

Article

The Influence of Ionizing Radiation on the Morphological Structure of the Fluoride-Releasing Restorative Materials in Cancer Patients: An In Vitro Study

Eva Klari´c 1,*, Bruno Špiljak ² [,](https://orcid.org/0000-0003-2545-4474) Luka Šimunovi´c ³ [,](https://orcid.org/0000-0003-2848-6041) Majana Soˇce ⁴ , Timor Grego ⁴ and Ana Ivaniševi´c [1](https://orcid.org/0000-0002-1380-0029)

- ¹ Department of Endodontics and Restorative Dental Medicine, School of Dental Medicine, 10000 Zagreb, Croatia
- ² Department of Oral Medicine, School of Dental Medicine, 10000 Zagreb, Croatia
- ³ Department of Orthodontics, School of Dental Medicine, 10000 Zagreb, Croatia; lsimunovic@sfzg.hr
⁴ Department of Orgal aan Radiathaneau Unit University Usanital Centre Zagreb, 10000 Zagreb, Cro
- ⁴ Department of Oncology, Radiotherapy Unit, University Hospital Centre Zagreb, 10000 Zagreb, Croatia
	- ***** Correspondence: eklaric@sfzg.hr

Abstract: Radiotherapy plays a key role in the treatment of the early and advanced stages of head and neck cancer. To date, there is still no consensus on the effects of radiotherapy on the mechanical properties of fluoride-releasing restorative materials which can be used in patients undergoing radiotherapy with increased incidence of caries. The fluoride-releasing materials Equia Forte HT and Cention N were compared to the resin-based materials Tetric EvoCeram and Tetric Power Fill. Standard irradiation was performed with a linear accelerator. Vickers microhardness, mass, surface roughness and color were measured before and after irradiation. Cention N and Tetric PowerFill showed stability in the mass, while the surface roughness did not change in any of the examined groups. Resistance to microhardness change was shown by Cention N, Tetric PowerFill and Tetric EvoCeram, and the color change was significant in all groups ($p < 0.05$). It should be remembered that patients receiving head and neck radiation therapy may experience adverse effects from the treatment, including changes in the mechanical properties of the restorative materials. The obtained results suggest that Cention N can be used as a material in patients undergoing head and neck radiotherapy due to the mechanical stability and depo effect of fluoride release.

Keywords: radiotherapy; dental materials; microhardness; surface roughness; mass; color

1. Introduction

Radiotherapy plays a key role in the treatment of early and advanced stages of head and neck cancer in a dose range of 54 to 70 Gray (Gy), administered with a standard fractionation schedule of 2 Gy per 5-fraction per week [\[1\]](#page-9-0). Gray serves as the unit of ionizing radiation dose in the International System of Units. Although technological advances have led to the spread of radiotherapy indications in everyday clinical practice, the application of radiotherapy in the head and neck area is still marked by a series of acute and late side effects affecting cells of numerous anatomical structures in the oral cavity, head and neck [\[2](#page-9-1)[–5\]](#page-9-2). In addition, radiation alone can also affect restorative materials and cause clinically significant changes depending on the properties of the material [\[6,](#page-9-3)[7\]](#page-9-4). Composite materials and glass ionomer cements have largely found their use as restorative materials due to their corresponding clinical efficacy [\[8\]](#page-9-5). However, a number of factors, including ionizing radiation, can affect material properties and contribute to early structural deformations [\[9,](#page-9-6)[10\]](#page-10-0). Doctors often recommend dental treatment to patients immediately before head and neck radiotherapy [\[11](#page-10-1)[–13\]](#page-10-2). Such treatment usually requires replacement of amalgam fillings with composite materials and glass ionomer cements, since in the presence of amalgam, ionizing radiation interacts with the amalgam filling, which in turn enhances radiation and can lead to localized mucositis $[10,12,14]$ $[10,12,14]$ $[10,12,14]$. This secondary radiation

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mainly depends on the atomic number of the material components [\[14\]](#page-10-4), which is why this effect should be reduced by the use of composites and glass ionomer cements, since they have the ability to absorb radiation.

