

Effects of Ionizing Radiation on the Shear Bond Strength of Composite Materials to Dentin

Mohenski, Dora; Vrebac, Mihaela; Sever, Eva Klarić; Grego, Timor; Goršeta, Kristina; Ivanišević, Ana

Source / Izvornik: **Journal of Composites Science, 2024, 8**

Journal article, Published version

Rad u časopisu, Objavljena verzija rada (izdavačev PDF)

<https://doi.org/10.3390/jcs8070261>

Permanent link / Trajna poveznica: <https://urn.nsk.hr/urn:nbn:hr:127:711112>

Rights / Prava: [Attribution 4.0 International](#)/[Imenovanje 4.0 međunarodna](#)

Download date / Datum preuzimanja: **2025-02-10**



Repository / Repozitorij:

[University of Zagreb School of Dental Medicine
Repository](#)



Article

Effects of Ionizing Radiation on the Shear Bond Strength of Composite Materials to Dentin

Dora Mohenski ¹, Mihaela Vrebac ², Eva Klarić Sever ³, Timor Grego ⁴, Kristina Goršeta ^{3,*}  and Ana Ivanišević ^{3,*} 

¹ Private Dental Practice, 10000 Zagreb, Croatia; zagorac.dora@gmail.com

² Private Dental Practice, 4020 Linz, Austria; mihaela.os161@gmail.com

³ School of Dental Medicine, University of Zagreb, 10000 Zagreb, Croatia; eklaric@sfzg.hr

⁴ Department of Oncology, University Hospital Centre Zagreb, 10000 Zagreb, Croatia; tgrego@kbc-zagreb.hr

* Correspondence: gorseta@sfzg.hr (K.G.); aivanisevic@sfzg.hr (A.I.); Tel.: +385-1-4802-193 (A.I.)

Abstract: Ionizing radiation in therapeutic doses alters the composition and properties of dentin and resin composites. This may influence the adhesion of restorative materials to irradiated dentin and compromise the success of the restorative treatment. The objective of this study was to evaluate the effect of ionizing radiation on the shear bond strength (SBS) of bulk-fill composite materials to dentin. Coronal dentin slabs ($N = 90$) were embedded in acrylate and randomly assigned to six groups ($N = 15$) depending on the time of radiation (70 Gy) and material (SDR Plus Bulk Fill Flowable and Tetric EvoFlow Bulk Fill (TET)): (1) control group (CG) SDR; (2) CG TET; (3) radiation + SDR; (4) radiation + TET; (5) SDR + radiation; and (6) TET + radiation. Composite cylinders were bonded to the dentin slabs using Scotchbond Universal Plus Adhesive. The specimens were stored in distilled water and fractured in shear mode after 7 weeks. Radiation before and after restoration resulted in an SBS decrease. The SBS was statistically significantly lower in groups 5 and 6 ($p < 0.05$). The difference between the bulk-fill composites was not significant ($p > 0.05$). In the CGs, adhesive fractures prevailed. In groups 3 and 4, cohesive fractures in the dentin were more frequent, and in groups 5 and 6, cohesive fractures in the material. Radiotherapy affects the SBS of bulk-fill composites to dentin. Immediate radiation after restoration resulted in the lowest SBS in both bulk-fill composite materials.

Keywords: bulk-fill composites; dentin; head and neck neoplasms; radiotherapy; shear bond strength



Citation: Mohenski, D.; Vrebac, M.; Sever, E.K.; Grego, T.; Goršeta, K.; Ivanišević, A. Effects of Ionizing Radiation on the Shear Bond Strength of Composite Materials to Dentin. *J. Compos. Sci.* **2024**, *8*, 261. <https://doi.org/10.3390/jcs8070261>

Academic Editor: Salvatore Brischetto

Received: 30 April 2024

Revised: 25 June 2024

Accepted: 3 July 2024

Published: 5 July 2024



Copyright: © 2024 by the authors. Licensee MDPI, Basel, Switzerland. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license (<https://creativecommons.org/licenses/by/4.0/>).

1. Introduction

Head and neck cancers (HNCs) include cancers of the lips and oral cavity, salivary glands, pharynx and larynx. They are responsible for more than 900,000 new cases and more than 340,000 deaths per year [1]. The treatment of HNCs usually involves radiation therapy (RT). Ionizing radiation causes the formation of free radicals that disrupt biochemical processes in malignant cells, the replication of deoxyribonucleic acid (DNA) and the regulation of the cell cycle, ultimately leading to cell death. Despite improved irradiation techniques, high-energy radiation used for therapeutic purposes has adverse effects on the surrounding tissues. Healthy surrounding tissues with a high degree of mitotic activity, such as the oral mucosa, are affected first [2]. Mucositis and dermatitis are therefore the main acute side effects of RT, while complications related to the salivary glands (apoptosis) and masticatory muscles (fibrosis) typically begin several weeks to months after RT and manifest clinically as dry mouth and trismus. There are several delayed complications: osteoradionecrosis, mucosal necrosis, dystrophic soft tissue calcification, radiation caries and others [2,3].

The causes of radiation caries include reduced saliva volume and altered saliva composition as well as altered oral flora with an increased number of acidogenic bacteria [4]. In addition, the characteristic features of radiation caries, such as exposed dentin due to the initial breakage of enamel on cervical and labial surfaces, cusps and incisal ridges,

can be attributed to the direct detrimental effects of RT on dental tissues [5–7]. Indeed, ionizing radiation at therapeutic doses has been shown to affect the mechanical properties of enamel and dentin, with the enamel–dentin junction (EDJ) being particularly affected, even at subtherapeutic cumulative doses of 30 Gy [8]. The early destruction of the EDJ and the enamel near the EDJ in conjunction with the reduced enamel crystallinity due to radiation may be associated with the formation of the characteristic radiation caries [8]. The changes in dental tissues' properties and aggressive radiation caries require monitoring, intervention and effective restorative treatments.

