A COMPARATIVE STUDY BETWEEN NANO-CADMIUM OXIDE AND LEAD OXIDE REINFORCED IN HIGH DENSITY POLYETHYLENE AS GAMMA RAYS SHIELDING COMPOSITES

by

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In this work, polymer composites of high density polyethylene reinforced by micro-sized and nanosized cadmium oxide, lead oxide, and a mixture of both with filler weight fraction of 30% were prepared by compression molding technique and characterized by scanning electron microscope. This investigation aims to present a comparative study between cadmium oxide and lead oxide according to their sizes as fillers in high density polyethylene polymeric matrix for gamma-radiation shielding applications. The mass and linear attenuation coefficients of the investigated composites were measured as a function of γ-ray energies ranging from 59.53 keV to 1408.01 keV using standard radioactive point sources (^{241}Am, ^{133}Ba, ^{137}Cs, ^{60}Co, and ^{152}Eu). The measurements were made with a narrow beam geometry setup using a well calibrated hyper pure germanium cylindrical detector. The theoretical values of the mass attenuation coefficients were evaluated using the XCOM program database. The experimental results demonstrated that, according to the filler size, cadmium oxide composite is better as a gamma absorber in the energy region less than 81 keV, while lead oxide composite is better in the energy region greater than 81 keV. Moreover, for the same chemical structure and weight fraction of the composite, nano fillers show better attenuation performance than micro fillers in high density polyethylene based radiation shielding material.

Key words: linear attenuation coefficient, high density polyethylene composites, nano-cadmium oxide, nano-lead oxide, gamma radiation shielding

INTRODUCTION

In the recent decades, the studying of attenuation of γ-rays by polymer composites [1] has become an interesting field of research. This is due to the extensive use of radioactive gamma sources in medicine, agriculture, industry, scientific research and in many applied fields [2]. The γ-rays have a hazardous detriment to nearby instruments and humans in long term exposure [3]. The attenuation characteristics of γ-rays for a specific application are required to determine the amount of shielding necessary [4].

Polymer composites reinforced by inorganic metal oxides were widely studied as protective radiation shielding materials. In the literature, many polymers such as polyethylene [5], recycled polyethylene [6], epoxy [7], polyester [8], styrene butadiene rubber [9], ethylene-propylene-dine monomer (EPDM) [10], polyimide [11], natural rubber [12] and polystyrene [13] were investigated as radiation shielding matrixes. Various metal oxides like PbO [8], $PbWO_4$ [10], Sm_2O_3 [11], WO_3 [14], Bi_2O_3 [15] and Gd_2O_3 [3], with different particle sizes, were used as fillers in the polymeric matrix to shield X-rays [16] and γ-rays. However, the most frequently used filler is PbO due to its high atomic number and high density compared to other metal oxides.

El-Khatib *et al*. [5] studied the effect of micro- and nano-cadmium oxide (CdO) particles, of different loadings dispersed in high density polyethylene (HDPE) on the γ-rays transmission, ranging from 59.53 keV to 1480.01 keV and found that the CdO particles were more effective to attenuate γ-rays specially at lower photon energies. Hence, the aim of this inves-

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tigation is to present a comparative study between CdO and PbO according to their sizes as fillers, in HDPE polymeric matrix for gamma-radiation shielding applications. For this purpose, HDPE filled with 30 wt.% micro CdO, 30 wt.% micro lead oxide (PbO) and 15 wt.% micro CdO + 15 wt.% micro PbO composites were prepared and the mass attenuation coefficients of these composites were evaluated at different photon energies.

MATERIALS AND METHODS

High density polyethylene (HD5403EA grade) with a melt flow index of 0.35 g per 10 minutes and density of 0.955 gcm^{-3} was a commercial product and supplied by Sidpec Sidi Kerir Petrochemicals Company. The CdO and PbO micro fillers, in powder form, with average particle size in the range of 0.5 to 1.0 μm, were purchased from Loba Chemie, India. Lead (II) acetate trihydrate, $Pb(CH_3COO)_2$. $3H_2O$ (FW 379.33 $gmol^{-1}$ and assay >99 %) was purchased from Sigma Aldrich, USA. Sodium hydroxide NaOH (FW 40 $gmol^{-1}$ and assay >96 %) was purchased from BDH, UK. CdO-nano particles with average particle size 50 nm were procured from Nanotech Company (Egypt).

