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RESEARCH ARTICLE

STUDY ON POLYMER CLAY LAYERED NANOCOMPOSITES AS SHIELDING MATERIALS FOR IONIZING RADIATION

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ABSTRACT

This work discusses the preparation of Styrene Butadiene Rubber (SBR-1502)/Montmorillonite nanocomposites in presence of some metal oxides and their suitability as shielding materials for ionizing radiations. So, the goal of this work is finding novel materials that can be used as good γ-shielding materials at minimal cost. Four Composite types of radiation shielding sheets made from a combination of styrene-butadiene rubber (SBR-1502) and montomorillonite in addition to one of the following oxides; Ferric(III) Oxide (Fe2O3), Zinc(II) Oxide (ZnO), Molybdenum(II) Oxide (MoO), and Titanium(II) Oxide (TiO), were demonstrated in this study. The microstructure of the composite samples was characterized by Scanning Electron Microscope (SEM) with Energy Dispersive X-ray (EDX). A comparison of gamma ray attenuation properties of the four studied types of shielding materials was done. The experimental results show that SBR with montmorillonite nanocomposite is better attenuating shielding for γ-ray than SBR shielding alone. The samples that treated with Molybdenum Oxide are the best attenuating shielding for γ-ray compared to the other investigated samples. The theoretical and the experimental calculations of attenuation coefficient were found to be in a good agreement. Thus, it is promising for application of polymer clay layered that treated with Molybdenum Oxide as a novel radiation protection material for γ-ray. So Montmorillonite SBR/MoO can be used in nuclear investigation centers and/or nuclear medicine departments as shielding materials at very low thickness to reduce the harmfulness and increase the economic feasibility.

INTRODUCTION

Polymeric nanocomposites are a hybrid material where inorganic substances with nanometric dimensions are dispersed in a polymeric matrix **(**Alexandre *et al,* 2000**)**. Actually, polymeric nanocomposites have attracted great industrial and scientific interest due to attainment of materials with better mechanical, barrier, thermal and flammability properties (Giannelis, 1996). These improvements can be obtained when small concentrations of the inorganic filler are added to the polymeric matrix and its layers are exfoliated and well dispersed (Wang*et al,* 2005). Modified organically clays can be efficiently exfoliated in polar polymers using adequate conditions of processing (Krishnamoorti *et al,* 2001; Vaia *et al,* 1998). Clay is a naturally occurring material, composed of primarily of fine grained minerals which show plasticity through a variable range of water content, and can be hardened when dried or fired. Clay deposits are mostly composed of clay minerals (Pinniavaia, 2000). Montmorillonite (Mt) is the most common applied clay mineral for the preparation of polymer nanocomposites, thinks to its large availability, low cost and high surface area. The main challenge for preparing nanocomposites is the nanoscale dispersion of clay in the biopolymer matrix (Pinniavaia, 2000). Sources of ionizing radiation have been in existence for technological ages. The natural sources and the artificial sources are the major sources of these radiations. These ionizing radiations have found applications in different facets of human applications which include medicine, research, industry and agriculture (Kim *et al,* 2003; Kim S.C *et al,* 2012). In all these areas of applications the use of radiation sources has been found to produce a lot of benefits. The principle that summarizes the radiation safety called ALARA which states that radiation doses to persons involved in the use of ionizing radiation must be As Low As Reasonable Achievable (International atomic energy agency, 1962). Generally the ALARA principles are ensured by (i) Minimizing the time of an individual spend around radiation sources. (ii) Staying far away from the radiation sources. (iii) By shielding the sources away from individuals and (iv) A combination of all the three approaches or any two of them. There were many research works focused on the preparation of gamma ray -shielding materials such as TiO and ZnO, as they have good gamma ray protection ability (El-Toni *et al,* 2006a). The high photocatalytic activity of TiO and ZnO facilitated the generation of reactive oxygen species, which raised safety concerns (El-Toni *et al,* 2006b). Natural materials, including clay minerals, have many benefits for human health and are utilized in various types of pharmaceutical and cosmetic

