce its dependence on substrate condition and allow stable polymerization even when the dentin structure is altered. Previous studies investigating different radiation doses and adhesive systems have shown that irradiation-induced changes in enamel and dentin do not necessarily reduce bond strength. The bonding performance may depend on the adhesive system and experimental conditions, which may explain why irradiation did not significantly reduce bond strength in the present study [22].

The higher bond strength in the etch-and-rinse groups may be due to the effective removal of the smear layer and deeper demineralization, which allows better resin infiltration and hybrid layer formation [23, 24]. In contrast, the self-etch strategy produces shallower demineralization, which may limit resin penetration and reduce bonding effectiveness.

Previous studies have shown that universal adhesives can maintain bond performance on compromised substrates. The use of a dual-cure activator has been reported to improve bonding to bleached enamel and preserve interfacial integrity despite structural changes [25]. This suggests that certain adhesive systems including Palfique Universal Bond may be less sensitive to substrate alterations and can maintain bonding performance under compromised conditions.

The absence of a significant reduction in bond strength may be related to irradiation protocols and experimental conditions. A single-dose irradiation model of 60 Gy was used, which may induce less pronounced alterations compared to fractionated clinical radiotherapy. This protocol was selected due to limited access to irradiation facilities and specimens. Although

previous studies have suggested that single-dose irradiation can simulate cumulative radiation effects, this model may underestimate the progressive damage observed in clinical fractionated radiotherapy.

Failure mode analysis supported the bond strength results. Mixed failure was predominant, indicating adequate bonding performance. Some variations were observed among groups, but cohesive failure in dentin was rare, suggesting that dentin integrity was not severely compromised.

This study has limitations. It was conducted in vitro and cannot fully replicate clinical conditions. The use of a single-dose irradiation model may not fully represent clinical radiotherapy [26]. Mid-coronal dentin was used for standardization, but it does not reflect cervical and root dentin lesions in irradiated patients. Therefore, applicability to root caries and cervical lesion restoration in xerostomic patients should be interpreted with caution.

Future studies should include larger sample sizes, long-term aging, and evaluation of root dentin under clinically relevant conditions.

## Conclusion

Within the limitations of this study, irradiation did not significantly affect bond strength. The etch-and-rinse approach produced higher bond strength than the self-etch approach, regardless of irradiation condition.

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## Declarations

### Ethical approval and consent to participate

The study followed the Declaration of Helsinki and received ethical approval from the Mahidol University Institutional Review Board (MU-DT/PY-IRB2025/DT163). Extracted teeth were collected with all personal identifiers removed to ensure confidentiality.

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