



Synthesis and Characterization of PMMA/Bi₂O₃:Fe₂O₃ Composites as Gamma Radiation Shielding

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Abstract

This study investigates several mechanical properties, including hardness, as well as the radiation attenuation capacity of samples prepared from PMMA (Polymethyl Methacrylate) as the base material, along with nano-oxides comprising Nano Bismuth Trioxide (Bi₂O₃) and Nano Iron Trioxide (Fe₂O₃) at varying doping percentages (0%, 0.5%, 1%, 3%, 5%). The linear attenuation coefficient (μ), absorptivity (A), and transmittance (T) were determined using the radioactive isotopes Bismuth-207 (Bi²⁰⁷) and Cesium-137 (Cs¹³⁷). The experimental results exhibit a notable enhancement in hardness and radiation attenuation coefficients in samples as the doping percentage increases. Particularly, samples with a 5% doping ratio demonstrated the most promising outcomes. Consequently, they can be regarded as superior shielding materials suitable for radiation applications due to their increased linear attenuation coefficient values. Furthermore, absorptivity values witnessed an increment with heightened radiation effectiveness of the radioactive source and an increase in both shield thickness and doping ratio. In contrast, transmittance values declined with the augmentation of sample thickness and doping ratio.

Introduction

Undesirable exposure to high-energy radiation poses significant risks to both living organisms and inanimate structures. High-energy radiation plays a vital role in various fields, including nuclear industries, medicine,

spacecraft, and agriculture, necessitating the implementation of rigorous radiation protection measures. Historically, lead has been a preferred choice for radiation shielding due to its high density, affordability, and effective radiation attenuation properties. It has been employed in various forms such as sheets, plates, foils, laminates, bricks, and blocks [1]. However, lead exhibits limitations in terms of versatility, chemical stability, mechanical strength, and its inherent toxicity. In light of these shortcomings, extensive research efforts have been underway for the past few decades to identify alternative shielding materials capable of replacing lead. Polymer composites have emerged as a promising avenue in the realm of radiation protection. Researchers have explored diverse types of polymers as matrix materials, integrating various fillers as reinforcements tailored to their specific applications.

Gwaily (2002) [2] conducted a study on the gamma attenuation properties of Galena-rubber composites exposed to 0.662 MeV gamma rays. The study reported a linear attenuation coefficient of 29.5 m^{-1} and a half-value layer (HVL) of 0.023 m for the highest Galena-filled composite (500 phr). In another investigation by Ambala et al. (2017) [3], polymer composites based on Isophthalic resin and filled with Bi_2O_3 were fabricated with varying weight percentages (0, 5, 10, 20, 30, 40, 50, and 60%). The results demonstrated enhanced shielding capabilities against Cs^{137} gamma radiations, suggesting the potential of these composites as shielding materials, particularly for low dose rates.

Cao et al. (2020) [4] produced poly (methyl methacrylate) composites filled with Bi_2O_3 for shielding 0.662 MeV gamma rays, yielding a linear attenuation coefficient of 0.206 cm^{-1} for the 50 wt% Bi_2O_3 composite. Their work compared the shielding performance of these composites with conventional materials and demonstrated favorable shielding properties, especially for lower and medium gamma energies.

In a study by Korkut et al. (2013) [5], epoxy composites incorporating Ferrochromium slag were investigated for their radiation shielding effects against X-rays (85 keV), gamma rays (1250 keV), and neutrons (2–10 MeV). The research found that the inclusion of 50% FeCr slag in epoxy resin provided effective shielding, making it applicable in environments such as radiotherapy rooms, the nuclear industry, and containers for radioactive sources.

Furthermore, various researchers have explored the use of different matrices such as styrene-Polyaniline (Hosseini et al., 2014) [6], high-density polyethylene (Udagani and Seshadri, 2012) [7], and epoxy (Eid et al., 2013) [8], coupled with metal or metal oxides as fillers, primarily focusing on attenuation studies. However, it is noteworthy that only a limited number of studies have delved into comprehending the structural, thermal, and mechanical stability of these composites. These aspects are crucial for the evaluation of any material as an efficient radiation shield.

Experiments

1. Materials

Poly(methyl methacrylate) (PMMA) is a transparent, colorless polymer with a molecular formula of $(C_5H_9O_2)$ and a density of 1.19 g/cm^3 [9]. It belongs to the category of thermoplastic polymers and is used as a base material for preparing polymeric composite materials. PMMA is manufactured by the company Ottobock. This polymer undergoes a transition from a liquid to a solid state when a hardening agent, Benzoyl Peroxide, is added. The hardening agent is in the form of white powder and is added to PMMA in a ratio of 1:100.