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products. Thus, they are sought after as potential candidates in gamma radiation protection (Gilman, 1999). A shield is a physical entity interposed between a source of ionizing radiation and an object to be protected which leads to reduce the radiation level at the position of that object. Lead is the most widely and effective material used as radiation shield because of its high density, large atomic number, high resistance to chemical corrosion and easy to fabricate. However lead is not common and is very expensive hence there is a limitation to its availability as radiation shield. Uranium is an excellent radiation shield material because of its high density, however, it is highly active chemically and oxidizes when expose to air in addition to being difficult to fabricate. Concrete is another versatile and widely shielding material because it is easy to fabricate and relatively inexpensive but due to its ability to lose its hydrogen content, its ability as a radiation shielding material is greatly reduced (Gwaily *et al*, 2002). Clay has been suggested as alternative material for shielding ionizing radiation (Ami *et al*, 1997). This study aims to develop an environmentally novel radiation protection composite sheet with good radiation shielding characteristics of γ-ray and has a minimal cost. Therefore, in this study, well-dispersed rubber–clay nanocomposites with four different kinds of metal oxides were prepared**.**

MATERIALS AND METHODS

Four types of radiation shielding sheets were prepared to investigate their linear attenuation coefficients for gamma rays. They were prepared by mixing Styrene-Butadiene Rubber powder-1502 (SBR) and Montmorillonite with metal oxides. SBR was commercial product and purchased from Egyptian Petroleum Company. Zinc(II) Oxide (ZnO), Molybdenum(II) Oxide (MoO), Titanium(II) Oxide (TiO), Ferric(III) Oxide (Fe_2O_3), Stearic acid, Aromatic oil, Mercaptobenzothiazole (MBTS) and 2,2-dithio-bis-benzothiazole, Tetramethyl Thiuram Disulphide (TMTD) and Sulphr. All chemical materials were obtained from the British Drug Houses (BDH Laboratory Chem. Ltd.), Poole, England. The Montmorillonite $(Na, Ca)_0,3(Al, Mg)_2Si_4O_{10}(OH)_2\bullet n(H_2O)$,was obtained from Aswan mountain region, South of Egypt and used as received, after making a representative sample by the quartering technique. The linear attenuation coefficients of the investigated samples were measured for gamma rays of energy 662 keV which have been obtained from ^{137}Cs point source. The experiment has been performed using gamma ray spectrometer which consists of 3‘‘×3’’ NaI(Tl) Scintillation detector, amplifier and 16k multi channel analyzer (Egyptian Nuclear and Radiological Regulatory Authority). For each sample, the gamma ray spectrum was recorded as a function of the thickness of the material. And the area under the photo peak of the spectrum is used to evaluate the intensity (I) of the transmitted beam by using the initial intensity (I_o) which is the area under the photo peak obtained without any sample between detector and source.

Determination of elements and chemical components in montmorillonite

100g of montmorillonite sample was accurately weighed, put in a 2-way 500-ml round bottomed flask, 200 ml conc H_2SO_4 was added and the mixture heated for 2h using an isomanitle. The mixture was cooled, filtered using a pre-weighed sintered glass crucible of porosity No. 3, the residue thoroughly washed with distilled water, the washings added to the filtrate and the solution made up to 100 ml using distilled water. This solution was examined for various elements following standard analytical procedures (Medham *et al*, 2000)**.** The acid-insoluble residue was dried at 130°C for 3h in an oven, cooled in a desiccators, weighed and analyzed for various elements using XRFS(solid). These measurements were conducted in the labs of the National Research Center, Egypt.

