INVESTIGATION OF RADIATION ATTENUATION PROPERTIES OF TI-HA BIOCOMPOSITE

Tİ-HA BİYOKOMPOZİTİN RADYASYON ZAYIFLATMA ÖZELLİKLERİNİN İNCELENMESİ

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Özet

Biyouyumlu malzemelerin radyasyon ortamlarında kullanımı, özellikle medikal ve malzeme bilimi uygulamalarında giderek daha fazla önem kazanmaktadır. Bu çalışmada, hidroksiapatit (HA) tabanlı ve titanyum (Ti) katkılı biyokompozit yapının sinterlenmemiş formundaki radyasyon zayıflatma özellikleri deneysel olarak incelenmiştir.

Deneysel çalışmada, öncelikle hacimce %95 HA (2,83 g) ve %5 Ti (0,212 g) içeren homojen toz karışımı hazırlanmıştır. Daha sonra hacimce %60 oranındaki 95HA+5Ti toz karışımına hacimce %40 oranında gözenek oluşturucu NaCl (1,36 g) eklenerek nihai homojen toz karışımı elde edilmiştir. Elde edilen bu karışıma bağlayıcı olarak kuru 0,09 gram (ıslak 1,8 g) PVA eklenerek homojenizasyonun sağlanması için tekrar karıştırılmıştır. Hazırlanan bu karışım 10mm çapındaki kalıpta 1,25 ton kuvvetinde basılarak 3,52 mm kalınlığında pelet haline getirilmiştir.

Numune, silindirik kalıpta şekillendirilmiş ve sinterlememiş haliyle değerlendirilmiştir. Radyasyon ölçümleri, 59,5 keV enerjili Am-241 gama kaynağı ve HPGe (yüksek saflıkta germanyum) dedektörü kullanılarak gerçekleştirilmiştir.

Elde edilen sonuçlara göre Ti-HA numunesi için lineer zayıflatma katsayısı 0,6629 cm^{-1}, ortalama serbest yol 1,5085 cm, yarı değer kalınlığı 1,0454 cm ve radyasyon koruma verimliliği (RPE) %20,81 olarak hesaplanmıştır. Bu bulgular, sinterlenmemiş Ti-HA-NaCl biyokompozitin düşük enerjili gama radyasyonuna karşı belirli bir zayıflatma kapasitesine sahip olduğunu göstermektedir.

Çalışmanın ilerleyen aşamalarında aynı kompozitin sinterlenmiş ve suda yıkanarak NaCl'den arındırılmış böylece de gözenekli hale gelmiş formu da hazırlanarak deneysel olarak incelenecek ve sinterleme işleminin radyasyon zayıflatma özellikleri üzerindeki etkisi karşılaştırmalı olarak değerlendirilecektir. Bu yönüyle çalışma, biyouyumlu ve fonksiyonel radyasyon zayıflatıcı malzemelerin geliştirilmesine katkı sunmayı amaçlamaktadır.

Anahtar kelimeler: Titanyum, Hidroksiapatit, Biyokompozit, Radyasyon Zayıflatma, Gözenek Yapıcı NaCl

Abstract

The use of biocompatible materials in radiation environments is gaining increasing importance, particularly in medical and materials science applications. In this study, the radiation attenuation properties of a hydroxyapatite (HA)-based and titanium (Ti)-reinforced biocomposite structure were experimentally investigated in its unsintered form.

In the experimental procedure, a homogeneous powder mixture containing 95% HA (2.83 g) and 5% Ti (0.212 g) by volume was initially prepared. Subsequently, the final homogeneous mixture was obtained by adding 40% pore-forming NaCl (1.36 g) by volume to the HA-Ti powder mixture, which constituted 60% of the total volume. To ensure homogenization, 0.09 grams of dry (1.8 g wet) PVA was added as a binder and mixed again. The resulting mixture was pressed in a 10mm diameter die under 1.25 tons of force to produce a pellet with a thickness of 3.52 mm.