The PbO nanoparticles were synthesized by chemical method [17] by adding 60 ml of 1.0 M $Pb(C_2H_3O_2)$ $_2O$ (lead (II) acetate) aqueous solution at 90 °C to an aqueous solution of 50 ml of 19 M NaOH in a beaker and stirred vigorously. When the solution became peach colored, stirring was stopped, and the precipitate was allowed to settle. The supernatant was then decanted, washed with de-ionized water repeatedly, and dried at 90 °C in an oven, for 8 hours. The resulting product was then removed and lightly grinded by an agate pestle and mortar to obtain amorphous structure.

The investigated micro and nanocomposite sheets were prepared by the compression-molding technique. In this process, HDPE was weighted accurately and then completely melted using two roll mixer at 180 °C for 10 min with the rotator speed of 40 rpm (revolutions per minute). Then, the powdered filler was gradually added with continuous blending for 15 min to obtain a homogenously dispersed composite. Completely mixed sample was then poured into a stainless steel molds of 0.25 cm in thickness for hot-pressing using a hydraulic press with an applied pressure 10 MPa at 180 °C. The produced sheet was let in the press to cool down gradually to the room temperature. Finally, the composite sheet was cut into circular discs of 8.4 cm in diameter to execute γ-radiation shielding tests.

Five standard radioactive point sources [^{241}Am, ^{133}Ba, ^{137}Cs, ^{60}Co, and ^{152}Eu], purchased from Physikalisch-Technische Bundesanstalt PTB in Braunschweig and Berlin, emitting energies in the range from 59.53 keV to 1408.01 keV were used in gamma-radiation measurements. The experimental measurements were carried out using γ-ray spectrometer which consists of a well calibrated Hyper Pure Germanium cylindrical detector (HPGe) from Canberra (Model GC1520), amplifier and multichannel analyzer (MCA). The HPGe detector had a resolution of 1.85 keV at 1.33 MeV γ-ray peak ^{60}Co and relative efficiency of 15 % in the energy range from 50 keV to 10 MeV [18]. The HPGe detector was housed in a lead shield of 15 cm thickness with copper lining on the inside to diminish the background radiations and minimize the X-ray interferences. The distance between the radioactive source and the detector surface was adjusted at 508.67 mm to get very narrow beam and also to ignore the effect of detector dead time [19]. The tested composite of 2.5 mm thickness was placed on a holder between the point source and the detector. The setup of the measurement system is depicted in fig. 1.

For each composite of thickness x and at a certain energy, $N(0)$ and $N(x)$, which are the detector counts without and with the composite target, respectively, were determined from the net area under the photo peak of the recorded γ-ray spectrum. According to Lambert- Beer law [8] the linear attenuation coefficient μ [cm^{-1}] of each composite material can be obtained for γ-ray of appropriate energy by eq. (1)

$$\mu \quad \frac{1}{x} \ln \frac{N(0)}{N(x)} \tag{1}$$

The mass attenuation coefficient μ_m [cm^2g^{-1}], can be determined by dividing μ by the measured density ρ of the sample [20].

CHARACTERIZATION

The size of the synthesized PbO nanoparticles (NP) was analyzed by employing the field emission transmission electron microscope (FE-TEM) (JEM 2100F, JEOL, Japan) at 200 kV. The FE-TEM sample