Nanocomposites Preparation

Initially the nano montmorillonite clay $(Na,Ca)_0$, $3(Al, Mg)_2Si_4O_{10}(OH)_2\bullet n(H_2O)$ was prepared by solid mixing using an agate pestle and mortar to obtain nano montmorillonite material. Then added distilled water with mass ratio of 30% H_2O:70% Montmorillonite. Then 5 mL of ammonia solution was also added during the synthesis process to enhance the precipitation of the nano-montmorillonite **(**Xie W. *et al*, 2002**).** Secondly the shield of metallic oxide with montmorillonite and Styrene-Butadiene Rubber (SBR) was prepared as following: 2 phr stearic acid was added to 32 phr of SBR and mixed for 5 min, after that 10 phr aromatic oil was add and mixed for 2 min to produce a mixture. The nano-montmorillonite clay was added into this mixture with ratio 50phr. Mercaptobenzothiazole disulfide (MBTS), Tetramethyl Thiuram.

Disulphide (TMTD) and sulphur were subsequently added and mixed for 2 min before discharge. The metallic oxide (Fe_2O_3, ZnO, MoO or TiO) was added with ratio 50 phr from the total previous mixture and mixed for 2 min, so four composite samples were produced in addition to a control sample. The rubber compound was then compression-moulded for 30 min under a pressure of 40 kg/cm^2 by using a hot-platen hydraulic in order to give vulcanized rubber sheet using a cure temperature of 150^{o}C (Gilman *et al*, 1999 ; Malden *et al*, 1999)**.** The control sample has all previous chemical compounds except metallic oxides**.** All samples were cut into thickness degrees at (0.05, 0.1, 0.15 and 0.2 cm)**.** Table (1) shows the compound formulation details of all samples.

Table 1 Materials and chemical additives in nano montmorillonite -SBR composite

Ingredients (phr)	**Control (clay+ SBR)**	**Clay + SBR + Fe_2O_3**	**Clay + SBR + ZnO**	**Clay + SBR + MoO**	**Clay + SBR + TiO**
SBR	32	32	32	32	32
Stearic acid	2	2	2	2	2
Ormatic oil	10	10	10	10	10
MBTS	2	2	2	2	2
TMTD	1	1	1	1	1
S	3	3	3	3	3
Montmorillonite	50	50	50	50	50
Fe_2O_3	-	50	-	-	-
ZnO	-	-	50	-	-
MoO	-	-	-	50	-
TiO	-	-	-	-	50

- phr: parts per hundred parts

Examine Montmorillonite – SBR polymer matrix Scanning by Electron Microscopy (SEM) with Energy Dispersive X-ray scattering (EDX)

Scanning Electron Microscope with Energy Dispersive X-ray scattering SEM/EDX, model EVO 60, with 10KV accelerating voltage, (central lab – ENNRA- Egypt) was used to examine the SBR/Montmorillonite microstructure samples and were subjected to EDX analysis in order to examine chemical composition as well as level of chemical homogeneity between these components.

Transmission experimental detail

The block diagram of transmission experiment set up is shown in figure 1. The source and absorber system were mounted on composite of adjustable stands. With the help of a screw arrangement the platform having material was also made capable of movement in the transverse direction to the incident beam for proper alignment. The ^{137}Cs radioactive source of 5µCi strength was used. The incident and transmitted gamma-rays intensities were determined for a fixed preset time in each experiment by recording the corresponding counts, using the 3″×3″NaI(Tl) detector (oxford model) at Egyptian Nuclear and Radiological Regulatory Authority. The NaI(Tl) having an energy resolution of 10.2% at 662 keV, with PCA3 program.

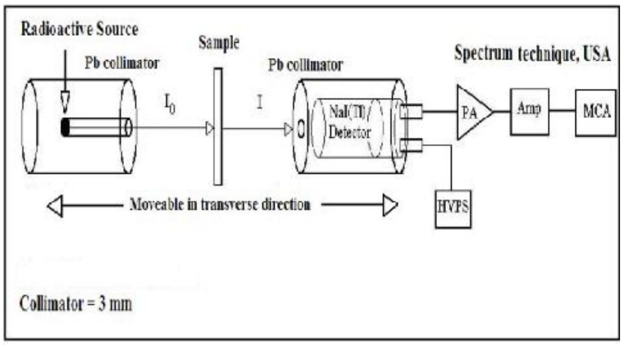


Fig. 1 transmission experiment set up

Shielding calculations

The linear attenuation coefficients (µ) can be extracted with the thicknesses of the material by the standard equation:

$$I = I_o e^{-\mu x} \text{..........} \quad \text{..........} \quad \text{.....}(1)$$

Plotting ln(I$_o$/I) versus x would give straight line and µ can be obtained from the value of the slope. The correlation between the linear attenuation coefficients and the thickness of the composite is used to confirm the linearity.