2. Preparation of polymer composites

To prepare a 100g sample of pure PMMA polymer, 100g of the polymer is carefully weighed using an electronic balance. Subsequently, 1g of the hardening agent, specifically Benzoyl Peroxide from Central Drug House (CDH), is added to the PMMA at room temperature in a precise 1:100 ratio. The mixture is then meticulously blended using a mechanical mixer for a duration ranging between 25 to 30 minutes, ensuring the achievement of homogeneity.

For the preparation of nano-composite samples comprising PMMA/ Fe_2O_3 / Bi_2O_3 , an appropriate quantity of PMMA polymer is weighed. Following this, the required amount of nano iron oxide and nano bismuth oxide, each in proportions of (0.5, 1, 3, 5) by weight and with a 1:1 ratio, is carefully weighed according to the desired weight fraction for each sample. The blending process is conducted meticulously in an electric mixer for a duration of one hour, aimed at achieving a homogeneous mixture. Subsequently, the mixture undergoes a 30-minute treatment on a vibrating apparatus to eliminate bubbles and ensure uniformity. Following this, the hardening agent is introduced at a precise 1:100 ratio. The mixer continues to operate for a duration ranging from 3 to 5 minutes, during which a rise in the composite's temperature signifies the initiation of the reaction among its constituents.

The samples are left undisturbed within the molds for a period of 24 hours, allowing them to fully solidify. After this curing period, the samples are carefully extracted from the molds.

3. Characterization

Scanning electron microscope

Scanning Electron Microscope (SEM) images were employed to investigate the shape and distribution of (Bi_2O_3 : Fe_2O_3) particles within the interstitial spaces of the PMMA polymer and its forms (1-a, b, c, d, and e). The SEM images reveal that the particles are unevenly distributed within the matrix with an appropriate dispersion. The doping materials (Bi_2O_3 : Fe_2O_3) tend to aggregate at one or more points along the radial fronts, adjacent to the edge pathways, which could potentially result in crack propagation. Furthermore, it has been observed that the

protrusions became less pronounced as the loading of the particles increased, attributed to the reduction in sample stress, resulting in a smoother surface. The particle size for all samples (276.655, 260, 394.67, 312.465, 551.67) nanometers for PMMA with the addition of (0, 0.5, 1, 3, 5) % of (Bi_2O_3 : Fe_2O_3), respectively. The resin's positioning within PMMA within the agglomerations of the added materials was also confirmed. Additionally, larger particle agglomerations of (Bi_2O_3 : Fe_2O_3) were observed in form (1-e) for composites with a 5% filler ratio, but these should be minimized to ensure proper functionality [10].