The sample was shaped in a cylindrical die and evaluated in its unsintered state. Radiation measurements were performed using an Am-241 gamma source with an energy of 59.5 keV and an HPGe (High-Purity Germanium) detector. According to the results obtained, the linear attenuation coefficient for the Ti-HA sample was calculated as 0.6629 cm^{-1}, the mean free path as 1.5085 cm, the half-value layer as 1.0454 cm, and the radiation protection efficiency (RPE) as 20.81%. These

findings indicate that the unsintered Ti-HA-NaCl biocomposite possesses a specific attenuation capacity against low-energy gamma radiation.

In the subsequent stages of the study, the sintered form of the same composite-purified from NaCl by water leaching to achieve porosity-will be prepared and experimentally analyzed, and the effect of the sintering process on radiation attenuation properties will be evaluated comparatively. In this respect, the study aims to contribute to the development of biocompatible and functional radiation-attenuating materials.

Keywords: Titanium, Hydroxyapatite, Biocomposite, Radiation Attenuation, Porogen NaCl

1. INTRODUCTION

The behavior of biocompatible materials in radiation environments is of critical importance today in both medical diagnostic methods and radiotherapy applications (Chu et al., 2002). Pure metallic implants, particularly commercially pure titanium and its alloys, are widely preferred in orthopedic applications due to their high mechanical strength and excellent corrosion resistance. However, a significant clinical limitation of these solid metallic implants is the "stress shielding" effect. This phenomenon occurs because metallic implants possess a much higher modulus of elasticity compared to natural human bone, leading to bone resorption and implant loosening (Torres et al., 2012; Neto et al., 2024). To address this, biocomposite structures, where a Hydroxyapatite (HA) matrix is reinforced with Titanium (Ti) particles, have been developed to mimic the natural mineral phase of bone while providing structural reinforcement (Chu et al., 2004; Watari et al., 2004).

In the selection of these implant materials, radiological properties are as vital as biomechanical compatibility for long-term patient care. While HA exhibits superior biocompatibility and osteoinductive properties, its high brittleness restricts its use in load-bearing applications unless combined with a reinforcement phase like titanium (Fan, 2019). The development of porous scaffolds is essential to match the elastic modulus of the implant to the surrounding bone tissue (Pérez et al., 2015). Although the literature extensively discusses the bioactivity and mechanical regulation of HA/Ti scaffolds (Fan, 2019), the radiological behavior of these composites, especially at different production stages, requires precise quantification.

The fabrication of such porous structures typically utilizes Powder Metallurgy (PM) in conjunction with the Space Holder Technique (SHT), which allows for controlled density and complex pore architectures (Jha et al., 2013; Salleh et al., 2017). Sodium Chloride (NaCl) is a preferred temporary space holder because it is low-cost, non-toxic, and can be efficiently removed through dissolution in hot water (Torres et al., 2012; Jha et al., 2013). To ensure sufficient green strength in cold-compacted pellets, organic binders such as polyvinyl alcohol (PVA) are incorporated into the powder mixture (Jha et al., 2013). The relationship between porosity and spacer content is known to be linear, allowing for predictable adjustments to the material's structural properties (Jian et al., 2015).

Within the scope of this study, the radiological characterization of a Ti-HA biocomposite structure, designed with a target controlled porosity of 40% using NaCl as a space holder and PVA as a binder, was conducted at the initial stage of the production process (unsintered form). By examining the interaction of 59.5 keV gamma rays from an Am-241 source with the material, fundamental dosimetric parameters including the linear attenuation coefficient, half-value layer, and radiation protection efficiency (RPE) were calculated. These findings establish a critical baseline to evaluate the specific effects of subsequent sintering and leaching processes-which may induce structural modifications and the formation of new bioactive phases-on the overall radiation attenuation and bio-corrosion performance (Fan, 2019).

