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Original research

Effect of ionizing radiation on the microstructure and physical properties of endodontic gutta-percha points

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Purpose
Patients undergoing radiotherapy for head or neck cancer often require root canal treatments, which can be compromised by the effects of radiation. This investigation aimed to determine whether ionizing radiation (IR), in doses similar to those used in conventional therapy, affects the surface and physicomechanical properties of various brands of endodontic gutta-percha points (EGPs).

Materials and Methods
One hundred and twenty-three EGPs from three brands (Meta-Biomed, Dentsply, and Hygenic) were divided into groups and either exposed or not exposed to IR at a total dose of 50 Gy, divided into 25 fractions. Tensile strength and microhardness tests were performed on all EGPs. Scanning electron microscopy was utilized to identify possible microstructural surface changes due to IR exposure. The proportion of organic to inorganic components in each brand was also determined.

Results
Exposure to IR resulted in significant changes only in the EGPs from the Meta-Biomed brand, including a notable decrease in tensile strength and an increase in microhardness. Furthermore, the surface microstructure of these EGPs displayed dark lines and striations over a large area, with some lines deeply embedded in the center and cavities of variable depths and extensions observed, leading to irregular and non-smooth surfaces. This brand had the highest proportion of organic components.

Conclusion
The physicomechanical properties and surface microstructure of Meta-Biomed brand EGPs were significantly affected by IR at doses used in conventional therapy for head or neck cancer, while the other brands were less affected or unaffected.

Keywords: *Radiotherapy, endodontic gutta-percha points, microstructure, head and neck cancer, root canal therapy*

Introduction

Head and neck cancers are serious and debilitating illnesses typically treated with a combination therapy approach, including surgery, chemotherapy, and radiotherapy using ionizing radiation (IR) (1). Radiotherapy has been proven effective in controlling and curing malignant tumors (2). However, it is well known that radiotherapy can cause oral complications as side effects on healthy soft and hard tissues, significantly affecting quality of life (3). These complications can be temporary or permanent, requiring ongoing dental care due to their long-lasting effects, sometimes persisting for months or years. The most common complications include mucositis, xerostomia, dysesthesia, bacterial and fungal infections, trismus, osteoradionecrosis, periodontitis, and radiation-induced dental caries (4–7).

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A thorough oral examination should be performed before initiating radiotherapy, and necessary treatments should be administered, with efforts made to avoid tooth extraction whenever possible to reduce the risk of osteoradionecrosis during or after IR exposure. Consequently, root canal treatment is frequently performed on many patients (1). Despite the high success rate of current root canal treatments, the prognosis may be compromised in immunosuppressed patients with xerostomia and alterations in oral microbiota; thus, these factors must be considered during treatment (8,9). Furthermore, it is crucial to consider the materials used in root canal treatments, such as those for filling root canals and for provisional or definitive tooth restoration, as these materials will also be exposed to IR. Few studies have explored the impact of IR on the adhesive properties of restorative materials (10) and different types of endodontic sealers (11,12). However, the effects of IR on endodontic gutta-percha points (EGPs), which are the core of root canal fillings and considered the "gold standard" among endodontic filling materials, have not been thoroughly investigated (13). EGPs are available in various brands, primarily composed of zinc oxide, gutta-percha polymer, waxes, resins, and barium sulfate (14), with compositions varying by brand (15). These differences in composition can lead to variations in physicomechanical properties, such as brittleness, stiffness, tensile strength, and radiopacity, largely depending on the ratios of organic (gutta-percha polymer and waxes/resins) to inorganic (zinc oxide and metal sulfates) components (16).

Given their composition, it is logical to hypothesize that IR could affect the properties of EGPs similarly to its effects o n enamel and dentin tissues (17,18). This study aims to determine whether IR, in doses akin to those used in conventional therapy for head or neck cancer, impacts the surface microstructure and physicomechanical properties of different brands of EGPs. There would be no significant difference in the surface microstructure and physicomechanical properties of different brands of EGPs after exposure to IR in doses similar to those used in conventional therapy for head or neck cancer.