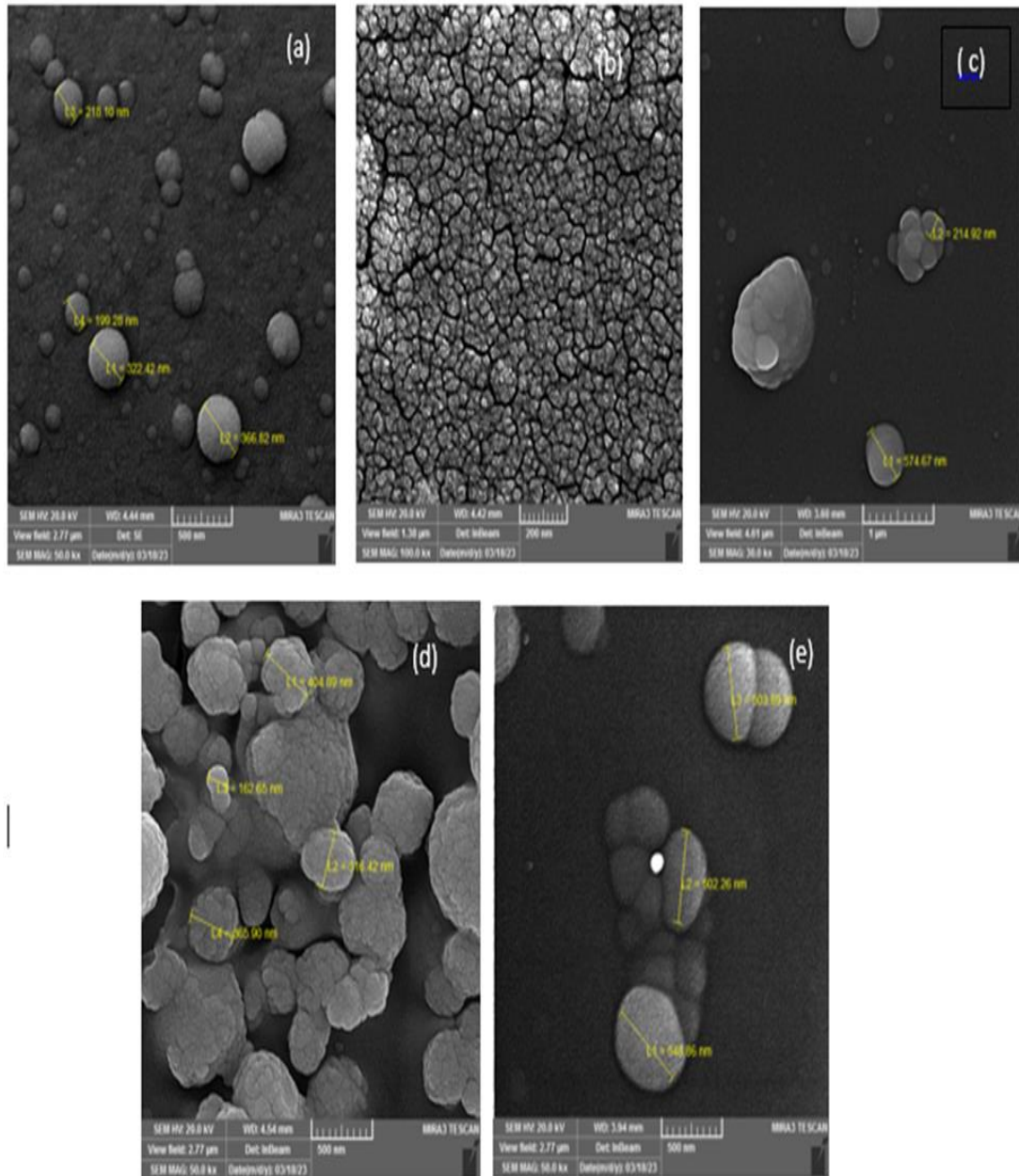


Figure 1. Electron microscope images of samples prepared from PMMA:(Bi_2O_3 : Fe_2O_3)

4. Gamma shielding

Experiments were conducted to shield against gamma radiation, utilizing gamma radiation sources, namely, the highly radioactive cesium source Cs^{137} (with an activity of 4.5 μCi), emitting gamma rays with a high intensity of 48, and the bismuth source Bi^{207} (with an activity of 10 μCi) and an intensity of 122. The exposure time of the samples to gamma radiation was 60 seconds. The sources were placed in front of a lead block, and the radiation exited through an aperture onto the sample. The amount of transmitted radiation, represented as (I), was measured using a Geiger counter.

In this study, various doping percentages were considered, including 5%, 3%, 1%, and 0.5%. These doping ratios were equivalent for both nano iron oxide and nano bismuth oxide. The weight of the PMMA polymer and the nano oxides was 100 grams. Four samples were prepared accordingly.

The linear attenuation coefficient was determined as a function of thickness [11].

$$I = I_0 e^{-\mu x} \quad (1)$$

I_0 represents the intensity of the source, I is the transmitted radiation intensity, x denotes the sample thickness, and μ is the linear attenuation coefficient. The transmittance factor was calculated using the following equation [12]

$$T = \frac{I}{I_0} \quad (2)$$

Similarly, absorptivity was calculated using the equation [13].

$$A = -\log \frac{I}{I_0} \quad (3)$$

5. Hardness test

Hardness tests were conducted for all prepared samples using the Shore D method. This method involves a manual device consisting of a spring-loaded needle-shaped indenter that penetrates the surface of the sample. When the needle penetrates the sample's surface, a reading appears on the device's screen, indicating the hardness

of the sample at that point. Five regions of each sample were tested, and readings were recorded for each of them. The average of these readings was then calculated.

Results and discussion

1. Hardness Test results

The hardness test relies on the resistance to penetration and deformation, serving as a measure of the material's ability to withstand external loading. The Shore D hardness test was conducted to determine the hardness of the nano-composite samples, as indicated in Table 1

Table 1. The hardness values for the nano-composite materials

Impregnation ratio	nanocomposites %			Hardness (Shore D)
	PMMA	Fe ₂ O ₃	Bi ₂ O ₃	
0	6	0	0	71.6
0.5 %	5.97	0.015	0.015	73.4
1 %	5.94	0.03	0.03	79
3%	5.82	0.09	0.09	83.6
5%	5.7	0.15	0.15	89

The results of this test revealed that the addition of nano iron oxide and nano bismuth oxide enhances hardness with increasing doping percentages. The presence of reinforcing materials (oxides) within the base material (PMMA) leads to an increase in hardness values. Furthermore, hardness is significantly influenced by molecular bonding strength, surface type, and temperature, as depicted in Figure 2. These results are consistent with the findings of previous researchers who observed an increase in hardness.[14]