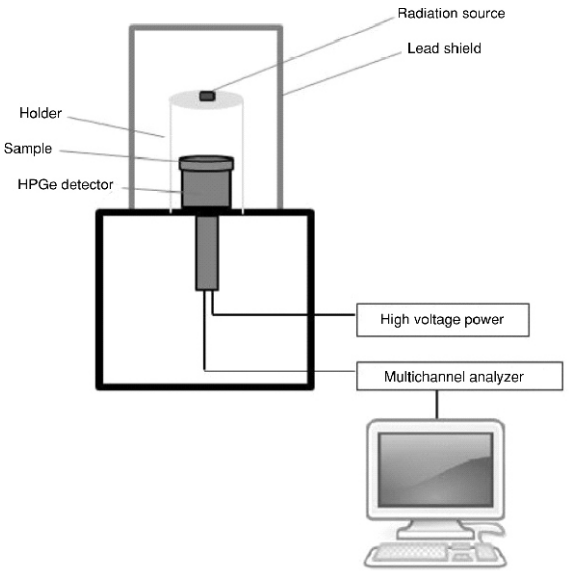

Figure 1. The experimental set-up for measuring -ray shielding properties

was prepared by dispersing the powder in ethanol by ultrasonic vibration on a Cu grid. The average particle size was estimated by averaging a number of measured bars in the obtained images of each sample. Figure 2 displays the image of PbO NP. It is clearly evident from this image that the nanoparticles have average particle size of ~50 nm.

A scanning electron microscope (SAM) (JSM-6010LV, JEOL) was used to observe the morphologies of the prepared composites. The SEM images were obtained at magnification of order 5000x at 20 kV. Figure 3 shows the SEM images of the microstructures of HDPE filled with 30 wt.% micro PbO and 30 wt.% nano PbO. As can be seen from fig. 3, there is a significant variation in the size and dispersion of PbO NP compared to those of micro PbO particles.

Figure 4 compares also the SEM images of the microstructures of HDPE filled with (15 wt.% micro PbO +15 wt.% micro CdO) and (15 wt.% nanoPbO +15 wt.% nanoCdO). Figure 4 verifies the discrepancy

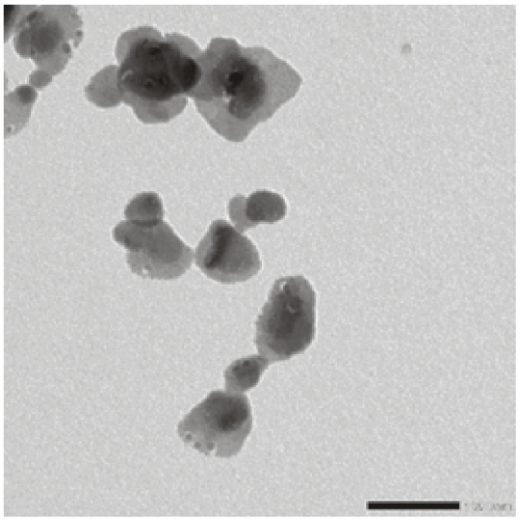

Figure 2. The FE-TEM images of PbO NP

in size and distribution of micro- and nano-particles in the polymeric matrix. Based on the SEM images, the distribution of nano-particles should be more uniform