Materials and methods

Gutta-percha points and study groups

One hundred and twenty-three #45, 0.2 taper EGPs from three brands, as listed in Table 1, were utilized. These were divided into four groups. Group 1 included 60 EGPs, with 20 from each brand; half of these were subjected to IR and all were tested for tensile strength. Group 2 comprised 45 EGPs, 15 from each brand, to assess surface microhardness before and after IR exposure. Group 3 consisted of 18 EGPs, six from each brand, with half receiving IR; all were examined using a scanning electron microscope (SEM). Group 4 involved using 1g of EGPs from each brand to determine the organic/inorganic content ratio. Given that the physicomechanical tests conducted on groups 1 and 2 are unconventional for these materials, and no standards exist regarding the methodology or the required sample size, the approach was first standardized through pilot tests. These preliminary tests identified optimal experimental conditions, and the sample sizes for each group were determined based on methodologies from other studies that examined similar physicomechanical properties in different dental materials.

Table 1: *Endodontic gutta-percha points selected for this study*

Brand	Manufacturer	Lot number
Meta-Biomed	Meta Biomed Co, Ltd, Chungbuk, Korea	GE19030068
Dentsply	Dentsply Maillefer, Ballaigues, Switzerland	031217
Hygienic	Hygienic, Coltene/ Whaledent, Inc., USA	K17196

A crucial step in the pretest procedure involved converting the conical shape of each EGP into a flat form in a standardized manner. This was accomplished by pressing each EGP at 400 Newtons between two metallic plates using a computer-controlled universal testing machine (UTM) (CMS Metrology, Model WDW-5Y, Querétaro, Mexico), thereby facilitating all subsequent tests.

Ionizing radiation exposure

All EGPs were wrapped in gauze and submerged in sterile distilled water. Each group was then placed inside plastic bags and categorized based on whether they were designated to receive IR exposure. The bags not intended for IR exposure were stored at room temperature, shielded from light, heat, and any potential sources of IR. Conversely, the bags designated for IR exposure were processed by the Oncologic Center of Querétaro S.A. de C.V. This was done using a medical linear accelerator machine (Trilogy Linear Accelerator; Varian Medical Systems), which delivered IR using 6 MV X-rays from a distance of 100 cm. A total dose of 50 Gy, divided into 25 fractions (2 Gy per fraction), was administered over five consecutive days per week for six weeks, mirroring the standard radiotherapy protocol for head and neck cancer.

Tensile strength test

The tensile strength of each EGP in Group 1 was measured using the same UTM previously mentioned, but this time equipped with rubber grips. Paper tape was applied to both ends of each EGP, leaving a 10 mm section exposed. The rubber grips secured the ends of the EGP during the test. A crosshead speed of 1.0 mm/min was applied until the EGP was pulled apart, and the maximum load was recorded in Newtons. The room temperature was maintained at 29 ± 1°C.

Microhardness test

Microhardness for Group 2 EGPs was assessed before and after IR exposure using a microhardness tester (CMS Metrology, Model CHV-1, Querétaro, Mexico). A force of 0.98N (0.1kgf) was applied with a diamond indenter for 10 seconds. Measurements were recorded in Vickers hardness number (VHN), calculated with the equation: VHN = 1.854 (L/d^2), where L is the applied load (kgf) and d is the mean diagonal length (mm). The final value was derived from three indentations on different areas of one side of each EGP.

Surface microstructural observations (SEM)

EGPs from Group 3 were examined for potential microstructural surface changes due to IR exposure. Both IR-exposed and non-exposed EGPs were mounted on a holder and scanned with an SEM (Hitachi TM1000, Mito City, Japan) operating at 15 kV. Images were captured from at least three different locations at various magnifications using a backscattering electron detector.

Organic-inorganic proportion

To differentiate and quantify the organic and inorganic components of each brand, a recognized method was employed (19). Briefly, 1 g of EGPs from each brand was dissolved in 20 ml of chloroform for 24 hours. The solution was then centrifuged for 15 minutes at 10,000 rpm. The inorganic components solidified and were separated from the organic supernatant; solids were collected by filtration and the mass of both phases was determined after evaporating the solvent.

Statistical analysis

The results were statistically analyzed using the Student's t-test and the paired t-test, as appropriate, and two-way ANOVA with post hoc Tukey-Kramer multiple comparison test, following the normal distribution confirmation by the Smirnov-Kolmogorov test. All analyses were conducted using GraphPad Instat, version 3.0 (GraphPad Software, San Diego, CA, USA). Statistical significance was set at $p < 0.05$.