Currently, the choice of restorative materials in patients undergoing head and neck radiotherapy is primarily based on personal clinical preferences rather than scientific evidence [\[15](#page-10-5)[,16\]](#page-10-6). Due to the fact that restorative fillings are also within the area of the primary field of the irradiated tumor, the question of their sensitivity to the direct effects of radiotherapy arises. Some in vitro studies show a negative interaction between ionizing radiation and amalgam fillings, increasing the original radiation dose due to high density, atomic number and conductivity [\[17](#page-10-7)[,18\]](#page-10-8). Furthermore, some authors state that the mechanical properties and clinical life of restorative dental materials, such as conventional glass ionomer cements and resin-modified glass ionomer cements, are severely affected by the indirect hyposalivation effect closely associated with radiation damage to the salivary glands [\[19,](#page-10-9)[20\]](#page-10-10).

In view of the above, composite materials seem to be the material of choice: in addition to excellent optical properties, they also have an enamel and dentin-like elastic modulus that allows more even distribution of masticatory load and show greater biocompatibility compared to amalgam fillings [\[21\]](#page-10-11). These issues are relevant during restorative treatment of patients with head and neck cancer who have undergone radiotherapy. One of the foundations of today's restorative dentistry is the desire to preserve healthy hard dental tissues to the greatest extent possible with adequate bond strength, avoiding the replacement of restorative fillings after the start of radiotherapy. However, as previously shown in a number of studies, radiotherapy of tumors in the maxillofacial region has a direct and indirect impact on restorative fillings in the irradiated area [\[18,](#page-10-8)[19,](#page-10-9)[22\]](#page-10-12).

Unfortunately, there is still no consensus on the effects of radiotherapy on the mechanical properties of fluoride-releasing materials. Also, there are still discussions about which is the most suitable restorative material for use in patients treated with head and neck radiotherapy. The aim of this study was to demonstrate the potential effects of ionizing radiation on four different modern restorative materials. The hypothesis of the study was that different types of ionizing radiation have no effect on changing the morphological properties and color of restorative materials.

2. Materials and Methods

2.1. Sample Preparation

In this study, two materials with fluoride release, Equia Forte HT, referred to in the later text as EQ (GC, Tokyo, Japan), and Cention N, referred to in later text as CEN (Ivoclar Vivadent, Schaan, Liechtenstein), and two composite materials, Tetric EvoCeram, referred to in later text as TC and Tetric PowerFill, referred to in later text as PF (Ivoclar Vivadent, Schaan, Liechtenstein), were used (Table [1\)](#page-2-0). Equia Forte HT is a strong biocompatible long-term bulk-fill restorative glass hybrid system with enhanced mechanical properties, superior fluoride release, excellent handling and improved translucency. Cention N is an alkasite bioactive ion-release material with especially long-term fluoride release, which is claimed to be significantly higher even after 180 days when compared to glass ionomer [\[23\]](#page-10-13). On the other hand, Tetric EvoCeram is a sculptable universal composite for the incremental layering technique, while Tetric PowerFill is a sculptable 4 mm composite for esthetic results in the posterior region, and both are inert resin composite materials without any fluoride-release activity.

It was necessary to make 30 samples of each material (total $n = 120$), to have 10 samples $(n = 10)$ in each category. Category 1 was samples irradiated for 35 days at 2 Gy/day; Category 2 was samples irradiated with an impact dose of 70 Gy; and Category 3 was the control group without irradiation (Figure [1\)](#page-2-1). The materials were prepared in Teflon molds ($4 \times 4 \times 3$ mm), under which the glass was placed; then, the materials were mixed in an automatic mixer (Septodont, France) for 10 s, placed in the mold and pressed again with glass to expel the excess material (Figure [2a](#page-3-0)). The samples were then polymerized for

20 s from both sides using an LED curing lamp (Ivoclar Vivadent, Liechtenstein) with a power of 1100 mW/cm². Specimens were fine furbishings with disks (Water Proof Silicon Carbide Paper, 4000 grit; Buehler, Dusseldorf, Germany) and 1.0 µm, 0.3 and 0.05 µm professional polishing silicon granules (Buehler, Dusseldorf, Germany). The polishing procedure was carried out using a polishing appliance (Minitech 250, Presi, France). After that, samples were washed in distilled water. The samples were randomly divided into groups of 10 samples ($n = 10$).