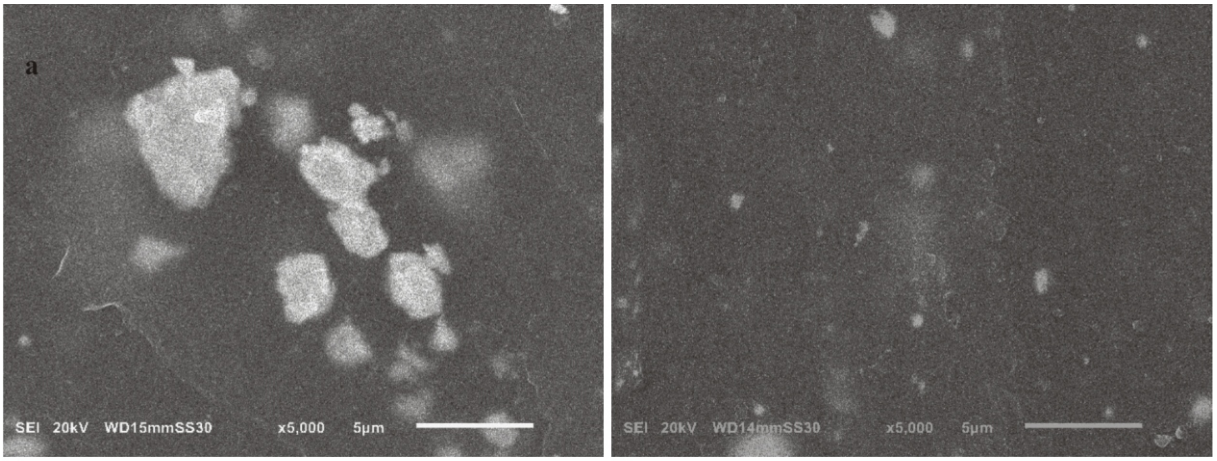

Figure 3. The SEM images of (a) 30 wt.% micro PbO/HDPE, and (b) 30 wt.% nanoPbO/HDPE composites

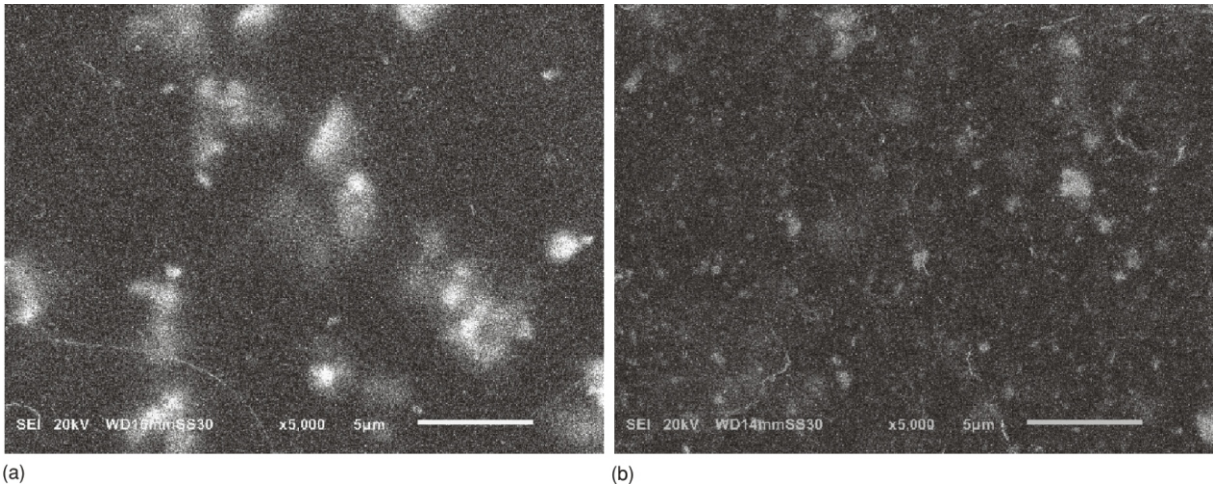

Figure 4. The SEM images of (a) (15 wt.% micro PbO +15 wt.% micro CdO) /HDPE, and (b) (15 wt.% nanoPbO +15 wt.% nanoCdO) /HDPE composites

than micro-particles, so that higher shielding performance is expected by these nanocomposites.

RESULTS AND DISCUSSION

Table 1 lists out the variation of the experimentally measured values of mass attenuation coefficients μ_m, theoretical values of μ_m obtained using XCOM program and the discrepancy (Δ%) between them as a function of γ-ray energy for HDPE filled with 30 wt.% micro CdO, 30 wt.% micro PbO, 15 wt.% micro CdO + 15 wt.% micro PbO composites. It is noted from the magnitude of Δ% that the measured mass attenuation coefficients for all composites are in close agreement with the database of the reference mass attenuation coefficients using XCOM program.