Results

The tensile strength analysis of the EGPs not subjected to IR revealed that the Hygienic and Dentsply brands had similar tensile strengths, yet both were significantly different ($p<0.0001$) from the Meta-Biomed brand, which exhibited the lowest tensile strength. Furthermore, after IR exposure, the Meta-Biomed brand was the only one to experience a significant decrease in tensile strength ($p<0.0001$). Conversely, the Dentsply and Hygienic brands did not show any significant change in tensile strength following IR exposure (Table 2). A similar pattern was observed in the microhardness test; the Meta-Biomed EGPs underwent a significant increase in microhardness ($p<0.0001$) after IR exposure, indicating they were the most affected among the brands tested (Table 3).

The surface microstructure of the three EGP brands, when not exposed to IR, displayed smooth, homogeneous surfaces with uniform contrast at lower magnifications (x250, x500). However, at higher magnifications (x2000, x10000), several particles with higher contrast, all smaller than one micron, were observed, immersed in a matrix of organic elements with lower contrast. After IR exposure, the Hygienic brand exhibited no noticeable changes on its surface. Meanwhile, the Dentsply EGPs displayed a few dark lines and striations across their entire surface. These features were even more abundant in the Meta-Biomed brand, covering a large surface area. Additionally, some of these lines were deep, and cavities of varying depths and extents were noted in the center of some, resulting in irregular and non-smooth surfaces (Figure 1). With respect to the organic/inorganic content in each group, very similar proportions in the Dentsply and Hygienic EGPs were observed. In contrast, a high proportion of organic components was found in Meta-Biomed EGP (Table 4).

Table 2: *Tensile strength (Newtons) of different brands of EGPs exposed or not to IR*

	Hygenic (n=20)	Dentsply (n=20)	Meta-Biomed (n=20)	P value[b]
		X ± SD (Range)		
No IR (n=10)	5.11 ± 0.57 (4.15 - 6.15)	4.97 ± 0.64 (3.85 - 5.80)	3.73 ± 0.60 (2.75 - 4.50)	≤ 0.0001
After IR (n=10)	4.67 ± 0.63 (3.80 - 6.10)	4.58 ± 0.39 (4.10 - 5.20)	1.57 ± 0.35 (1.05 - 2.40)	≤ 0.0001
P value[a]	0.0636	0.1115	≤ 0.0001	

X: Mean; SD: Standard deviation; EGPs: Endodontic gutta-percha points; IR: Ionizing radiation; [a]: Student t-test; [b]: ANOVA; Post hoc Tukey-Kramer test in the no radiated and radiated groups comparisons resulted in no statistical significance when comparing Hygenic Vs. Dentsply, while Hygenic Vs. Meta-Biomed and Dentsply Vs. Meta-Biomed resulted significative ($p<0.0001$).

Table 3. *Microhardness (HV0.1) of different brands of EGPs before and after being exposed to IR*

	Hygenic (n=15)	Dentsply (n=15)	Meta-Biomed (n=15)	P value[b]
		X ± SD (Range)		
Before IR	49.01 ± 5.00 (42.2 - 55.2)	47.83 ± 4.92 (41.1 - 58.7)	35.36 ± 3.04 (31.6 - 40.8)	≤ 0.0001
After IR	49.36 ± 4.76 (42.8 - 56.0)	49.74 ± 6.59 (41.7 - 60.5)	43.60 ± 2.66 (39.8 - 47.6)	0.0161
P value[a]	0.1222	0.1146	≤ 0.0001	

X: Mean; SD: Standard deviation; EGPs: Endodontic gutta-percha points; IR: Ionizing radiation; [a]: Paired t-test; [b]: ANOVA; Post hoc Tukey-Kramer test in pre-Ionizing Radiation comparisons resulted in no statistical significance ($p>0.05$) when comparing Hygenic Vs. Dentsply. Hygenic Vs. Meta-Biomed, and Dentsply Vs. Meta-Biomed were different ($p<0.0001$). Comparing post-Ionizing Radiation groups: Hygenic Vs. Dentsply were not statistically significant, while Hygenic Vs. Meta-Biomed and Dentsply Vs. Meta-Biomed were ($p<0.01$).