Table 1. The type and composition of the investigated materials according to the manufacturer's instructions.

Figure 1. Diagram of materials, groups and type of measurement used in this study. Materials used: **Figure 1.** Diagram of materials, groups and type of measurement used in this study. Materials used: Equia Forte HT (GC, Japan), Cention N (Ivoclar Vivadent, Schaan, Liechtenstein), Tetric EvoCeram and Tetric PowerFill (Ivoclar Vivadent, Schaan, Liechtenstein).

Figure 2. (a) Teflon molds $(4 \times 4 \times 3$ mm) (b) Linear accelerator Siemens Primus (Siemens Healthineers AG, Erlangen, Germany). neers AG, Erlangen, Germany).

2.2. Irradiation Procedure

To simulate oral cancer radiotherapy, the materials in Category 1 were exposed to 2 Gray (Gy) fractions, 5 days a week for 7 weeks for a total of 35 fractions equal to 70 Gy (frequent oral cancer dose). One gray (Gy) is the international system of units (SI) equivalent of 100 rads, which is equal to an absorbed dose of 1 Joule/kilogram. An absorbed dose of 0.01 Gy means that 1 g of material absorbed 100 ergs of energy (a small but measurable amount) as a result of exposure to radiation. Over the weekend, the samples were not irradiated. The materials in Category 2 were exposed to one experimental 70 Gy irradiation dose. Radiation was performed at the Department of Oncology, University Hospital Centre Zagreb, with a linear accelerator Siemens Primus (Siemens Healtheneers AG, Erlangen, Germany) radiotherapy unit (Figure [2b](#page-3-0)). A 6 MV radiation beam was used with a SSD (source to surface distance) of 100 cm setup for sample irradiation. Two centimeters of buildup material was placed above and below samples to ensure sufficient buildup and scatter conditions. Between the radiation cycles, samples were stored in deionized water, which was renewed daily, in an incubator at 37° C (INEL, Zagreb, Croatia). Also, the impact of a shock dose of ionizing radiation with one single dose of 70 Gy on the materials was also investigated.

2.3. Surface Microhardness Analysis

The microhardness of the enamel was measured at three different time points: after polishing, the initial measurement and after irradiation. The Vickers microhardness (HV) of the material surface was determined with a microhardness tester (CSV-10; ESI Prüftechnik GmbH, Wendlingen, Germany) using a load of 100 g and a dwell time of 10 s (Figure [3a](#page-4-0)). A diamond pyramid applied to the sample surface was used for measurement. Measurements were performed at three different points on the enamel: the surface, middle, and deep part (at 50 µm intervals) (Figure [3b](#page-4-0)). The average of all three Vickers hardness values obtained from the enamel was recorded as the total value of enamel hardness [\[24\]](#page-10-14).

Figure 3. (a) Microhardness tester (CSV-10; ESI Prüftechnik GmbH, Wendlingen, Germany); (b) diaamond pyramid that remained imprinted on the surface of the sample. mond pyramid that remained imprinted on the surface of the sample.

obtained from the enamel was recorded as the total value of enamel hardness [24].

2.4. Prophylometry 2.4. Prophylometry

The measurement of the surface roughness of the sample was performed with a high-high-precision profilometer (Mitutoyo, Japan) (Figure [4a](#page-4-1)). The following measurement parameters were used: stylus speed: 0.1 mm/s, stylus force: 4 mN, cut-off length: 0.25 mm, sampling length: 0.8 mm, and number of sampling lengths: 5. Evaluations were carried out at three different sites of each specimen within a radius of 3 mm from the specimen center and the mean value was calculated (Figure 4b). These tests determined the roughness parameter Ra after polishing, before irradiation and after ionizing radiation of all groups of materials in order to determine the effect of radiation on the exposed material's surface. A ness", which is often communicated as "Ra". Ra is the calculated average between peaks and valleys on a surface. The lower the Ra value, the less variation between the peaks The measurement of the surface roughness of the sample was performed with a common unit of measurement of surface roughness is by measuring the "average roughand troughs on a surface, making the surface smoother. In addition to surface roughness, they can also present other deviations such as waveforms and surface defects in the form of pores.