Direct adhesive restoration with dental composites is the main treatment option for caries-related tooth tissue loss. The adhesion of dental composites to dental tissues is achieved by infiltration and micromechanical interlocking of dental adhesives in the microspaces obtained by etching (H_3PO_4 in Etch&Rinse (ER) or acidic monomers in the Self-Etch (SE) technique), which leads to the formation of a hybrid layer in the dentin [9–11]. The stability of the hybrid layer depends, among other things, on the protocol used for tissue preparation (ER or SE), the infiltration capacity of the monomers of the adhesive system and the conformation of the polymer network [9]. The adhesive bond between the dental tissue and the restoration fails over time, which can lead to the accumulation of oral biofilm and the development of secondary caries [9,10]. This can be particularly problematic in irradiated patients [12]. In order to achieve proper adhesion and durability of the interfacial layer, the structural characteristics of the substrate tissue (dentin and enamel) and the changes in their composition and mechanical properties caused by radiotherapy must be taken into account [12,13]. According to the available literature, there is no consensus on the ideal time for placement of the restoration, nor on whether irradiation affects the bond strength of the restorative material (within 24 h) [13–21]. The influence of ionizing radiation on the bond strength of ER and SE adhesives has been extensively investigated [14–21]. Some studies report a reduction in the bond strength to enamel and/or dentin, particularly when irradiation is applied prior to restoration [14–19], while other studies find no significant reduction in the bond strength [20,21]. There are few studies on the effects of radiotherapy on the bond strength of universal adhesives, and the results are also inconsistent [21,22]. According to Ugurlu et al. [22], radiotherapy reduces the bond strength of eighth-generation universal adhesives to dentin, whereas according to da Cunha et al. [21] and Oglakci et al. [23], it does not significantly affect the bond strength. Jacker-Guhr et al. [24] compared the shear bond strength of different universal adhesives to enamel and dentin with and without additional etching with phosphoric acid, and their results showed that the bond strength to dentin differed between the different universal adhesives when they were applied in self-adhesive mode. This suggests that the composition of the adhesives influences the bond strength. Therefore, the choice of the Scotchbond Universal Plus adhesive, which contains 3M's proprietary Vitrebond® copolymer and which has not yet been investigated in radiation and bond strength studies, is of great value for the present study. In addition, previous studies have shown that the chemical composition influences the mechanical and biological behavior of conventional, bulk-fill and self-adhesive composite materials, including the bond strength to dentin [25,26]. There are certain differences between Tetric EvoFlow Bulk Fill and SDR Plus Bulk Fill Flowable in terms of the monomer content and filler quantity, with SDR Plus having a higher filler load and a proprietary modified urethane dimethacrylate resin with a higher molecular mass. For this reason, it was considered useful to investigate the influence of ionizing radiation on the bond strength of both bulk-fill materials.

The aim of this study was to investigate the effect of ionizing radiation corresponding to a cumulative therapeutic dose of 70 Gy before and after restoration on the bond strength of universal adhesive and two flowable bulk-fill composites to dentin. The null hypothesis was that irradiation would not significantly affect the bond strength, regardless of the time of irradiation—before or after the restoration. Another null hypothesis was that the differences between the restorative materials would not be significant.

2. Materials and Methods

Forty intact impacted or semi-impacted third molars were used in this study. The patients provided their written consent for surgical extraction, which was medically indicated. After extraction, the teeth were disinfected in 1% chloramine solution for three days and then stored in saline solution for a maximum of three months until the start of the experiment. The use of the extracted teeth in this study was approved by the Ethics Committee of the School of Dental Medicine, University of Zagreb, 05-PA-30-155-22/2023.

The dentin substrates were created using a low-speed saw (IsoMet, Buehler; Lake Bluff, IL, USA) at 300 rpm and continuous water cooling. The crowns were sectioned perpendicular to the longitudinal axis of the tooth into 2.2 mm thick dentin slabs. The sections from the mid-coronal region were cut in half and the slabs were kept in saline until mounting in a cold-curing methacrylate resin (Technovit 4004, Kulzer, Germany) using the Ultradent mold (Ultradent Products, South Jordan, UT, USA). To create a flat bonding area on the coronal surface of the embedded dentin slabs, the specimens were polished with 600 grit silicon carbide (SiC) paper (PRESI, Eybenes, France) and stored in distilled water until the bonding procedure. A total of 90 samples were prepared and randomly divided into four experimental groups and two control groups (CG) of 15 samples each, depending on the bulk-fill composite material and time of dentin irradiation (before or after bonding of the composite cylinder to the dentin). The materials used were SDR Plus Bulk Fill Flowable (SDR, Dentsply DeTrey GmbH, Konstanz, Germany) and Tetric EvoFlow Bulk Fill (TET, Ivoclar Vivadent, Schaan, Liechtenstein). The same adhesive, 3M Scotchbond Universal Plus Adhesive (3M ESPE, Neuss, Germany), was used for all the samples (Table 1).

Table 1. Materials used in the study.

| Material | Type | Chemical Formulation * | Manufacturer and LOT No. |
|---------------------------------------|------------------------------------|---|---|
| SDR Plus Bulk Fill Flowable (SDR) | Bulk-fill flowable resin composite | Modified UDMA, Bis-EMA, TEGDMA, barium-alumino-fluoro-borosilicate glass, strontium alumino-fluoro-silicate glass, CQ photoinitiator, photoaccelerator, BHT, UV stabilizer, titanium dioxide, iron oxide pigments fluorescing agent. Filler load: 70.5 wt%, 47.4 vol% | Dentsply Sirona, Konstanz, Germany LOT: 2101000559, 2208000286 |
| Tetric EvoFlow Bulk Fill (TET) | Bulk-fill flowable resin composite | Bis-GMA, Bis-EMA, UDMA, barium-alumino-silicate glass, ytterbium trifluoride, copolymers, mixed oxide. Filler load: 68.2 wt%, 46.4 vol% | Ivoclar Vivadent, Schaan, Liechtenstein LOT: Z00V4H |
| 3M Scotchbond Universal Plus Adhesive | Adhesive, universal | 10-MDP, dimethacrylate resins, HEMA, Vitrebond copolymer, silica filler, ethanol, water, initiators based on CQ, silane, dual-cure accelerator | 3M ESPE, Neuss, Germany LOT: 8039902 |