Table 1. The variation of the measured values of μ_m, theoretical values of μ_m obtained using XCOM program and the discrepancy (%) between them as a function of γ energy for HDPE filled with 30 wt.% micro CdO, 30 wt.% micro PbO, 15 wt.% micro CdO + 15 wt.% micro PbO composites

Composite	Energy [keV]	Mass attenuation coefficient [cm^2g^{-1}]		
		Measured	XCOM	Δ [%]
HDPE filled with 30 wt.% micro CdO	59.53	1.66864	1.68200	–0.79 %
	80.99	0.79184	0.79330	–0.18 %
	121.78	0.34174	0.34250	–0.22 %
	244.69	0.14662	0.14650	0.08 %
	344.28	0.11655	0.11670	–0.13 %
	356.01	0.11512	0.11450	0.54 %
	661.66	0.08369	0.08359	0.12 %
	778.90	0.07706	0.07720	–0.18 %
	964.13	0.06937	0.06943	–0.08 %
	1173.23	0.06318	0.06287	0.49 %
	1332.50	0.05857	0.05888	–0.53 %
	1408.01	0.05746	0.05725	0.37 %
HDPE filled with 30 wt.% micro PbO	59.53	1.42788	1.42400	0.27 %
	80.99	0.69596	0.69660	–0.09 %
	121.78	1.01637	1.01500	0.14 %
	244.69	0.25658	0.25600	0.23 %
	344.28	0.16220	0.16170	0.31 %
	356.01	0.15613	0.15580	0.21 %
	661.66	0.09212	0.09210	0.02 %
	778.90	0.08297	0.08283	0.16 %
	964.13	0.07259	0.07267	–0.11 %
	1173.23	0.06487	0.06481	0.09 %
	1332.50	0.06039	0.06033	0.10 %
	1408.01	0.05868	0.05856	0.21 %
HDPE filled with 15 wt.% micro CdO + 15 wt.% micro PbO	59.53	1.55104	1.55300	–0.13 %
	80.99	0.74765	0.74490	0.37 %
	121.78	0.67822	0.67880	–0.08 %
	244.69	0.20215	0.20120	0.47 %
	344.28	0.13893	0.13920	–0.19 %
	356.01	0.13549	0.13510	0.29 %
	661.66	0.08788	0.08785	0.03 %
	778.90	0.08002	0.08001	0.02 %
	964.13	0.07124	0.07105	0.27 %
	1173.23	0.06388	0.06384	0.06 %
	1332.50	0.05951	0.05960	–0.16 %
	1408.01	0.05800	0.05791	0.16 %

As a first approximation from examining tab. 1, one can find that shielding properties approximately depend on the chemical composition of the shielding material and the energy of the incident γ-rays [21]. In addition, μ_m for various photon interaction processes at the start is high and then decreases sharply with the increase of the photon energy up to 200 keV and at higher energies it decreases slightly and this trend is observed for all the investigated composites. Such behavior can be attributed to the dominance of different photon interaction mechanisms for different photon energies. The type of interactions between the attenuated photons and constituent of the absorber at a particular energy play an important role in determination of which probability will take place (photoelectric or Compton, or Compton first then followed by photoelectric effect).

In the energy region below 200 keV the predominant interactions of photons are photoelectric absorption while when the incident photons exceed 200 keV this probability will drop remarkably with energy and the predominant interactions will be Compton scattering. In this probability the photon, to be completely absorbed, must undergo through successive scattering processes that decrease its energy to the energy region where the probability to be absorbed by photoelectric effect is high, otherwise the photon will escap from the medium losing only a part of its energy.

In order to compare the γ-ray shielding properties of composites filled with CdO to composites filled with PbO and those filled with a mixture of both, according to their sizes at different γ-ray energies, fig. 5 is introduced. Figure 5 shows the linear attenuation coefficients versus photon energy for pure HDPE, 30 wt.% micro CdO composite, 30 wt.% micro PbO composite and 15 wt.% micro CdO + 15 wt.% micro PbO composite.

Figure 5 indicates that the interaction of photon with the absorbing medium depends on the incident photon energy, chemical composition of the absorbing material and the size of the filler. It is observed from fig. 5 that, at photon energy in the range from 59.53 keV to 80.99 keV, the linear attenuation coefficient of composites filled with CdO is higher than those filled with PbO at the same filler wt.%, which indicate a higher absorbance of CdO in this range. The mass attenuation coefficients of all composites decrease sharply as the photon energy increases in this range.