2. METHODS

The samples used in the study were prepared using the powder metallurgy method. The preparation workflow is presented in Figure 1.

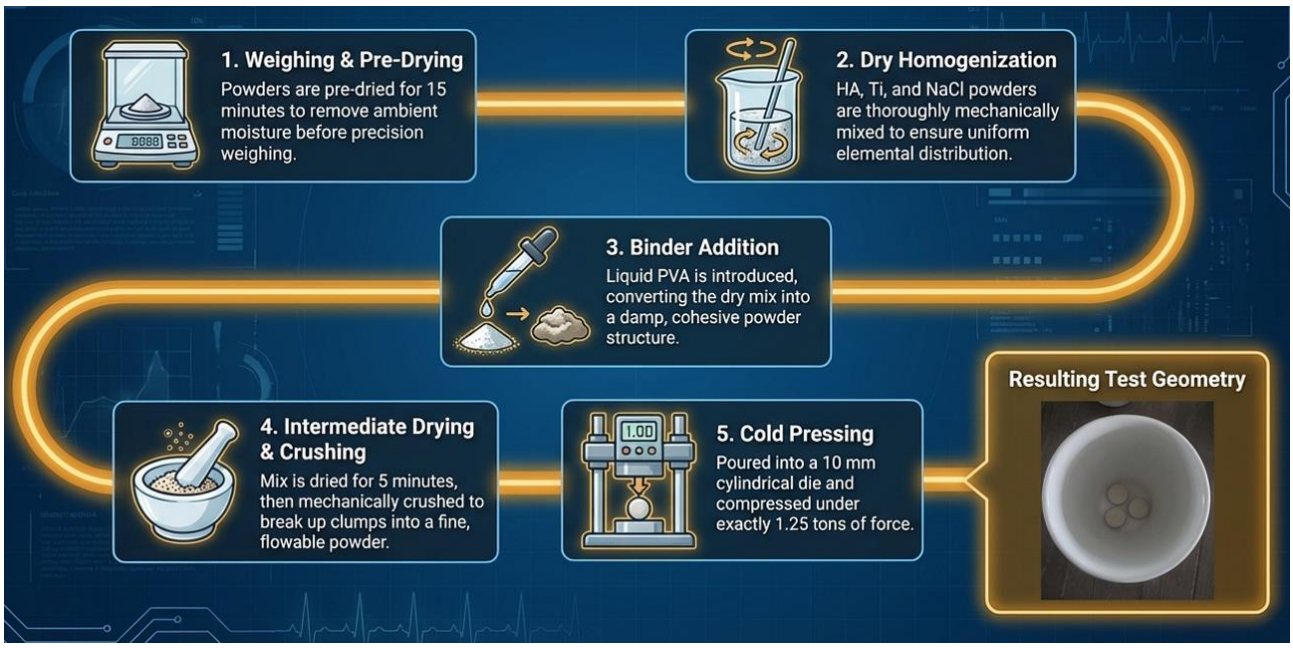


Figure 1. Preparation Workflow

2.1. Sample Preparation:

A main matrix containing 95 vol% HA (2.83 g) and 5 vol% Ti (0.212 g) was prepared; to this mixture, NaCl (1.36 g) was added as a space holder (temporary pore former) at a ratio of 40 vol%. As a binder, 0.09 g of dry PVA moistened with 1.71 g of water was added to the mixture. The resulting mixture was dried at 80°C for 5 minutes and then remixed to ensure a homogeneous distribution. Subsequently, the homogeneously mixed powder was pressed in a 10 mm diameter mold under a force of 1.25 tons. The thickness of the produced pellets was measured as 3.52 mm. The appearance of the obtained pellets is shown in Figure 2.



Figure 2. Specimens

2.2. Radiation Measurements:

Experimental measurements were performed using an Am-241 gamma source with an energy level of 59.5 keV. A high-resolution HPGe (High-Purity Germanium) detector (Figure 3) was used for photon counting.