* According to the manufacturers' information. UDMA: urethane dimethacrylate; Bis-EMA: ethoxylated bisphenol A dimethacrylate; TEGDMA: triethylene glycol dimethacrylate; CQ: camphorquinone; BHT: butylated hydroxyl toluene; BisGMA: bisphenol-A glycidyl methacrylate; 10-MDP: 10-methacryloyloxydecyl dihydrogen phosphate; HEMA: 2-hydroxyethyl methacrylate.

There were six groups in total ($n = 14-15$): (1) SDR CG; (2) TET CG; (3) irradiation + SDR; (4) irradiation + TET; (5) SDR + irradiation; and (6) TET + irradiation. The control groups were not exposed to irradiation. The samples from experimental groups 3 and 4 were exposed to ionizing radiation at a daily dose of 2 Gy, 5 days a week, for 7 weeks. A total of 35 fractures were performed according to the treatment protocol for radiotherapy of head and neck cancer, which corresponds to a cumulative dose of 70 Gy. The samples were stored in distilled water for the entire time. The radiation dose for oral cavity cancers

ranges from 70 Gy for gross disease, 60–66 Gy for high-risk regions, and 50–54 Gy for low-risk areas [27]. In research on the effect of radiation on dental tissues and restorative materials, the maximum ionizing radiation dose ranged from 60 to 80 Gy [7,8,13,23].

Two hours after the last ionizing radiation fracture in groups 3 and 4, the bonding procedure was performed in all the groups. The dentin surface was gently air-dried and the bonding site was marked with a self-adhesive polymer tape with a 2.4 mm diameter hole and a thickness of 0.2 mm. The Scotchbond Universal Plus adhesive was applied according to the manufacturer's instructions (application 20 s, drying 5 s and curing 10 s). Bulk composite cylinders (d = 2.38 mm, h = 4.0 mm) were created on the bonding surface using a clamp and Teflon mold inserts according to the UltraTester (Ultradent Products, SAS Institute Inc., Cary, NC, USA). A Bluephase Style LED lamp (Ivoclar Vivadent, Schaan, Liechtenstein) with an intensity of 1100 mW/cm² was used to light-cure the adhesive (10 s) and the composite (20 s).

After bonding the composite cylinders to the dentin slabs in acrylate, the specimens were returned to distilled water. The samples from groups 5 and 6 were exposed to the same irradiation protocol as the samples from groups 3 and 4. After 7 weeks, the shear bond strength was tested according to the ISO 29022 standard [28] using the UltraTester device. The UltraTester contains a special notched crosshead that allows a larger surface area of the sample to come in contact with it compared to other shear tests. During the bond strength test, the sample was placed in a holder. The notched crosshead surrounded half of the sample and was located at the junction between the surface of the dentin and the composite material. There were 15 specimens in each group, but during the SBS testing, one specimen in group 4 (irradiation + TET) was lost, so the final number of samples in group 4 was 14. The test was performed by loading the sample at a constant speed of 1 mm/min until the adhesive bond broke, i.e., until the composite cylinders detached from the dentin surface. The measurement was carried out in accordance with ISO 29022. The shear bond strength values were calculated according to the equation

$$\sigma = F/A$$

where σ (MPa)—shear bonding strength, F (N)—breaking force, i.e., the maximum force at which breakage occurred, and A (mm²)—bonding surface.

The recorded shear bond strength values in MPa were analyzed using the two-way ANOVA test and the Tukey HSD post hoc test to determine statistically significant differences between the groups. Statistical analysis was performed using the SPSS 23.0 program (SPSS Inc., Chicago, IL, USA). The statistical significance level was set at $\alpha = 0.05$.

After fracture in shear mode with the UltraTester, all the fractured samples were viewed under a Dino-Lite AM4113T digital microscope (Dino-Lite products, Almere, the Netherlands) to determine the type of fracture. The optical magnification was 10–200 \times and the resolution was 1.3 megapixels.

3. Results

3.1. Shear Bond Strength of Bulk-Fill Composite Materials to Dentin

Both materials showed a similar trend in terms of the SBS reduction in the irradiated samples. The mean SBS was highest for the SDR and TET CGs: 29.607 ± 5.3494 MPa and 23.553 ± 6.3709 MPa, respectively. The SBS was lowest in the groups irradiated after restoration and was 18.247 ± 8.0667 MPa and 16.693 ± 8.2235 MPa for the SDR and TET groups, respectively (Figure 1).