At energy 121.78 keV, it is noticed from fig. 5 that the value of the linear attenuation coefficient of PbO filled composites are superior over those filled with CdO. This peak is due to appearance of the *K* absorption edge of Pb at 88 keV [22], thus PbO composites have a higher absorbance of γ-ray compared to CdO composites at this region. Furthermore, in the energy region

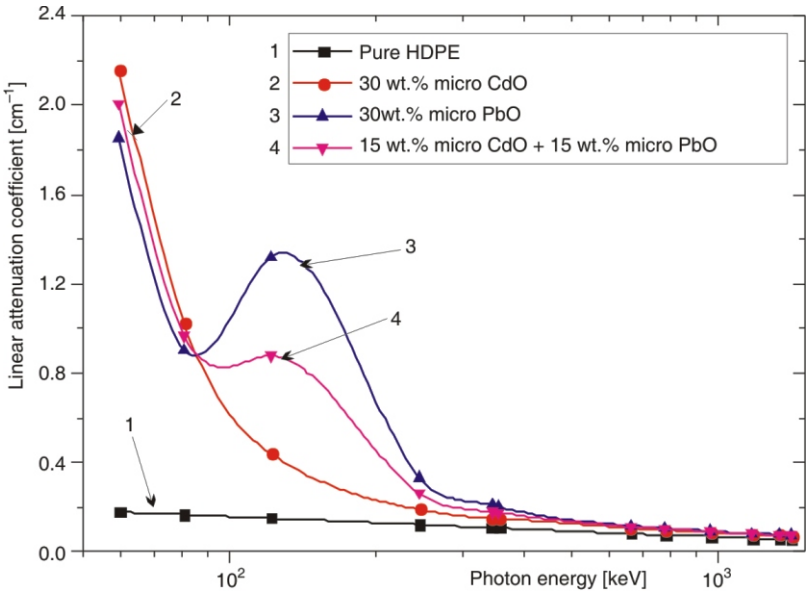

Figure 5. The linear attenuation coefficients *vs.* photon energy for pure HDPE, 30 wt.% micro CdO composite, 30 wt.% micro PbO composite and 15 wt.% micro CdO + 15 wt.% micro PbO composite

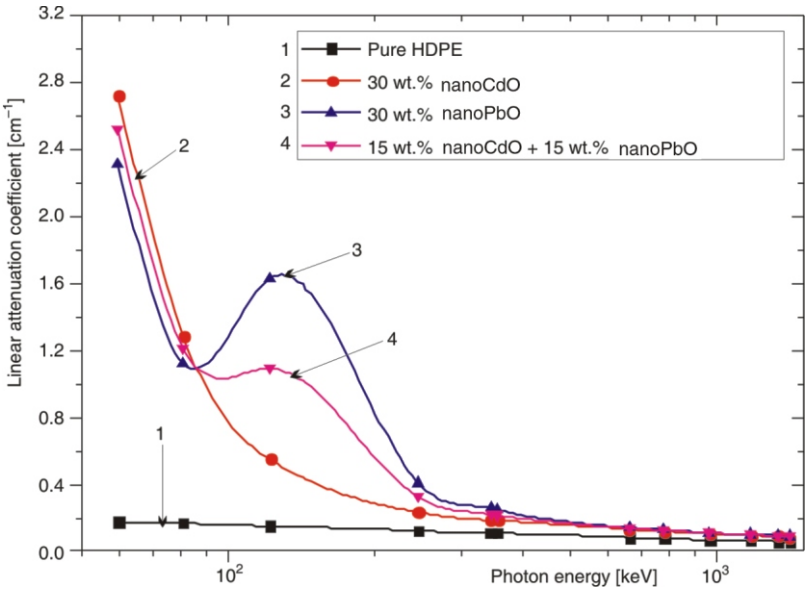

Figure 6. The linear attenuation coefficients *vs.* photon energy for pure HDPE, 30 wt% nanoCdO composite, 30 wt% nanoPbO composite and 15 wt% nanoCdO + 15 wt% nanoPbO composite

(121.78 keV-661.66 keV), the linear attenuation coefficient of both composites decrease with increasing the photon energy at all the filler wt.%.