Figure 4. (a) Portable device for measuring surface roughness (Surftest SJ-210; Mitutoyo, Houston, TX, USA); (**b**) presentation of surface roughness measurements of the tested sample. TX, USA); (**b**) presentation of surface roughness measurements of the tested sample.

2.5. Measurement of Color Change 2.5. Measurement of Color Change

The color of restorative materials was measured by a VITA Easyshade (Vita Zahnfabrik, Bad Säckingen, Germany) spectrometer (Fi[gu](#page-5-0)re 5a). The materials were measured in such a way that the peak of the spectrometer, which was coated with a protective cap to defend the optical elements of the instrument against damage, was directed to the center of the sample surface. Measurements were repeated three times and the instrument automatically

averaged the measurements for each sample, which was then used for the overall data analysis. All measurements were performed by the same researcher, under the same conditions. The color difference before and after radiation (ΔE) was calculated (Figure [5b](#page-5-0)).

Figure 5. (**a**) Measurement of color change with a spectrophotometer with a probe (VITA Easyshade **Figure 5.** (**a**) Measurement of color change with a spectrophotometer with a probe (VITA Easyshade III; Vita, Bad Säckingen, Germany); (**b**) reading L^{*}, a^{*}, and b^{*} values from the device.

2.6. Mass Measurement

2.6. Mass Measurement Toledo scale (Columbus, OH, USA) (Figure $\overline{6}$). Samples were weighed before and after ionizing radiation. Each sample was measured three times, from which the arithmetic in each was calculated. Each sample was measured three times, from which the arithmetic structure \mathbb{R}^n The measurement of the mass of the sample was performed using a precision Mettler

Figure 6. Precision Mettler Toledo scale (Columbus, OH, USA). **Figure 6.** Precision Mettler Toledo scale (Columbus, OH, USA).

2.7. Statistical Analysis

2.7. Statistical Analysis The results of the research are presented descriptively in the form of arithmetic means, standard deviations and 95% confidence intervals. Initial measurements and measurements after different types of irradiations were compared separately for each parameter. Changes caused by irradiation were calculated by the Wilcoxon rank test, since the measured

parameters did not follow the normal distribution, which was confirmed by the Shapiro– Wilk normality test and Kolmogorov test for each parameter. Also, indicators of asymmetry and roundness indicated an abnormal distribution. Changes in color parameters showed normal distribution and the selection test was the *t*-test for dependent samples. The results are considered statistically significant at a significance level of 0.05. The analysis was performed using the Software package statistics (TIBICO Statistica Version 13.5.0.17).

3. Results

CEN showed the greatest resistance to mass change during a shock dose of ionizing radiation. With long-term exposure to gamma rays for 35 days, CEN and PF did not show statistically significant changes in mass (Table [2\)](#page-6-0). By comparing the measurement of the Ra parameter before and after exposure of the material to radiation, no statistically significant change was found in any group. A statistically significant decrease in microhardness after irradiation for 35 days with 2 Gy/day was recorded only in the EQ material $p = 0.006911$. The change in Vickers microhardness (HV.01) after impact radiation with 70 Gy was recorded only with $PF p = 0.006911$ (Table [3\)](#page-6-1). By including the measured parameters in the formula ΔE * = $[(ΔL[*])² + (Δa[*])² + (Δb[*])²]$ ¹/₂, experimental results were obtained (CEN 7.25, EQ 5.99, PF 5.28 and EC 5.30), as well as impact samples (CEN 9.88, EQ 7.97, PF 8.18 and EC 10.30) in the interval 2–10, which means that the color change is "perceptible at a glance". The exception is the shock EC $E^* = 10.30$, so it does not belong to the same interval. It is defined as "Colors more similar than opposite". Comparing parameters L, a and b individually, we obtained the following: For parameter L (luminance), in the group that was irradiated for 35 days with 2 Gy/day (experimental group), there was no statistically significant change in any material. While in the group that received a shock dose of radiation of 70 Gy, there was a statistically significant change in all materials except PF. For parameter a (red-green axis), in the experimental group, a statistically significant difference was found with EQ and PF, while in the impact group it was present with all materials. And for Parameter b (blue-yellow axis), in the experimental group, a statistically significant difference was found with EQ and PF, while in the impact group it was present with all materials (Tables [4](#page-7-0) and [5\)](#page-7-1).