Figure 3. HPGe (High Purity Germanium) Detector

Measurements were conducted on the material in its initial, unsintered state containing NaCl.

The radiation attenuation and analysis setup presented in the Figure 3 illustrates the structural framework for precise measurements utilizing a high-purity germanium (HPGe) detector. As detailed in the schematic, the experimental setup employs a multi-layered shielding system engineered to minimize environment-induced artifacts and enhance the reliability of the spectroscopic analyses.

The primary stage of the system, designated as Environmental Isolation, consists of a thick lead (Pb) outer armor that acts as an absolute barrier, effectively blocking all ambient background radiation present in the laboratory. Furthermore, to prevent secondary X-rays (Bremsstrahlung radiation)-which may be generated through the interaction of incident radiation with the external lead shield-from inducing signal noise within the HPGe detector, the inner surface of the shield is supplemented with a Secondary Shielding (Inner Lining) fabricated from pure copper (Cu). This copper layer efficiently absorbs backscattered low-energy secondary photons, thereby preserving the measurement sensitivity of the detector.

At the core of the setup's Instrumentation lies an Am-241 radioactive source, which emits 59.5 keV low-energy gamma photons and constitutes the foundation of the measurement process. As shown in Figure 3, these photons are targeted directly through the precise center of the 3.52 mm thick Titanium-doped Hydroxyapatite (Ti-HA) composite specimen. The attenuated gamma rays propagating through the specimen are subsequently detected with high energy resolution by the HPGe detector positioned beneath the apparatus. Consequently, the radiation absorption capacity and attenuation coefficients of the targeted Ti-HA material can be characterized with exceptional accuracy.

3. RESULTS AND DISCUSSION

Figure 4 illustrates the fundamental attenuation mechanics governing the interaction between incoming radiation and the biocomposite structure, modeled according to the Beer-Lambert law. The physical process of radiation shielding is mathematically defined by the equation:

$$I = I_0 e^{-\mu x}$$

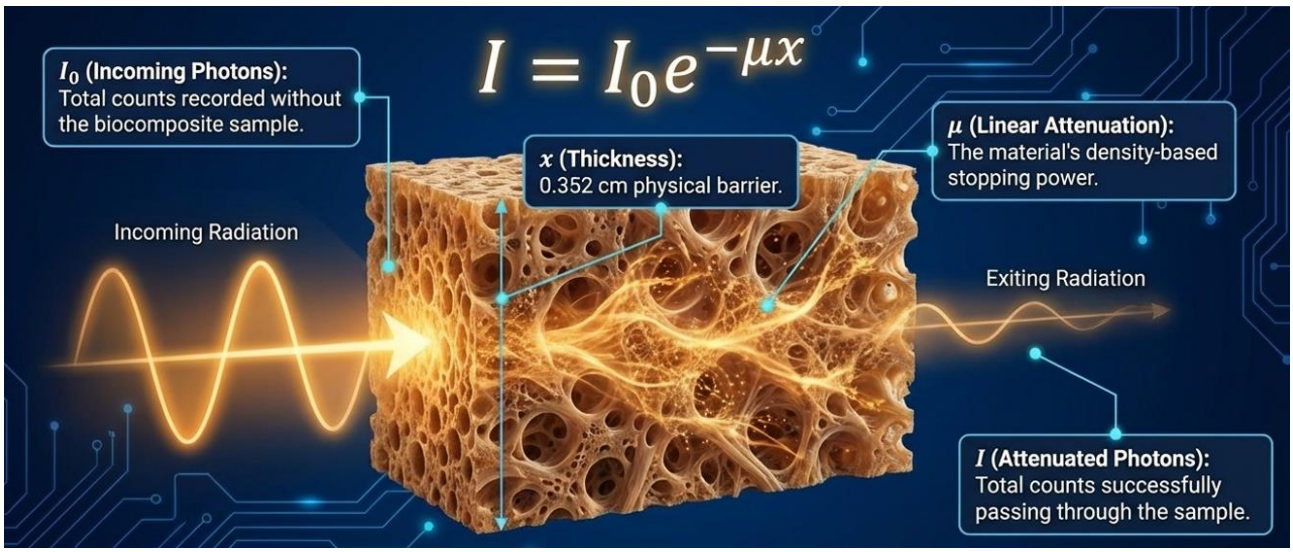


Figure 4. Attenuation Mechanics

In this application, I_0 represents the initial intensity, defined as the total incoming photon counts recorded in the absence of the sample. Conversely, I denotes the intensity of the attenuated photons, which is the total count of radiation that successfully penetrates and exits the material. The variable x corresponds to the physical thickness of the barrier, specifically established as 0.352 cm for this specimen. The parameter μ indicates the linear attenuation coefficient, quantifying the density-based stopping power of the material.

As depicted in the schematic, when the incident radiation enters the porous Titanium-doped Hydroxyapatite (Ti-HA) matrix, the initial photon intensity undergoes a significant reduction. This macroscopic attenuation is primarily driven by fundamental physical interactions at the atomic level-specifically, photoelectric absorption and Compton scattering within the matrix framework. Consequently, these scattering and absorption events collectively dictate the overall shielding efficacy of the Ti-HA composite barrier.

Following the experimental measurements, the fundamental radiation shielding parameters of the Titanium-doped Hydroxyapatite (Ti-HA) biocomposite were quantitatively evaluated. As detailed in the figure, the linear attenuation coefficient (μ) was derived using the rearranged Beer-Lambert equation:

$$\mu = \frac{\ln\left(\frac{I_0}{I}\right)}{x}$$

$$\mu_{Ti} = \ln\left(\frac{3661}{2899}\right)\frac{1}{0.352\ cm} = 0.6629\ cm^{-1}$$

By substituting the recorded initial photon count (I_0) of 3661 and the attenuated photon count (I) of 2899 measured across the 0.352 cm thick specimen, the linear attenuation coefficient was calculated to be 0.6629 cm^{-1}. This foundational metric represents the specific capacity of the Ti-HA composite to absorb or scatter incident gamma radiation per centimeter of material depth.

Furthermore, to fully understand the penetrability of the radiation within the matrix, the Mean Free Path (*MFP,* λ) was determined. Calculated as the reciprocal of the linear attenuation coefficient:

$$\lambda\ (MFP) = {}^{1}/_{\mu}$$

$$\lambda\ (MFP) = {}^{1}/_{0.6629\ cm^{-1}} = 1.5085\ cm$$

The MFP was established at 1.5085 cm. This value indicates the exact average physical distance a single gamma photon can traverse through the Ti-HA composite structure before undergoing a scattering or absorption collision with the constituent atoms. Together, these derived metrics provide critical quantitative insights into the structural efficacy and stopping power of the synthesized barrier material.

The calculation of the half-value layer is shown in Figure 5.