Moreover, in the energy region (661.66 keV -1408.01 keV), the values of μ of composites filled with CdO and composites filled with PbO approximately have the same value over this energy range. This is because at this intermediate energy range, the effect of photoelectric absorption decreases and the Compton scattering become the dominant mechanism. The probability of Compton effect depends on the number of electrons per unit mass which is proportional to the ratio of the atomic number to the atomic weight (Z/M) and this ratio is approximately equal to 0.5 for all elements except for hydrogen and the heavy elements [23]. That is to say, at energies where the Compton scattering dominates, values of μ tend to be nearly the same for all elements. As a result, changing the filler in polyethylene matrix in this energy range does not have a remarkable change in the value of μ at certain energy.

As can be seen from fig. 5, the curves of μ *vs.* photon energy of the HDPE composites filled with a mixture of both CdO and PbO are always between those of CdO/HDPE composites and PbO/HDPE composites in any energy region. It is observed that the mixture composites are better than PbO/HDPE composites as gamma absorber in the energy region less than 81 keV, while the mixture composites are better than CdO/HDPE composites in the energy region greater than 81 keV.

Table 2 presents a comparison between the measured values of linear attenuation coefficients of nanocomposites *vs.* micro composites for HDPE filled with 30 wt.% CdO, 30 wt.% PbO, 15 wt.% CdO + 15 wt.% PbO composites at 59.53, 80.99, 121.78, 244.69,

Table 2. Comparison between the linear attenuation coefficients values of the investigated micro composites vs. nanocomposites and the relative increase rate at different g-ray energies

Sample	Energy [keV]	Linear attenuation coefficient [cm^{-1}]		
		Nano composites	Micro composites	δ [%]
30 wt.% CdO	59.53	2.7212	2.15421	26.32 %
	80.99	1.28656	1.02226	25.85 %
	121.78	0.55114	0.44119	24.92 %
	244.69	0.2349	0.18928	24.10 %
	344.28	0.18626	0.15046	23.79 %
	356.01	0.18369	0.14862	23.60 %
	661.66	0.13262	0.10804	22.75 %
	778.9	0.1214	0.09949	22.02 %
	964.13	0.10866	0.08956	21.33 %
	1173.23	0.09836	0.08156	20.60 %
	1332.50	0.09052	0.07561	19.72 %
	1408.01	0.08858	0.07418	19.41 %
30 wt.% PbO	59.53	2.31480	1.85339	24.90 %
	80.99	1.12159	0.90336	24.16 %
	121.78	1.62741	1.31925	23.36 %
	244.69	0.40808	0.33304	22.53 %
	344.28	0.25676	0.21054	21.95 %
	356.01	0.24686	0.20266	21.81 %
	661.66	0.14495	0.11957	21.23 %
	778.9	0.13001	0.10769	20.73 %
	964.13	0.11309	0.09422	20.03 %
	1173.23	0.10057	0.08420	19.44 %
	1332.50	0.09324	0.07839	18.94 %
	1408.01	0.09001	0.07617	18.17 %
15 wt.% CdO + 15 wt.% PbO	59.53	2.52597	2.00860	25.76 %
	80.99	1.21287	0.96821	25.27 %
	121.78	1.09339	0.87830	24.49 %
	244.69	0.32423	0.26179	23.85 %
	344.28	0.22169	0.17992	23.22 %
	356.01	0.21575	0.17546	22.96 %
	661.66	0.13891	0.11380	22.07 %
	778.9	0.12618	0.10363	21.76 %
	964.13	0.11163	0.09226	21.00 %
	1173.23	0.09956	0.08272	20.36 %
	1332.50	0.09215	0.07706	19.58 %
	1408.01	0.08944	0.07511	19.08 %

344.28, 356.01, 661.66, 778.9, 964.13, 1173.23, 1332.5, and 1408.01 keV. The relative increase rate δ [%] values between μ of nanocomposites and micro composites are also tabulated in tab. 2 and calculated by eq. (2)

$$\delta \quad \frac{\mu_{nano} \quad \mu_{micro}}{\mu_{micro}} 100[\%] \qquad (2)$$

Figure 6 depicts the linear attenuation coefficients *vs*. photon energy for pure HDPE, 30 wt.% nanoCdO composite, 30 wt.% nanoPbO composite, and 15 wt.% nanoCdO + 15 wt.% nanoPbO composite. The same behavior and trends were obtained as in fig. 5 for the case of micro composites, however, nano composites always have higher μ values than micro composites over the entire range of photon energies.