Table 2. Mass change after 35 days of ionizing radiation with 2 Gy/day and shock dose of ionizing radiation with 70 Gy.

Table 3. Surface microhardness (HV) before and after (a) 35 days of ionizing radiation with 2 Gy/day and (b) shock dose of ionizing radiation with 70 Gy.

Table 4. Changes in parameter L after 35 days of ionizing radiation with 2 Gy/day.

Table 5. Changes in parameter L after shock dose of ionizing radiation with 70 Gy.

	T-Test for Dependant Samples Marked Differences Are Significant at $p < 0.0500$									
Variable	Mean	Std.Dv.	N	Diff.	Std.Dv.	t	df	\boldsymbol{p}	Confidence	Confidence
L before CEN	68.640	3.712								
L after CEN	77.730	2.807	10	-9.090	5.088	-5.649	9	0.003	-12.729	-5.450
L before EO	57.810	4.383								
L after EO	63.140	2.270	10	-5.330	5.053	-3.336	9	0.008	-8.944	-1.715
L before PF	69.690	2.679								
L after PF	70.580	5.211	10	-0.890	5.332	-0.527	9	0.610	-4.704	2.942
L before EC	65.130	2.897								
L after EC	74.140	3.678	10	-9.100	4.788	-5.950	9	0.000	-12.435	-5.584

4. Discussion

In this research, the effects of radiation on several properties of dental restorative materials were examined using two radiation modes: 35 days at 2 Gy per day and a 70 Gy shock dose in a single day. For clinical practice, long-term radiation data (first mode) are particularly relevant, as they mimic the conditions experienced by patients undergoing head and neck radiotherapy. The shock dose, while not typical in clinical scenarios, was included to evaluate the impact of intense, short-duration radiation on the materials. Tables [1](#page-2-0) and [2](#page-6-0) show mass changes during both radiation modes. The CEN samples exhibited the highest stability or resistance to mass change under both 1-day and 35-day irradiation. The PF samples showed stability only with long-term radiation. This stability supports the use of these materials in areas subjected to higher occlusion loads, reducing the risk of microleakage and edge cracks. Both materials contain a germanium-based photoinitiator (Ivocerin) that enhances polymerization depth, contributing to their stability [\[25–](#page-10-15)[32\]](#page-10-16). Tables [3](#page-6-1) and [4](#page-7-0) indicate no statistically significant changes in surface roughness (Ra parameter) for any test groups post-irradiation. Surface roughness is a critical factor influencing the clinical life of restorative fillings, as increased roughness can lead to rapid colonization by microorganisms, plaque accumulation, secondary caries, gingivitis, and loss of periodontal attachment [\[28,](#page-10-17)[29\]](#page-10-18). Proper polishing of restorations is crucial for durability and esthetics [\[33](#page-10-19)[–36\]](#page-10-20), and this study suggests that radiation did not adversely affect surface roughness. The EQ material initially showed the highest strength but exhibited a significant decrease in microhardness after prolonged radiation. The PF showed a significant decrease after the shock dose. Patients undergoing head and neck radiotherapy