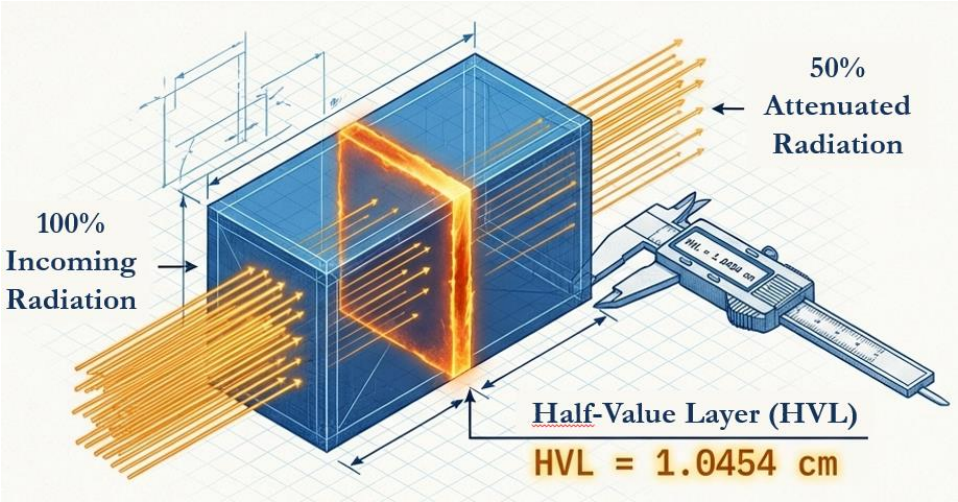


Figure 5. Half-Value Layer Calculation

A solid barrier constructed entirely from this specific unsintered mixture needs to be only ~1.05 cm thick to neutralize exactly half of the incoming 59.5 keV gamma radiation.

$$HVL = {}^{ln2}/_{\mu}$$

$$HVL = {}^{ln2}/_{0.6629\ cm^{-1}} = 1.0454\ cm$$

To evaluate the macroscopic shielding performance of the synthesized material through a holistic approach, the Radiation Protection Efficiency (*RPE*) parameter was calculated. RPE represents the percentage of incident photons successfully absorbed or scattered by the specimen, and is mathematically expressed by the following equation:

$$RPE = \left(1 - \frac{I}{I_0}\right) \times 100$$

$$RPE = \left(1 - \frac{2899}{3661}\right) \times 100 = 20.81\%$$

In this relationship, I_0 denotes the initial photon intensity from the reference (blank) measurement, while I represents the attenuated photon intensity transmitted through the material.

When the experimentally recorded count values-an initial intensity (I_0) of 3661 and a transmitted intensity (I) of 2899-are applied to this equation, the RPE for the 3.52 mm thick specimen is determined to be 20.81%.

This result is highly significant from a materials science perspective. Despite being in a raw (unsintered) state without any densifying heat treatment applied yet, and possessing a relatively thin profile (3.52 mm), the baseline Ti-HA-NaCl biocomposite matrix successfully blocked more than one-fifth (>20%) of the incident gamma radiation.

This demonstrated efficiency rate proves that the material possesses an excellent physical balance profile for its intended biomedical applications. The developed Titanium-doped HA structure provides the necessary radiolucency required for medical diagnostic and imaging systems, while simultaneously demonstrating the potential to function as a structural radiation shield to protect surrounding tissues and equipment in clinical or radiotherapy environments characterized by intense secondary radiation.

Following the experimental measurements, the calculated radiological parameters for the unsintered Ti-HA-NaCl specimen are presented in Table 1:

Table 1. Radiation attenuation parameters of the unsintered Ti-HA sample

Parameter	Symbol / Unit	Value
Linear Attenuation Coefficient	μ (cm^{-1})	0.6629
Mean Free Path	MFP (cm)	1.5085
Half-Value Layer	HVL (cm)	1.0454
Radiation Protection Efficiency	RPE (%)	20.81

The data obtained indicate that despite the sample being in a very thin form (3.52 mm), it successfully blocked 20.81% of the low-energy gamma radiation.

4. CONCLUSION, DISCUSSION, AND RECOMMENDATIONS

This study has established the fundamental radiological profile of the Ti-HA-NaCl biocomposite structure in its unsintered form. The calculated linear attenuation coefficient of 0.6629 cm^{-1} supports the potential of the material to serve as a functional attenuator in low-energy radiation environments. In the next stage of the study, the effect of the porous structure, which will be obtained through the sintering of the samples and the removal of NaCl, on radiation will be examined comparatively. In this way, the impacts of bone implants on dose distribution under radiotherapy can be evaluated more precisely.

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