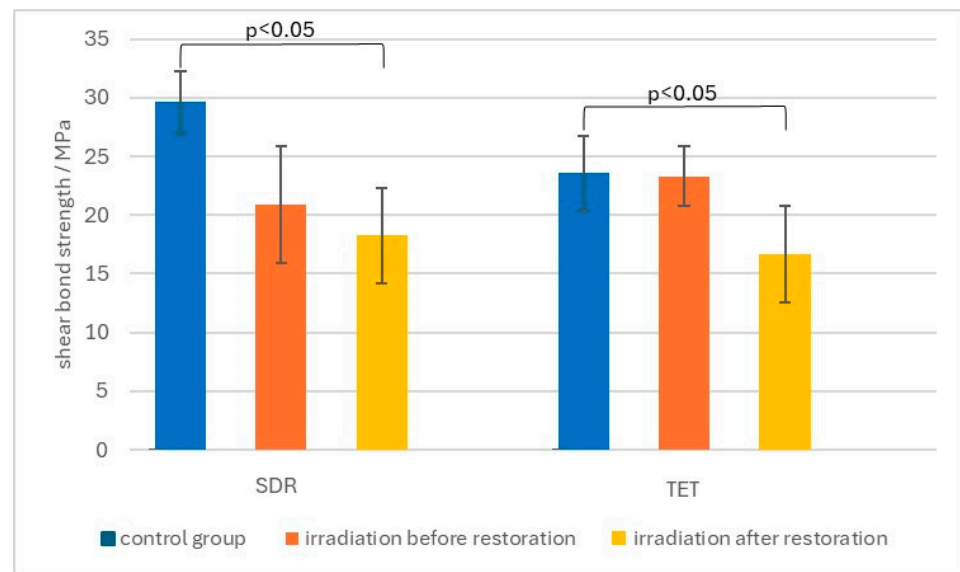


Figure 1. Mean shear bond strength values in different experimental groups. SDR = SDR Plus Bulk Fill Flowable; TET = Tetric EvoFlow Bulk Fill. The difference regarding the shear bond strength was statistically significant between the control groups and groups irradiated after restoration for both materials tested.

The ANOVA analysis showed that the difference in the SBS between the SDR and TET bulk materials was not statistically significant regardless of the irradiation factor ($df = 1$, $F = 1.225$, $p = 0.272$). Different irradiation regimes had a significant effect on the SBS, independent of the effect of the material factor ($df = 2$, $F = 11.383$, $p < 0.001$). Furthermore, there was no interaction effect between the material factor and the irradiation factor ($df = 2$, $F = 2.412$, $p = 0.096$).

The Tukey HSD post hoc test showed that the CGs of both materials exhibited statistically significantly higher SBS than the groups exposed to irradiation after restoration ($p < 0.05$). Furthermore, the SBS in the CGs did not differ significantly from the SBS in the groups exposed to radiation before restoration ($p > 0.05$). The differences between the groups irradiated before and after restoration were also not statistically significantly different (Figure 1).

3.2. Failure Mode Analysis

The type of fracture was determined for each sample. The fractures were categorized as adhesive (fracture in the adhesive layer only, A), mixed adhesive and cohesive in dentin (ACD), mixed adhesive and cohesive in material (ACM) and mixed cohesive in dentin and material (MC) (Figure 2). The proportion of a particular fracture in a particular group is expressed as a percentage. It was observed that adhesive fractures predominated in the control groups (73% and 67% for SDR and TET, respectively). In the groups that were irradiated before restoration, adhesive fractures still predominated (60% for SDR and 50% for TET), but the proportion of mixed fractures increased: ACD and MC accounted for 40% of all the fractures in SDR and 50% of all the fractures in TET. In the groups irradiated after restoration, the proportion of adhesive fractures was significantly lower (40% and 13% for SDR and TET, respectively). The proportion of fractures involving cohesive fractures in material (combined with either adhesive or cohesive fractures in dentin) was the highest in the groups irradiated after restoration: ACM and MC accounted for 53% of all the fractures in SDR and 80% of all the fractures in TET (Figure 3).

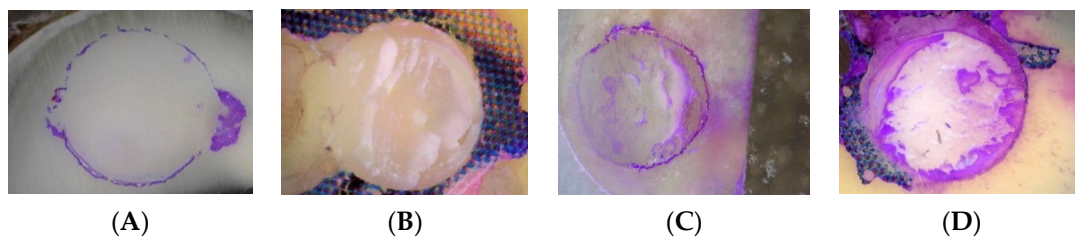


Figure 2. Representative fractures: (A) adhesive, (B) mixed adhesive and cohesive in dentin (ACD), (C) mixed adhesive and cohesive in material (ACM), and (D) mixed cohesive (MC).

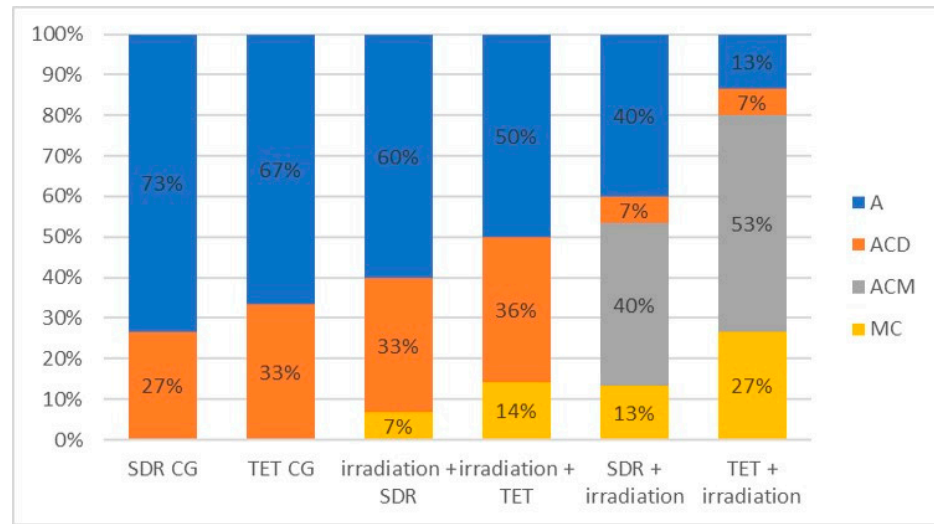


Figure 3. Fracture type distribution in different groups: SDR—SDR Plus Bulk Fill Flowable, TET—Tetric EvoFlow Bulk Fill and CG—control group. Fractures: A = adhesive; ACD = mixed adhesive and cohesive in dentin; ACM = mixed adhesive and cohesive in material, MC = mixed cohesive in dentin and material.