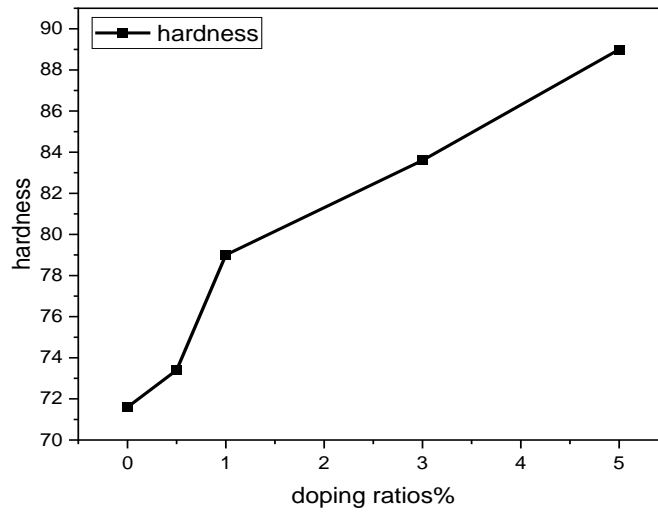


Figure 2 The hardness as a function of doping percentages

2. Radiological tests

Polymeric materials have been increasingly utilized in recent years for protection against harmful nuclear radiation. These materials have been developed by incorporating them with specific ratios of nano bismuth oxide and nano iron oxide to mitigate nuclear radiation. Among the most important tests conducted for attenuating gamma rays emitted from cesium-137 (with an intensity of 48) and bismuth-207 (with an intensity of 122) sources.

Table 2 the values of transmitted and absorbed radiation as a function of sample thickness (mm).

Sample name	Thickness (mm)	Cs ¹³⁷ Source, 4.5 μc			Bi ²⁰⁷ Source, 10μc,		
		I	I/I ₀	logI/I ₀	I	I/I ₀	logI/I ₀
	0	48	1	0	122	1	0
PMMA pure	8	32	0.666	-0.1765	56	0.459	-0.3381
	16	30	0.625	-0.2041	50	0.409	-0.3882
	24	28	0.583	-0.2343	47.5	0.389	-0.4100

	32	24	0.50	-0.3010	44	0.360	-0.4436
0.5% (Bi ₂ O ₃ :Fe ₂ O ₃)	8	30	0.625	-0.2041	47.5	0.389	-0.4100
	16	27	0.562	-0.2502	42	0.344	-0.4634
	24	23	0.479	-0.3196	40	0.327	-0.4854
	32	19	0.395	-0.4034	36	0.295	-0.5301
1% (Bi ₂ O ₃ :Fe ₂ O ₃)	8	28	0.583	-0.2343	44	0.360	-0.4436
	16	25	0.520	-0.2839	40	0.327	-0.4854
	24	22	0.458	-0.3391	35	0.286	-0.5436
	32	18	0.375	-0.4259	31	0.254	-0.5951
3% (Bi ₂ O ₃ :Fe ₂ O ₃)	8	26	0.541	-0.2668	39	0.319	-0.4962
	16	24	0.5	-0.3010	37	0.303	-0.5185
	24	21	0.437	-0.3595	34	0.278	-0.5559
	32	17	0.354	-0.4509	29	0.237	-0.6252
5% (Bi ₂ O ₃ :Fe ₂ O ₃)	8	22	0.458	-0.3391	31	0.254	-0.5951
	16	20	0.416	-0.3809	28	0.229	-0.6401
	24	17	0.354	-0.4509	23	0.188	-0.7258
	32	14	0.291	-0.5361	19	0.155	-0.8096

3. Radioactive transmittance

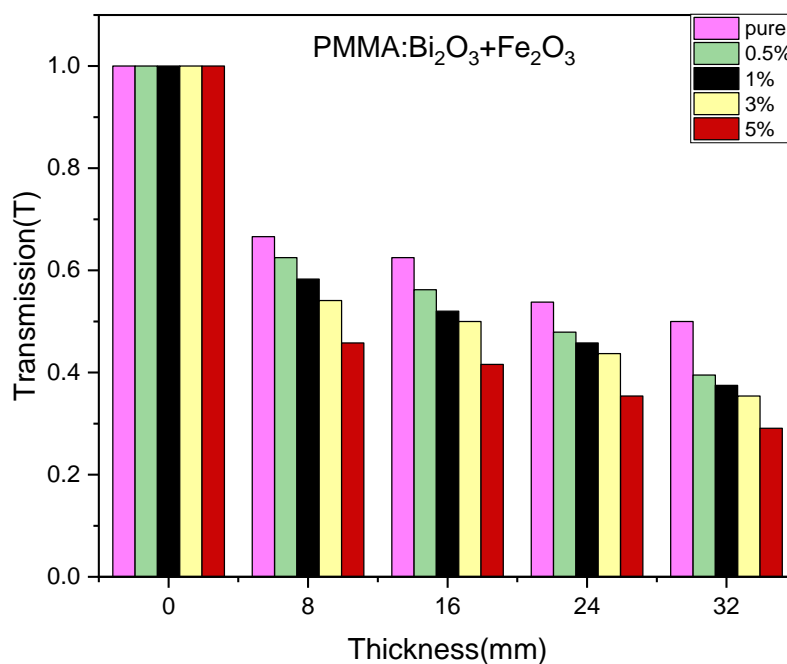
The study of radiation penetration is crucial in determining and studying the attenuation of prepared shields. In this project, Equation (3) was used to calculate the intensity of incident and transmitted radiation for cesium-137 (Cs-137) sources (with an activity of 4.5 μ Ci) and bismuth-207 (Bi-207) sources (with an activity of 10 μ Ci)