It is obvious from tab. 2, the values of the linear attenuation coefficients for nanocomposites are all higher than those of micro composites for the same wt.% at all the investigated γ-ray energies. As the particle size of the filler decreases to nanoscale, the particles will be homogenously dispersed over a lager surface area within the polymeric matrix, leading to an increase of the probability of interaction of incident photons with nanoparticles in nano composites compared with micro composites [24]. Therefore, the photon will undergo several scattering processes till its energy becomes less than 200 keV, then it will be absorbed through photoelectric effect. Thus, for the same chemical structure and weight fraction of the composite, nanoparticles show better attenuation performance than micro particles in HDPE based radiation shielding material.

CONCLUSIONS

A comparative study was performed to compare the γ-ray shielding properties of composites filled with CdO to those filled with PbO according to their sizes. It is found that CdO composite is better as a gamma absorber in the energy region below 81 keV, while PbO composite is better in the energy region above 81 keV. Also, the composites filled with a mixture of both CdO and PbO are better than PbO/HDPE composites as gamma absorbers in the energy region less than 81 keV, while the mixture composites are better than CdO/HDPE composites in the energy region greater than 81 keV. In addition, for the same chemical structure and weight fraction of the composite, nanoparticles show better attenuation performance than micro particles in HDPE based radiation shielding material. Therefore, it is suggested for future work to design a multilayer shielding material consisting of nano CdO/HDPE composite followed by nano PbO/HDPE composite to shield or to protect workers against neutrons and gamma rays.

AUTHORS' CONTRIBUTIONS

Experimental part was performed by M. T. Alabsy, M. S. Badawi, and A. M. El-Khatib. Theoretical analysis and writing were carried out by M. I. Abbas and G. A. Alharshan. The figures were arranged by M. A. Elzaher and D. A. Aloraini. The manuscript was conceived and written by all authors.

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КОМПАРАТИВНА СТУДИЈА НАНО ЧЕСТИЦА КАДМИЈУМ ОКСИДА И ОЛОВО ОКСИДА КАО ПОЈАЧАЊА У КОМПОЗИТИМА ПОЛИЕТИЛЕНА ВИСОКЕ ГУСТИНЕ ЗА ПОТРЕБЕ ЗАШТИТЕ ОД ГАМА ЗРАЧЕЊА

У овом раду применом технике компресијског обликовања припремљени су полимерни композити полиетилена високе густине побољшани микро и нано честица кадмијум оксида, олово оксида и њихове смеше са тежинским уделом од 30 %, док је карактеризација извршена помоћу електронског скенирајућег микроскопа. Ова испитивања имају за циљ приказ компаративне студије између кадмијум оксида и олово оксида о могућности њихове примене у заштити од гама зрачења у зависности од величине честице и удела у побољшању полиетилена високе густине. Масени и линеарни коефицијенти атенуације испитиваних композита измерени су као функција енергије гама зрачења у опсегу од 59.53 keV до 1408.01 keV применом стандардних тачкастих радиоактивних извора (^{241}Am, ^{133}Ba, ^{137}Cs, ^{60}Co, и ^{152}Eu). Мерења су спроведена у геометрији уског снопа користећи калибрисани цилиндрични детектор са германијумом високе чистоће. Теоријске вредности масених коефицијената слабљења одређене су применом XCOM базе података. Експериментални резултати показали су да су, у зависности од величине удела, композити кадмијум оксида бољи апсорбери у опсегу енергија испод 81 keV док су композити олово оксида бољи апсорбери у области енергија изнад 81 keV. Додатно, за исту хемијску структуру и тежински удео, побољшања са нано честицама показују боље способности слабљења него микро честице код заштитних материјала заснованих на полиетилену високе густине.

Кључне речи: линеарни коефицијент слабљења, полиетиленски композит високе густине, нано кадмијум оксид, нано олово оксид, заштита од гама зрачења