are particularly exposed to the mentioned changes due to damage to the salivary glands, which reduces saliva secretion [\[1\]](#page-9-0). This results in sticky mucinous saliva with a diminished capacity to wash away harmful microorganisms and their metabolites. Previous research by Billingham [\[37\]](#page-10-21) suggests that ion radiation can alter hardness and elasticity by forming free radicals, which may affect cross-linking in the materials. The TC material showed an increase in microhardness after shock dose irradiation, potentially due to the better chain connection post-radiation [\[14,](#page-10-4)[38](#page-10-22)[,39\]](#page-11-0). A recent analysis by the Radiation Therapy Oncology Group, a nonprofit organization dedicated to improving outcomes for cancer patients through the conduct of practice changing clinical trials, suggests that patients treated with more than 63 Gy in 1.8 to 2 Gy fractions had significantly better outcomes. A study by Kong at al. suggests that a higher dose (>70 Gy) through a personalized prescription can further improve local regional tumor control and overall survival, especially in patients treated concurrently with chemotherapy [\[40\]](#page-11-1). However, no correlation between surface roughness and microhardness was found, which is consistent with the findings of Bala et al. [\[41\]](#page-11-2). Color changes, measured by ∆E, ranged from 2 to 10, indicating visible changes. While brightness (L parameter) remained stable after 35 days of radiation, chromaticity (a and b parameters) showed significant changes in EQ and PF materials. The impact doses caused more pronounced color changes, decreasing brightness in all materials except PF and increasing chromaticity. Meena et al. [\[42\]](#page-11-3) found that nanohybrid composites are less prone to color changes in different liquids, which aligns with our findings for EQ [\[43](#page-11-4)[,44\]](#page-11-5). This research confirmed that the ∆E value for EC (nanohybrid composite) exceeds 10, indicating that the color change is classified as "Colors more similar than the opposite". Given that the purpose of this paper was to become acquainted with the changes in the mentioned properties of restorative materials after long-term irradiation with a dose of 2 Gy, in order to obtain knowledge related to the material of choice for head and neck radiotherapy patients, we believe that it is necessary to summarize the results obtained after 35 days of radiation in next few sentences:

- (1) After 35 days of radiation at 2 Gy per day, the CEN and PF materials demonstrated mass stability.
- (2) The surface roughness remained unchanged across all groups, while CEN, PF and EQ showed resistance to microhardness changes.
- (3) Significant color changes were observed in all groups, but brightness remained stable.
- (4) The CEN material showed the best overall stability and had an anti-caries effect, releasing ions under low pH conditions, which is beneficial for patients with xerostomia due to radiotherapy [\[25\]](#page-10-15).

This is particularly important for patients who experience irreversible damage to the salivary glands, resulting in xerostomia. Serous glands are more radiosensitive than mucous glands, making the radiation volume of the parotid gland a key factor in xerostomia development. Radiation doses of 60 Gy can reduce saliva secretion by 80%, leading to qualitative and quantitative changes in saliva. These changes include decreased pH due to lower bicarbonate ion concentration and altered bacterial microflora with increased acidogenic bacteria. These conditions contribute to chronic side effects of head and neck radiotherapy, such as radiation caries [\[44](#page-11-5)[–46\]](#page-11-6). Contrary findings in the literature, such as those of Lima et al. [\[47\]](#page-11-7), reported increased roughness in glass ionomer materials post-radiation, though these studies used different materials. However, our previous research supports the findings of this study, indicating no significant impact of radiation on the microhardness of glass hybrid, resin composite, and alkasite materials [\[48\]](#page-11-8). Atalay et al. [\[48\]](#page-11-8) found varied effects on roughness depending on the material, with the giomer Beautifl II and Cention roughness values being negatively affected by radiotherapy, while the roughness of the rest of the Equia and Activa Bioactive Restorative was not altered. However, this research has certain limitations. Firstly, in vitro conditions do not fully replicate the oral environment. Secondly, samples were stored in distilled water and were not exposed to saliva's buffering effects, temperature changes or cyclic mechanical loads. Therefore, future research should aim to mimic in vivo conditions more closely and

consider extended preservation in artificial saliva. Furthermore, variations in material composition necessitate further studies to generalize findings and resolve the conflicting literature on radiation's effects on restorative materials. Future studies should also assess other mechanical properties in clinical contexts.

5. Conclusions

In the context of this in vitro research and despite the limitations mentioned, it is possible to draw the following conclusions: ionizing radiation has a significant effect on the change in material mass of Equia Forte HT and Tetric Evo Ceram; ionizing radiation has no significant effect on the change in surface roughness of all materials tested; ionizing radiation has a significant effect on the change in microhardness of the Equia Forte HT material; and ionizing radiation has a significant effect on the change in color of all materials tested. Cention N should be the material of choice for patients undergoing radiotherapy in the head and neck region, as the morphological properties of the material itself are the most stable and it is known as a modern ion-release bioactive material.

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