4. Discussion

The treatment of head and neck cancers requires a multidisciplinary approach in which radiotherapy is the main therapeutic procedure or postoperative adjuvant therapy [29]. Ionizing radiation used in the treatment of head and neck cancers leads to numerous side effects, including changes in the structure of mineralized dental tissues [30]. These changes can affect the bond between a restorative material and dental tissues and can contribute, among other factors, to the development of radiation caries [30]. These changes may impair the bond between a restorative material and the tooth tissues and contribute to the development of radiation caries, among other factors. Indeed, the results of the present study showed that radiation affects the SBS of universal adhesive and bulk-fill restorative materials to coronal dentin. Irradiation prior to restoration did not lead to a significant reduction in the SBS. However, the SBS was significantly lower when the samples were irradiated after restoration. The first null hypothesis was therefore rejected. The difference in the SBS between the SDR and TET bulk material was not statistically significant regardless of the irradiation factor, so the second null hypothesis was confirmed.

The decrease in the bond strength when dentin was irradiated before restoration (although not statistically significant) may be explained by the altered structure and composition of the dentin. In fact, numerous studies have shown morphological changes in enamel and dentin, as well as changes in the organic and inorganic components of mineralized dental tissues, after ionizing radiation at therapeutic doses [15]. Ionizing radiation leads to the radiolysis of water, a process that has several steps and results in the creation of H₂, H₂O₂, electrons, H₃O⁺ ions and unstable species such as hydrogen atoms H[·] and

hydroxyl radicals OH \cdot [15,31,32]. Free radicals take electrons (they reduce themselves and oxidize the tissue) primarily from organic components, so that the effect of ionizing radiation is likely to be more pronounced than in enamel due to the higher proportion of organic components and water in dentin [33]. Furthermore, it can be assumed that the negative effect of ionizing radiation on collagen fibers prevents the formation of a stable hybrid layer and compromises adhesion to the dentin [15,34]. Fracture analysis also supports the assumption that the altered composition and reduced mechanical properties in irradiated dentin reduce the bond strength. Although adhesive fractures dominate (as in the control samples), mixed fractures are also present—adhesive and cohesive in the irradiated dentin. This finding is consistent with a previous study with universal adhesives conducted by Ugurlu et al. [22], who also showed that the prevalence of cohesive dentin fractures increases in irradiated samples. Universal adhesives contain bifunctional acidic monomers, such as 10-methacryloyloxydecyl dihydrogen phosphate (10-MDP) in Scotchbond Universal Plus Adhesive, and 10-MDP can form a chemical bond with calcium in hydroxyapatite crystals (HAp) on partially exposed collagen fibers due to the acidic pH of the universal adhesive [35,36]. In addition, Scotchbond Universal Plus contains a form of polyalkenoic acid—3M's proprietary Vitrebond™ copolymer. The carboxylic acid groups of Vitrebond™ can interact with the calcium in hydroxyapatite to form ionic bonds and with collagen via hydrogen bonding. Since 10-MDP also forms Ca-MDP salts, Vitrebond™ and 10-MDP compete for the calcium in hydroxyapatite, which is not the case with pure 10-MDP adhesives. The effect of ionizing radiation on the chemical bonds of Ca-10-MDP and Ca-Vitrebond™ could be estimated in future studies using X-ray photoelectron spectroscopy (XPS), and the comparison of Scotchbond Universal Plus, which contains Vitrebond, with pure 10-MDP universal adhesives could be estimated [37].

The samples irradiated after the restoration showed significantly lower bond strength for both materials tested. This result is not consistent with the results of a larger number of previous studies, which showed a significant reduction in the bond strength when irradiated before restoration [14–18]. Our results are in agreement with the results of Arid et al. [34], who also showed that the bond strength was lowest with irradiation after restoration. This result can be explained by the hydrolytic degradation of the hybrid layer (supported by the finding of adhesive fractures) and the hydrolytic degradation of the polymer matrix within the composite material (detection of cohesive fractures in the material). Although the degree of conversion of the Scotchbond Universal Plus adhesive is a high 92% according to the literature [38], the universal adhesive with nanofillers (Scotchbond contains filler particles according to the manufacturer) has a relatively higher water absorption than unfilled adhesives [39]. This can lead to degradation of the hybrid layer, especially when exposed to ionizing radiation due to the radiolysis of water. Although fillers in the adhesive composition reduce polymerization shrinkage and increase strength [36], fillers in the adhesive are not necessarily advantageous in the context of restorations in oncological patients and increased water absorption. There are generally two mechanisms for degradation of the hybrid layer: hydrolytic degradation of the polymer matrix and degradation of the collagen fibers [40]. It is a fact that the eighth-generation universal adhesives (like all single-component adhesives) must contain hydrophilic monomers such as HEMA to enable the wetting of the hydrophilic dentin substrate. In fact, HEMA is a component of the Scotchbond Universal Plus adhesive and the hydrophilic groups (such as hydroxyl, phosphate and ester) increase the water sorption and hydrolytic degradation of the hybrid layer [41]. The disintegration of the polymer matrix exposes collagen fibers, which are then exposed to proteolytic degradation and the negative effects of ionizing radiation.