as a function of the thickness of the protective shield made of polymethyl methacrylate (PMMA) doped with a composite of bismuth trioxide and iron trioxide ($\text{Bi}_2\text{O}_3:\text{Fe}_2\text{O}_3$).

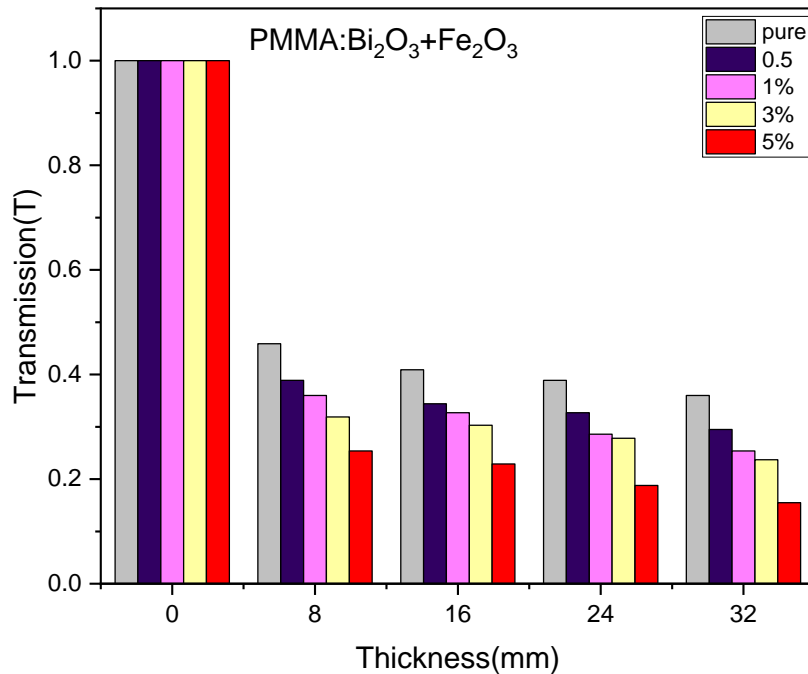
From Table 2, the values of incident and transmitted intensities from both sources were represented as a function of the prepared shield thickness. Plot (1) illustrates the relationship between transmittance and thickness for the cesium source. It is evident that the relationship between transmittance and shield thickness is an inverse one, where transmittance decreases with an increase in thickness and doping ratios. The highest transmittance was observed at 0.666 for the pure PMMA sample, while the lowest transmittance was for the 5% doped sample at 0.291.

Plot (2) demonstrates the linear relationship between incident and transmitted intensities for the same thicknesses for the bismuth source. It is noticeable that these results exhibit the same behavior as the cesium-137 source, with the highest transmittance being 0.459 for the undoped sample and the lowest transmittance being 0.155 for the 5% doped sample.

It is observed that the lowest transmittance was recorded for the 5% doped sample, as the oxides used have good attenuation capability for gamma radiation [16-15]. These findings are consistent with the results obtained by previous researchers [17-18].



Plot 1 The transmittance factor for the cesium-137 (Cs-137) source as a function of thickness



Plot 2 The transmittance factor as a function of shield thickness for the prepared samples for the bismuth-207 (Bi-207) source.

4. Radioactive absorbency

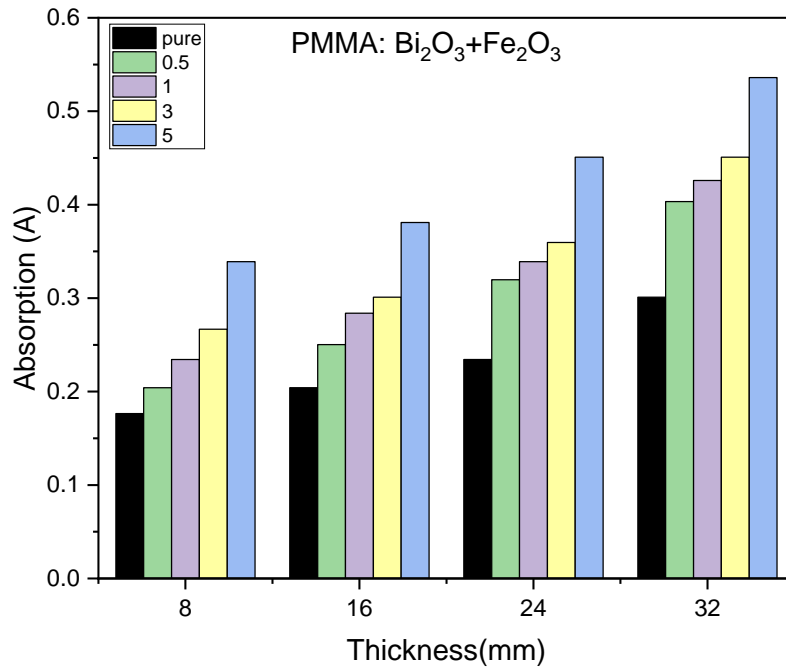
The absorbance is considered an important variable in characterizing radiation shields, and the absorbance values, as calculated and presented in Table 2, were obtained using Equation (3). These values were calculated for both cesium-137 and bismuth-207 sources as functions of shield thickness. The results are depicted in Plots (3) and (4).