The mass attenuation coefficient (µ$_m$) is written as following:

$$\mu_m = \frac{\ln(I_o/I)}{\rho x} \text{..........} \quad \text{.........}(2)$$

where: *I* is the measured attenuated gamma ray intensity,

I$_o$ is measured initial intensity (no studying shield),

µ is attenuation coefficient factor,

ρ is the density of material (g/cm^3)

x is the thickness of absorber (cm).

Theoretical values of the mass attenuation coefficients of the investigated samples have been calculated by WinXCom, based on mixture rule (Berger *et al*, 1987).

$$\mu_m = \sum_i w_i (\mu_m)_i \text{..........} \quad \text{........}(3)$$

Where: w_i is the weight fraction of element in a mixture,

$(\mu_m)_i$ is the mass attenuation coefficient for individual element in a mixture.

For attenuated photons in absorbing medium, the mean free path (mfp) is the distance over which the primary photon intensity is reduced by factor *1/e.*

mfp = (1/µ) ………………(4)

The simplest method for determining the effectiveness of the shielding material is using the concepts of half-value layers (HVL) and tenth-value layers (TVL). One half-value layer is defined as the amount of shielding material required to reduce the radiation intensity to one-half of the unshielded value.

HVL=(ln2/µ)= (0.693/µ) ……….(5)

The symbol µ is known as the linear attenuation coefficient and it is obtained from standard tables for various shielding materials.

One tenth-value layer is defined as the amount of shielding material required to reduce the radiation intensity to one-tenth of the unshielded value.

TVL= (ln10/µ)= (2.3026/µ) …….(6)

Both of these concepts are dependent on the energy of the photon radiation and a chart can be constructed to show the HVL and TVL values for photon energies.

RESULTS AND DISCUSSION

Composition of Montmorillonite

The chemical analyses of the studied montmorillonite clay sample are shown in Table (2). It is clear that montmorillonite clay sample has multiple heavy metals oxides with different ratios. SiO_2 represents the higher ratio in this structure and equals 39.892%. These varieties in types of metal oxides lead montmorillonite clay able to form strong chemical bonds with any other different chemical compounds so montmorillonite clay characterized by its expandable properties. Consequently, these metal oxides are support the structure of shielding materials against radiation. This may be due to their high ionic charge (Juang *et al*, 2002).

Transmission of γ-ray through the studied shielding materials

The shielding properties of the studied samples have been investigated in the field of gamma emitted by ^{137}Cs (energy 662

Table 2 Chemical analyses of montmorillonite clay sample

Metal oxide	Concentration (%)
SiO_2	39.892
CaO	18.067
MgO	13.290
Al_2O_3	10.270
PbO	0.018
K_2O	0.018
B_2O_3	4.705
SO_3	3.500
SrO	0.839
Fe_2O_3	0.828
TiO_2	5.654
As_2O_3	0.579
P_2O_5	0.383
Na_2O	0.162
MnO_2	0.132
BaO	0.095
Y_2O_3	0.093
ZnO	0.092
Nb_2O_5	0.034
Cr_2O_3	0.031
NiO	0.018
Total	98.7