In addition to the degradation of the hybrid layer, ionizing radiation could enhance the hydrolytic degradation of the polymer matrix within the bulk-fill materials. The amount of absorbed water and the hydrolytic degradation of the material are determined by the degree of conversion, the dimethacrylate composition, the proportion of filler particles and the silanization of these particles [42]. The light-curing composite materials are only 55–75% converted after polymerization, which means that more than 25% of the

monomer bonds are not involved in the formation of the polymer matrix [43]. Anseth et al. [44] estimated that with a composite conversion of 75%, 6.25% of the monomers were not incorporated into the polymer matrix. Therefore, polymerization is stopped after reaching the so-called vitrification (critical viscosity when monomer mobility is limited) [43]. Furthermore, the BisGMA in the composition of Tetric EvoFlow Bulk Fill and the TEGDMA in the composition of SDR are hydrophilic monomers, which may also contribute to water sorption and hydrolytic degradation (BisEMA and UDMA are more hydrophobic) [43]. In addition, the flowable bulk-fill composite materials used in this study have a lower proportion of filler particles than high-viscosity composites, which contributes to the water sorption [42,45]. The presence of water in the material leads to hydrolytic degradation but also the formation of hydrogen peroxide, and free hydrogen radicals (hydrogen atoms) could further weaken the restoration due to ionizing radiation. The occurrence of adhesive and cohesive cracks in both materials irradiated after restoration supports the assumption about the hydrolytic degradation of the hybrid layer in combination with the negative influence of ionizing radiation on the polymer matrix of the materials.

To a certain extent, the fracture analysis supports the SBS results in the present study. However, the results also raise further questions. Further investigations should include thermocycling for aging as well as the analysis of the dentin surface and the chemistry at the interface. Although no further analysis was performed besides fracture analysis, the results obtained are supported and justified by the findings of previous studies on the effects of ionizing radiation on dental tissues, materials and the interstitial layer [15,30,33,34]. Another limitation of this *in vitro* study is that the possible influence of the reduced saliva volume and altered oral flora composition after radiotherapy on the bond strength was not considered.

5. Conclusions

Within the limitations of this study, it can be concluded that ionizing radiation in therapeutic doses after restoration significantly reduces the bond strength of the universal adhesive and the bulk-fill flowable composites to coronal dentin. In the samples that were irradiated before the restoration, the bond strength was not significantly reduced. The results indicate the importance of continuous monitoring and evaluation of the quality of restorations in oncology patients who have undergone radiotherapy in the head and neck region, regardless of the time of placement of the restorations.

Author Contributions: Conceptualization, A.I. and E.K.S.; methodology, D.M. and M.V.; validation, D.M., M.V. and K.G.; formal analysis, D.M.; investigation, D.M. and T.G.; resources, E.K.S. and T.G.; data curation, A.I.; writing—original draft preparation, D.M.; writing—review and editing, A.I. and K.G.; visualization, D.M.; supervision, A.I.; project administration, E.K.S.; funding acquisition, E.K.S. All authors have read and agreed to the published version of the manuscript.

Funding: This research was funded by University of Zagreb research support for the academic year 2023/24, leader Professor Eva Klarić Sever.

Institutional Review Board Statement: This study was conducted in accordance with the Declaration of Helsinki and approved by the Ethics Committee of the School of Dental Medicine, University of Zagreb, 05-PA-30-155-22/2023.

Informed Consent Statement: Informed consent was obtained from all the patients involved in the study. The patients provided their written consent for surgical extraction.

Data Availability Statement: The datasets generated and analyzed during the current study are available from the corresponding authors on reasonable request.

Conflicts of Interest: Authors Dora Mohenski and Mihaela Vrebac are employed by the Private Dental Practice. The remaining authors declare that the research was conducted in the absence of any commercial or financial relationships that could be construed as potential conflicts of interest.