Plot (3) shows that the highest absorbance was achieved by the prepared and 5% doped sample, with a value of 0.55 at a thickness of 8 mm. The lowest absorbance was observed for the pure sample, with a value of 0.1857. The absorbance for all prepared samples increased with increasing thickness and doping ratios.

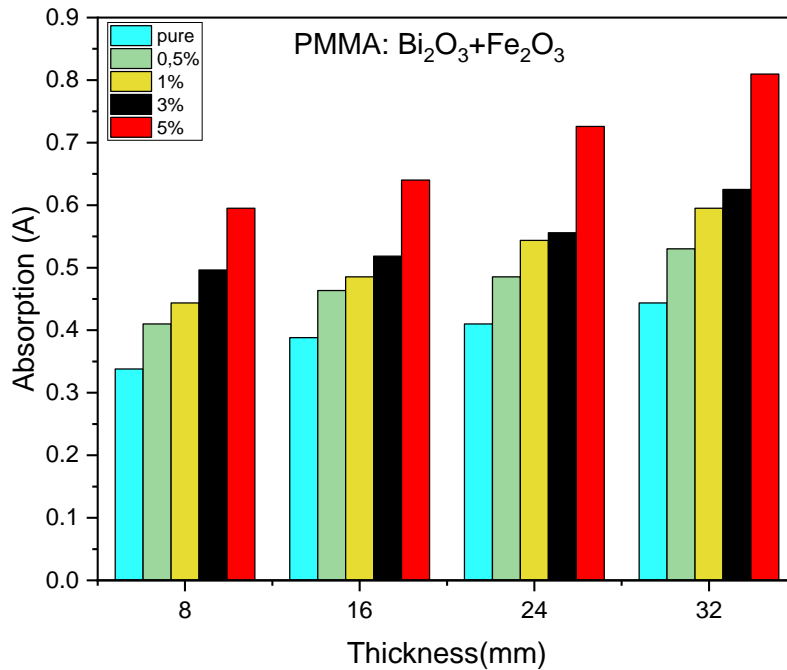
Plot (4), for the bismuth-207 radioactive source, exhibited a similar behavior to the prepared samples using the cesium-137 source. The highest absorbance was recorded at 0.5951 for the 5% doped sample at a thickness of 8 mm, while the lowest absorbance was observed for the pure sample, with a value of 0.3381 at the same thickness. These results are in accordance with the findings of a previous researcher [19].

Additionally, from these two plots, the behavior of absorbance for the prepared shields with varying doping ratios was studied. It was observed that absorbance increased with increasing doping ratios for all thicknesses

and for both radioactive sources. This suggests the effectiveness and adaptability of shield additives in attenuating radiation. This behavior aligns with the relationship between absorbance, thickness, and density.



Plot 3 The absorption coefficient as a function of thickness for the cesium-137 (Cs-137) source



Plot 4 The absorption coefficient as a function of shield thickness for the samples prepared for the bismuth-207 (Bi-207) source

Plots (3) and (4) illustrate the relationship between absorption coefficients and the sample or shield density. It can be observed from the plots, which represent the two radiation sources used in this study, that the absorption coefficients increase with the increase in shield density due to the higher doping ratios at a selected thickness, which is 8 mm in this case. These results clearly indicate that a significant portion of the incident radiation is absorbed by the shielding materials, especially the additives. This behavior aligns with many previous studies in this field [20-21].