Discussion

Patients undergoing therapy for head or neck cancer typically receive a cumulative dosage ranging from 30 to 70 Gy over five to seven weeks (18), which is sufficient to cause numerous undesirable changes in oral tissues, including mucosal, muscular, vascular, osseous, and dental tissues (20). Before therapy, patients subjected to IR should be orally evaluated and receive periodontal, dental, and endodontic treatments to eliminate all oral diseases and prevent or minimize complications in the post-IR period, thus providing better oral health conditions (21). Since the risk of developing osteoradionecrosis persists throughout a patient's life,

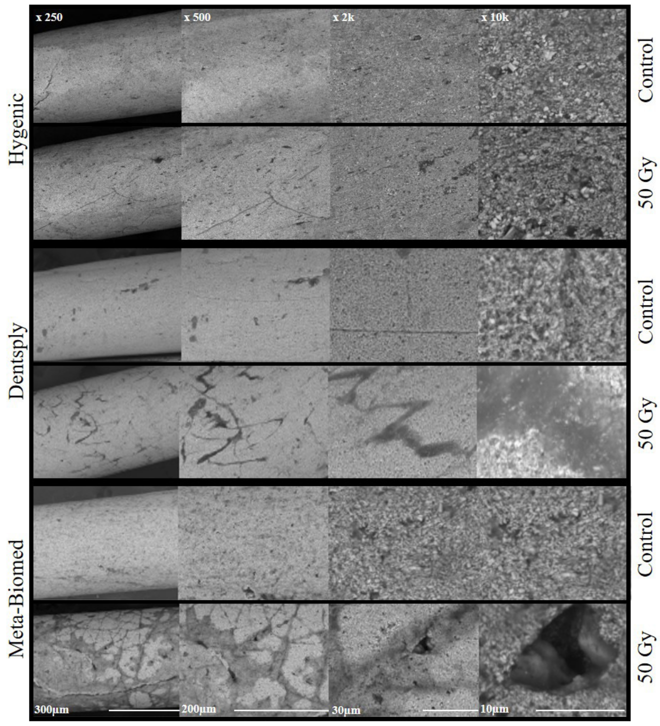

Figure 1. *Scanning electron microscope photographs at different magnifications (250, 500, 2000, 10,000 x) representatives of the surface microstructure of each EGP brand without IR and after IR exposure (50Gy). The scale bars indicated at the bottom apply to all images in the same column. Note the erosion of the Meta-Biomed brand after IR exposure.*

Table 4: *Percentage of organic and inorganic content in each EGP brand*

	Hygenic	Dentsply	Meta-Biomed
Organic	13.55 %	13.70 %	15.62 %
Inorganic	86.45 %	86.30 %	84.38 %

all efforts must be directed towards preventing extractions. Therefore, root canal treatment, both before and after IR therapy, emerges as an essential alternative for these patients (22), also offering the opportunity to rehabilitate teeth and improve the quality of life. Achieving a successful long-term root canal treatment hinges on several factors, including obtaining an hermetic seal through root canal filling—alongside cleaning and shaping the canal—as one of the key aspects to prevent bacterial passage and recontamination (23).

Despite EGPs being the primary material for root canal filling, achieving a hermetic seal is impossible without endodontic sealers, which come in various compositions (24). An ideal endodontic sealer must adhere firmly to both dentin and EGPs, among other properties. The interaction with dentin or EGPs might vary depending on their composition, leading to expected differences in adhesive properties. There is existing information on the effects of IR on different endodontic sealers and significant data on IR's impact on dentin and enamel (11, 25–27). However, the effects of IR on EGPs remain unclear, although logically, IR could affect them due to their organic and inorganic composition.

Historically, the composition of EGPs has varied over time and by manufacturer. The primary component, zinc oxide, constitutes a wide range of 36.6–75%, imparting antibacterial properties and serving as a vulcanizing agent; gutta-percha polymers account for 18–22%, and barium sulfate, added for radiopacity, ranges from 1.1–31.2% (19, 28, 29). The variance in components and their proportions directly influences the physicomechanical properties of EGPs. This study tested two such properties, providing a reference for the physical effects of IR on EGPs. Tensile strength, significantly correlated with the percentage of gutta-percha polymer (19), and the rigidity of EGPs are affected by the concentration of inorganic components and gutta-percha polymer, with small amounts of plasticizers enhancing flexibility and compactness (30, 31).