References

1. Sung, H.; Ferlay, J.; Siegel, R.L.; Laversanne, M.; Soerjomataram, I.; Jemal, A.; Bray, F. Global Cancer Statistics 2020: GLOBOCAN Estimates of Incidence and Mortality Worldwide for 36 Cancers in 185 Countries. *CA Cancer J. Clin.* **2021**, *71*, 209–249. [[CrossRef](#)]
2. Bray, F.; Ferlay, J.; Soerjomataram, I.; Siegel, R.L.; Torre, L.A.; Jemal, A. Global cancer statistics 2018: GLOBOCAN estimates of incidence and mortality worldwide for 36 cancers in 185 countries. *CA Cancer J. Clin.* **2018**, *68*, 394–424. [[CrossRef](#)]
3. Rocha, P.H.; Reali, R.M.; Decnop, M.; Souza, S.A.; Teixeira, L.A.; Júnior, A.L.; Sarpi, M.O.; Cintra, M.B.; Pinho, M.C.; Garcia, M.R. Adverse Radiation Therapy Effects in the Treatment of Head and Neck Tumors. *RadioGraphics* **2022**, *42*, 806–821. [[CrossRef](#)]
4. Grundmann, O.; Mitchell, G.C.; Limesand, K.H. Sensitivity of Salivary Glands to Radiation: From Animal Models to Therapies. *J. Dent. Res.* **2009**, *88*, 894–903. [[CrossRef](#)]
5. Silva, A.R.S.; Alves, F.A.; Antunes, A.; Goes, M.F.; Lopes, M.A. Patterns of Demineralization and Dentin Reactions in Radiation-Related Caries. *Caries Res.* **2009**, *43*, 43–49. [[CrossRef](#)]
6. Gonçalves, L.M.N.; Palma-Dibb, R.G.; Paula-Silva, F.W.G.; de Oliveira, H.F.; Nelson-Filho, P.; da Silva, L.A.B.; de Queiroz, A.M. Radiation therapy alters microhardness and microstructure of enamel and dentin of permanent human teeth. *J. Dent.* **2014**, *42*, 986–992. [[CrossRef](#)]
7. Velo, M.M.D.A.C.; Farha, A.L.H.; da Silva Santos, P.S.; Shiota, A.; Sansavino, S.Z.; Souza, A.T.; Honório, H.M.; Wang, L. Radiotherapy alters the composition, structural and mechanical properties of root dentin in vitro. *Clin. Oral. Investig.* **2018**, *22*, 2871–2878. [[CrossRef](#)]
8. Lu, H.; Zhao, Q.; Guo, J.; Zeng, B.; Yu, X.; Yu, D.; Zhao, W. Direct radiation-induced effects on dental hard tissue. *Radiat. Oncol.* **2019**, *14*, 5. [[CrossRef](#)]
9. Breschi, L.; Mazzoni, A.; Ruggeri, A.; Cadenaro, M.; Di Lenarda, R.; De Stefano Dorigo, E. Dental adhesion review: Aging and stability of the bonded interface. *Dent. Mater.* **2008**, *24*, 90–101. [[CrossRef](#)]
10. Bedran-Russo, A.; Leme-Kraus, A.A.; Vidal, C.M.P.; Teixeira, E.C. An Overview of Dental Adhesive Systems and the Dynamic Tooth–Adhesive Interface. *Dent. Clin. N. Am.* **2017**, *61*, 713–731. [[CrossRef](#)]
11. Pashley, D.H.; Tay, F.R.; Breschi, L.; Tjäderhane, L.; Carvalho, R.M.; Carrilho, M.; Tezvergil-Mutluay, A. State of the art etch-and-rinse adhesives. *Dent. Mater.* **2011**, *27*, 1–16. [[CrossRef](#)]
12. Niewald, M.; Mang, K.; Barbie, O.; Fleckenstein, J.; Holtmann, H.; Spitzer, W.J.; Rube, C. Dental status, dental treatment procedures and radiotherapy as risk factors for infected osteoradionecrosis (IORN) in patients with oral cancer—A comparison of two 10 years’ observation periods. *Springerplus* **2014**, *3*, 263. [[CrossRef](#)]
13. Madrid Troconis, C.C.; Santos-Silva, A.R.; Brandão, T.B.; Lopes, M.A.; de Goes, M.F. Impact of head and neck radiotherapy on the mechanical behavior of composite resins and adhesive systems: A systematic review. *Dent. Mater.* **2017**, *33*, 1229–1243. [[CrossRef](#)]
14. Naves, L.Z.; Novais, V.R.; Armstrong, S.R.; Correr-Sobrinho, L.; Soares, C.J. Effect of gamma radiation on bonding to human enamel and dentin. *Support. Care Cancer* **2012**, *20*, 2873–2878. [[CrossRef](#)]
15. Rodrigues, R.B.; Soares, C.J.; Junior, P.C.S.; Lara, V.C.; Arana-Chavez, V.E.; Novais, V.R. Influence of radiotherapy on the dentin properties and bond strength. *Clin. Oral. Investig.* **2018**, *22*, 875–883. [[CrossRef](#)]
16. Bulucu, B.; Yesilyurt, C.; Cakir, S.; Meydan, A.D. Influence of radiation on bond strength. *J. Adhes. Dent.* **2006**, *8*, 217–221.
17. Soares, E.F.; Naves, L.Z.; Correr, A.B.; Costa, A.R.; Consani, S.; Soares, C.J.; Garcia-Godoy, F.; Correr-Sobrinho, L. Effect of radiotherapy, adhesive systems and doxycycline on the bond strength of the dentin-composite interface. *Am. J. Dent.* **2016**, *29*, 352–356.
18. Yadav, S.; Yadav, H. Ionizing irradiation affects the microtensile resin dentin bond strength under simulated clinical conditions. *J. Conserv. Dent.* **2013**, *16*, 148–151. [[CrossRef](#)]
19. Biscaro, S.L.; Moraes, R.R.; Correr, A.B.; Almeida, S.M.; Boscolo, F.N.; Soares, C.J.; Correr-Sobrinho, L. Effect of X-ray radiation dose on the bond strength of different adhesive systems to dentin. *J. Adhes. Dent.* **2009**, *11*, 355–360. [[PubMed](#)]
20. Gernhardt, C.R.; Kielbassa, A.M.; Hahn, P.; Schaller, H.G. Tensile bond strengths of four different dentin adhesives on irradiated and non-irradiated human dentin in vitro. *J. Oral. Rehabil.* **2001**, *28*, 814–820. [[CrossRef](#)] [[PubMed](#)]
21. da Cunha, S.R.d.B.; Ramos, P.A.M.M.; Haddad, C.M.K.; da Silva, J.L.F.; Fregnani, E.R.; Aranha, A.C.C. Effects of Different Radiation Doses on the Bond Strengths of Two Different Adhesive Systems to Enamel and Dentin. *J. Adhes. Dent.* **2016**, *18*, 151–156. [[CrossRef](#)]
22. Ugurlu, M. Effect of the double application of universal adhesives on the dentine bond strength after radiotherapy. *Aust. Dent. J.* **2020**, *65*, 181–188. [[CrossRef](#)]
23. Oglacki, B.; Burduroğlu, D.; Eriş, A.H.; Mayadağlı, A.; Arhun, N. How Does Radiotherapy Affect the Adhesion of Universal Adhesive to Enamel and Dentin? A Qualitative and Quantitative Analysis? *Odovot Int. J. Dent. Sci.* **2022**, *24*, 75–90.
24. Jacker-Guhr, S.; Sander, J.; Luehrs, A.K. How “Universal” is Adhesion? Shear Bond Strength of Multi-mode Adhesives to Enamel and Dentin. *J. Adhes. Dent.* **2019**, *21*, 87–95. [[CrossRef](#)]
25. David, C.; Cuevas-Suárez, C.E.; de Cardoso, G.C.; Isolan, C.P.; de Moraes, R.R.; da Rosa, W.L.O.; Münchow, E.A.; da Silva, A.F. Characterization of Contemporary Conventional, Bulk-fill, and Self-adhesive Resin Composite Materials. *Oper. Dent.* **2022**, *47*, 392–402. [[CrossRef](#)]
26. de Freitas Chaves, L.V.; de Sousa Lima, R.X.; de Azevedo Silva, L.J.; Bruschi Alonso, R.C.; Geraldini, S.; Dutra Borges, B.C. Bonding performance and mechanical properties of flowable bulk-fill and traditional composites in high c-factor cavity models. *J. Conserv. Dent.* **2020**, *23*, 36–41. [[CrossRef](#)]