5. Linear Attenuation Coefficient (μ)

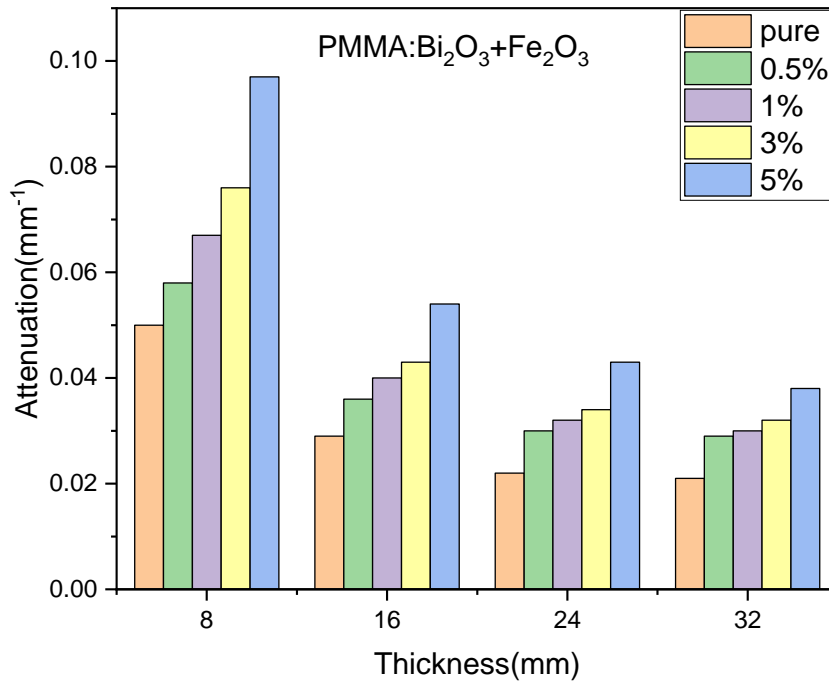
The values of the linear attenuation coefficient (μ) were determined for both cesium-137 (Cs¹³⁷) and bismuth-207 (Bi²⁰⁷) sources for the samples prepared from polymethyl methacrylate (PMMA) with different doping ratios (5%, 3%, 1%, 0.5%, 0%) of the (Bi₂O₃:Fe₂O₃) nanocomposite, as shown in Table (3). The relationship between Ln(I/I₀) as a function of the sample thickness was plotted for both sources. These experimental results provide the linear attenuation coefficient as a function of the thickness of the shields used for the Cs¹³⁷ and Bi²⁰⁷ sources.

Table 3 The experimental results for the linear attenuation coefficient (μ) in mm⁻¹ for the prepared models with different doping ratios.

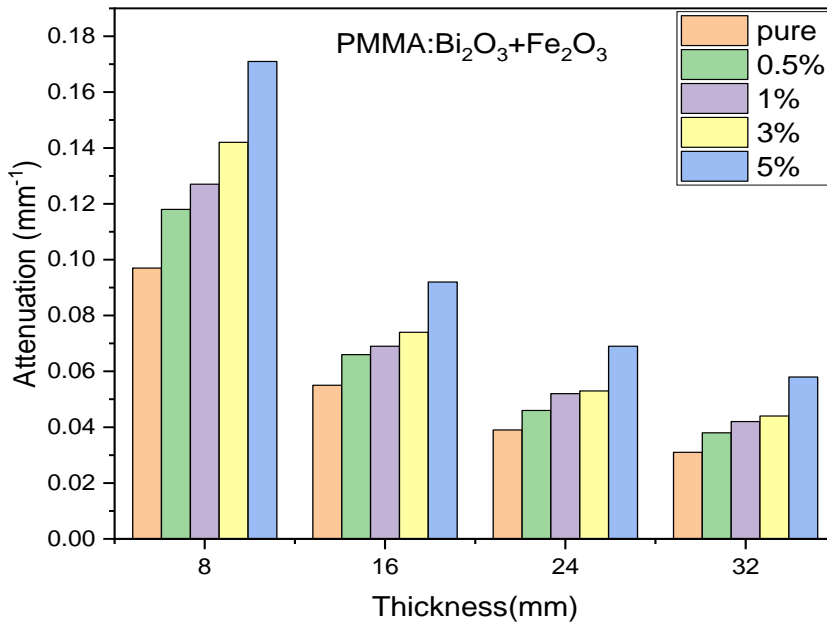
Sample name		Cs ¹³⁷ Source, 4.5 μ c	Bi ²⁰⁷ Source, 10 μ c,

	Thickness (mm)				
		$\mu(\text{mm}^{-1})$	$\mu(\text{mm}^{-1})/\text{sample}$	$\mu(\text{mm}^{-1})$	$\mu(\text{mm}^{-1})/\text{sample}$
PMMA pure	8	0.050	0.030	0.097	0.055
	16	0.029		0.055	
	24	0.022		0.039	
	32	0.021		0.031	
0.5% (Bi ₂ O ₃ :Fe ₂ O ₃)	8	0.058	0.038	0.118	0.067
	16	0.036		0.066	
	24	0.030		0.046	
	32	0.029		0.038	
1 % (Bi ₂ O ₃ :Fe ₂ O ₃)	8	0.067	0.042	0.127	0.072
	16	0.040		0.069	
	24	0.032		0.052	
	32	0.030		0.042	
3% (Bi ₂ O ₃ :Fe ₂ O ₃)	8	0.076	0.046	0.142	0.078
	16	0.043		0.074	
	24	0.034		0.053	
	32	0.032		0.044	
5% (Bi ₂ O ₃ :Fe ₂ O ₃)	8	0.097	0.058	0.171	0.097
	16	0.054		0.092	