The study revealed distinct differences between Hygienic and Dentsply brands compared to Meta-Biomed EGPs, with and without IR exposure, suggesting variations in component composition and proportions. These differences are consistent with surface microstructure changes, potentially linked to their composition (19, 28). However, without detailed information on the exact formulas, establishing clear explanations remains challenging. Although this study began to quantify the organic and inorganic phases present in each brand of EGP, the lack of detailed component analysis is a significant limitation; still, it was observed that Meta-Biomed EGPs had a higher organic content compared to others (32, 33).

IR's harmful effects on organic components could explain the observed surface striations and cavities, resulting from the degradation of organic matter into harmful byproducts (33). This degradation impacts the EGP's internal integrity and, by extension, its inorganic structure, leading to physical changes potentially caused by the thermal effects of IR absorption. The post-IR effects on some EGPs complicate the prognosis for patients receiving IR therapy, as the damage to EGPs adds to that already known to affect endodontic sealers and dentin, compromising adhesion and the success of root canal treatments (34).

This study's in vitro design limits the direct applicability of its findings to clinical situations, as root canal filling materials in patients do not directly receive IR. Further research is necessary to fully understand the effects of IR on EGPs, particularly through the investigation of the exact components and their proportions in each brand, to elucidate which are more susceptible to IR damage.

Conclusion

Despite the study's limitations, it was found that the physicomechanical properties and surface microstructure of Meta-Biomed EGPs were significantly affected by IR at doses typical of conventional head or neck cancer therapy, while other brands showed no such effects.

Türkçe özet: *İyonlaştırıcı radyasyonun endodontik güta-perkaların mikro yapısı ve fiziksel özellikleri üzerine etkisi. Amaç: Baş veya boyun kanserinde radyoterapi gören hastalar genellikle radyasyon etkileri nedeniyle kök kanal tedavilerine ihtiyaç duyarlar. Geleneksel tedavi sırasında kullanılanlara benzer dozlardaki iyonlaştırıcı radyasyonun (IR), farklı markalardaki endodontik güta-perka noktalarının (EGP'ler) yüzeyini ve fizikomekanik özelliklerini etkileyip etkilemediğinin belirlenmesi son*

derece önemlidir ve bu çalışmanın amacını oluşturmaktadır. Gereç ve Yöntem: Üç markaya (Meta-Biomed, Dentsply ve Hygenic) ait 123 EGP, gruplara ayrılarak 25 fraksiyona bölünmüş toplam 50 Gy dozda IR'ye maruz bırakılıp bırakılmadı. Tüm EGP'lere çekme mukavemeti ve mikrosertlik testleri yapıldı. IR'ye maruz kalma nedeniyle olası mikroyapısal yüzey değişikliklerini tanımlamak için taramalı elektron mikroskobu gözlemleri kullanıldı. Her markanın organik-inorganik oranı belirlendi. Bulgular: IR'ye maruz kaldıktan sonra yalnızca Meta-Biomed markasının EGP'leri önemli değişiklikler yaşadı, çekme mukavemetinde önemli bir azalma ve mikro sertlik arttı. Ayrıca yüzey mikro yapısında geniş bir yüzey alanını etkileyen koyu çizgiler görülüyordu; bu çizgilerden bazıları merkezde derindi ve düzensiz ve pürüzsüz olmayan yüzeyler oluşturan, değişken derinlik ve uzantılara sahip boşluklar gözlemlendi. Organik bileşen oranı en yüksek markaydı. Sonuç: Test edilen markalardan biri olan Meta-Biomed'in fiziko-mekanik özellikleri ve yüzey mikro yapısı, konvansiyonel baş veya boyun kanseri tedavisi sırasında kullanılan dozlarda IR'den önemli ölçüde etkilenirken, diğer markalar daha az etkilendi veya hiç etkilenmedi. Anahtar Kelimeler: radyoterapi; endodontik güta-perka; mikro yapı; baş ve boyun kanseri, kanal tedavisi

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