27. Anderson, G.; Ebadi, M.; Vo, K.; Novak, J.; Govindarajan, A.; Amini, A. An Updated Review on Head and Neck Cancer Treatment with Radiation Therapy. *Cancers* **2021**, *13*, 4912. [[CrossRef](#)] [[PubMed](#)] [[PubMed Central](#)]
28. ISO 29022; Dentistry—Adhesion—Notched Edge Shear Bond Strength Test. International Organization for Standardization: Geneva, Switzerland, 2013.
29. Alfouzan, A.F. Radiation therapy in head and neck cancer. *Saudi Med. J.* **2021**, *42*, 247–254. [[CrossRef](#)]
30. Munoz, M.A.; Garin-Correa, C.; Gonzalez-Arriagada, W.; Quintela Davila, X.; Häberle, P.; Bedran-Russo, A.; Luque-Martinez, I. The adverse effects of radiotherapy on the structure of dental hard tissues and longevity of dental restoration. *Int. J. Radiat. Biol.* **2020**, *96*, 910–918. [[CrossRef](#)] [[PubMed](#)]
31. Le Caër, S. Water Radiolysis: Influence of Oxide Surfaces on H₂ Production under Ionizing Radiation. *Water* **2011**, *3*, 235–253. [[CrossRef](#)]
32. Ray-Chaudhuri, A.; Shah, K.; Porter, R.J. The oral management of patients who have received radiotherapy to the head and neck region. *Br. Dent. J.* **2013**, *214*, 387–393. [[CrossRef](#)] [[PubMed](#)]
33. Pioch, T.; Golfels, D.; Staehle, H.J. An experimental study of the stability of irradiated teeth in the region of the dentinoenamel junction. *Endod. Dent. Traumatol.* **1992**, *8*, 241–244. [[CrossRef](#)] [[PubMed](#)]
34. Arid, J.; Palma-Dibb, R.G.; de Oliveira, H.F.; Nelson-Filho, P.; de Carvalho, F.K.; da Silva, L.A.B.; de Siqueira Mellara, T.; da Silva, R.A.B.; Faraoni, J.J.; de Queiroz, A.M. Radiotherapy impairs adhesive bonding in permanent teeth. *Support. Care Cancer* **2020**, *28*, 239–247. [[CrossRef](#)] [[PubMed](#)]
35. Van Meerbeek, B.; Yoshihara, K.; Yoshida, Y.; Mine, A.; De Munck, J.; Van Landuyt, K.L. State of the art of self-etch adhesives. *Dent. Mater.* **2011**, *27*, 17–28. [[CrossRef](#)] [[PubMed](#)]
36. Cadenaro, M.; Maravic, T.; Comba, A.; Mazzoni, A.; Fanfoni, L.; Hilton, T.; Ferracane, J.; Breschi, L. The role of polymerization in adhesive dentistry. *Dent. Mater.* **2019**, *35*, e1–e22. [[CrossRef](#)] [[PubMed](#)]
37. Yoshida, Y.; Inoue, S. Chemical analyses in dental adhesive technology. *Jap. Dent. Sci. Rev.* **2012**, *48*, 141–152. [[CrossRef](#)]
38. Scotchbond Universal Plus Adhesive Technical Product Profile. Available online: <https://multimedia.3m.com/mws/media/19106080/3m-scotchbond-universal-plus-adhesive-technical-product-profile-us.pdf> (accessed on 18 February 2024).
39. Araújo-Neto, V.G.; Moreira, M.M.; Ñaupari-Villasante, R.; De Paula, D.M.; Medeiros, S.T.C.B.; Loguercio, A.D.; Feitosa, V.P. Nanofiller Particles and Bonding Durability, Water Sorption, and Solubility of Universal Adhesives. *Oper. Dent.* **2021**, *46*, 690–697. [[CrossRef](#)]
40. Hashimoto, M.; Ohno, H.; Sano, H.; Kaga, M.; Oguchi, H. In vitro degradation of resin–dentin bonds analyzed by microtensile bond test, scanning and transmission electron microscopy. *Biomaterials* **2003**, *24*, 3795–3803. [[CrossRef](#)] [[PubMed](#)]
41. Ferracane, J.L. Hygroscopic and hydrolytic effects in dental polymer networks. *Dent. Mater.* **2006**, *22*, 211–222. [[CrossRef](#)]
42. Alshali, R.Z.; Salim, N.A.; Satterthwaite, J.D.; Silikas, N. Long-term sorption and solubility of bulk-fill and conventional resin-composites in water and artificial saliva. *J. Dent.* **2015**, *43*, 1511–1518. [[CrossRef](#)]
43. Schricker, S.R. Composite resin polymerization and relevant parameters. In *Orthodontic Applications of Biomaterials*; Eliades, T., Brantley, W.A., Eds.; Woodhead Publishing: Sawston, UK, 2017; pp. 153–170. [[CrossRef](#)]
44. Anseth, K.S.; Goodner, M.D.; Reil, M.A.; Kannurpatti, A.R.; Newman, S.M.; Bowman, C.N. The Influence of Comonomer Composition on Dimethacrylate Resin Properties for Dental Composites. *J. Dent. Res.* **1996**, *75*, 1607–1612. [[CrossRef](#)]
45. Mortier, E.; Gerdolle, D.A.; Jacquot, B.; Panighi, M.M. Importance of water sorption and solubility studies for couple bonding agent—Resin-based filling material. *Oper. Dent.* **2004**, *29*, 669–676.

Disclaimer/Publisher’s Note: The statements, opinions and data contained in all publications are solely those of the individual author(s) and contributor(s) and not of MDPI and/or the editor(s). MDPI and/or the editor(s) disclaim responsibility for any injury to people or property resulting from any ideas, methods, instructions or products referred to in the content.