	24	0.043		0.069	
	32	0.038		0.058	

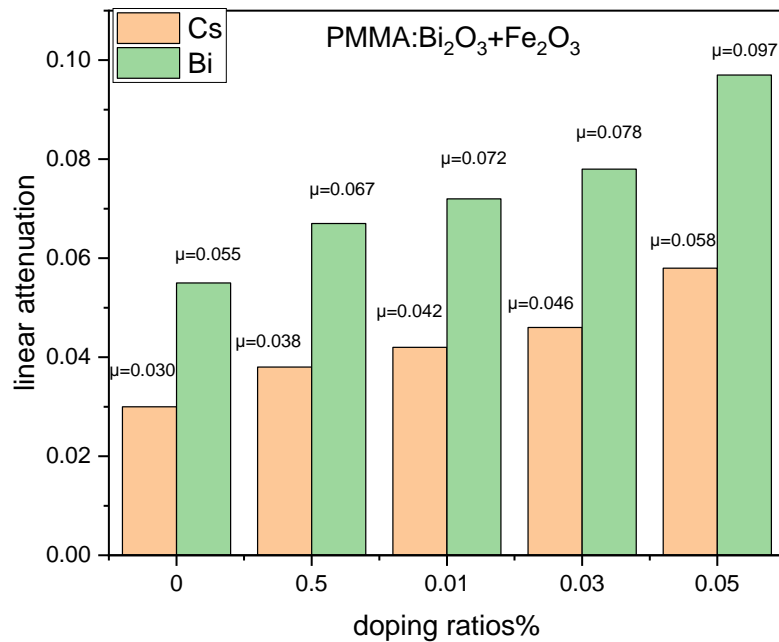


Plot 5 The linear attenuation coefficient (μ) for each thickness of the samples with various doping ratios, using the Cs137 source.



Plot 6 The linear attenuation coefficient (μ) for each thickness of the samples with various doping ratios for the Bi207 source.

The graph (7) illustrates the relationship between linear attenuation and doping ratios using both cesium and bismuth sources. It is evident from the graph that linear attenuation increases with higher concentrations of bismuth and iron, with the greatest effect observed for the bismuth source due to its higher radioactivity compared to the cesium CS¹³⁷ source.



Plot 7 The relationship between the doping ratio and linear attenuation for both the cesium Cs137 and bismuth Bi207 sources

Conclusions

In conclusion, this study investigated the radiation attenuation properties of polymethyl methacrylate (PMMA) when doped with nano-oxides, specifically iron oxide (Fe₂O₃) and bismuth oxide (Bi₂O₃), at varying doping ratios of 5%, 3%, 1%, and 0.5%. The findings of this research reveal several significant outcomes:

- Enhanced Hardness:** The addition of nano-oxides led to a notable increase in the hardness of PMMA, with the most substantial improvement observed at a 5% doping ratio. This enhancement suggests that nano-oxide reinforcement positively impacts the mechanical properties of PMMA.
- Radiation Permeability:** The study showed that radiation permeability, representing the intensity of transmitted gamma rays through shielding materials, decreased with increased sample thickness and doping ratios. Notably, the most effective attenuation was achieved with a 5% nano-oxide doping ratio.
- Linear Attenuation Coefficient (μ):** The linear attenuation coefficient (μ) exhibited an upward trend with increasing sample thickness and doping ratio for all composite samples, indicating improved radiation attenuation capabilities.
- Absorption Coefficient (A):** The absorption coefficient (A) increased proportionally with sample thickness and the addition of nano-oxides, emphasizing the effective absorption of ionizing radiation.

- (e) Optimal Shielding: Among the various doping ratios studied, the 5% doping ratio of nano-oxides ($\text{Fe}_2\text{O}_3 + \text{Bi}_2\text{O}_3$) was identified as the most effective configuration for radiation attenuation in PMMA, thus representing an optimal choice for radiation shielding applications.

These findings signify the potential utility of nano-oxide-doped PMMA as a robust radiation shielding material, particularly when configured with a 5% doping ratio. Moreover, this study contributes to the growing body of knowledge in the field of radiation shielding materials. Further research avenues may explore practical applications and address any limitations encountered during this investigation. It is essential to acknowledge that this study provides valuable insights into radiation attenuation but may have its own constraints and limitations that warrant consideration in future research endeavors.

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