keV). The obtained results are illustrated in table (3) and figures (2-7). Generally, it is clear that the transmission of γ-ray through the investigated composite samples decrease as the sheet thickness increase. And the transmission of γ-ray decrease by adding metal oxide to the composite samples than that of the control sample, as well as the sample which treated with MoO has the lowest transmission of γ-ray. In case of clay with SBR and TiO sample, the calculated transmission of γ-ray are 0.07846, 0.06799, 0.05636and 0.04531with sheet thickness of 0.05, 0.10, 0.15 and 0.20cm, respectively. This means that TiO additive increase the ability of composite to reduce the transmission of γ-ray and also the attenuation increases as the thickness increase. By investigation of SEM- EDX images in figures (3-7), the micrographs show highly agglomerated globular particles nano montmorillonite clay-SBR polymer with metal oxide molecules. SEM- EDX images reveal that the transmission of γ-ray through the investigated composite samples depends on a variety of metal oxides and exchange capacity of the clay with polarity of metal oxide molecules of the reaction medium. By investigation of SEM- EDX image in figure 3, the micrograph shows good agglomerated globular particles nano montmorillonite clay-SBR polymer with TiO molecules, i.e. SEM- EDX reveals that TiO support modified clay to adsorb or reflects γ-ray.

Table 3 Transmission of γ-ray through the studied shielding materials at different sheet thickness for ^{137}Cs γ-rays

Properties		Samples									
		Control (Clay +SBR)		Clay+ SBR+ TiO_2		Clay + SBR + Fe_2O_3		Clay + SBR + ZnO		Clay + SBR+ MoO	
Density (g/cm^2)		0.8		1.2		1.6		1.3		2.2	
Intensity of γ-ray (I_o= 9968563)		I	I/Io	I	I/Io	I	I/Io	I	I/Io	I	I/Io
Thickness	0.05cm	6366096	0.63862	782201	0.07846	672035	0.06741	468521	0.04699	255689	0.02565
	0.1cm	6080801	0.60999	677860	0.06799	588143	0.05899	368835	0.037	183158	0.01837
	0.15cm	5452621	0.54698	561865	0.05636	488462	0.04900	271722	0.02725	135613	0.01360
	0.2cm	4884595	0.48999	451697	0.04531	371783	0.03729	205273	0.02059	105354	0.01057

These results well agreement with that reported by Wilson (Wilson *et al*, 1991) which concluded that γ-ray contract when TiO value increase, where TiO enjoy with some properties such as absorption and scattering γ-ray and its interaction with materials leads to increase of composite materials to contract γ-ray transmission. Rubber gloves filled with TiO powder are used to insure a good protection to operators exposed to ionizing radiation in hospitals (Nita *et al*, 2005**).** In case of clay with SBR and MoO sample, the calculated transmission of γ-ray are 0.02565, 0.01837, 0.01360 and 0.01057 with sheet thickness of 0.05, 0.10, 0.15 and 0.20cm, respectively. This means that MoO additive increase the ability of composite to reduce the transmission of γ-ray and also the transmission decreases as the thickness increase, i.e. nano Montmorillonite-SBR /MoO has highly efficiency to absorb or reflect γ-ray. SEM image and EDX analysis of figure7 reveals the microstructure of nano Montmorillonite-SBR /MoO. It is observed that there are highly agglomerated globular particles of nano montmorillonite polymer, white particles, are distributed in the boundary of MoO molecules. Moreover, the morphology shows a homogeneous surface. Also, sheets of ZnO and or Fe_2O_3/ nano montmorillonite are able to absorb or reflect γ-ray, but they are lower than MoO/ nano montmorillonite sheet. Gilman reported that clays, such as bentonite, showed potential for γ-ray protection through the absorption and/or reflection of gamma radiation. Furthermore, several parameters, including grain size distribution and chemical composition, could play an important role in determining the γ-ray protection ability of these materials (Gilman *et al*, 1998).

Shielding material properties

In this study both experimental and theoretical calculations for effective penetration of γ-ray at nano montmorillonite/SBR and metal oxides shielding materials have been calculated. Some shielding material properties of the investigated samples have been illustrated in table (4). It is bright that the nano montmorillonite/SBR sample which treated with MoO has highest linear attenuation coefficient, 0.067cm, and the lowest one is the nano montmorillonite/SBR sample which treated with TiO, 0.031cm. Nano montmorillonite/SBR with MoO polymer sheet has the lowest values of HVL and TVL 10.34cm and 34.36cm, respectively. Also, the values of μ and mfp of nano montmorillonite/SBR with MoO are 0.067 and 14.93 respectively. While Nano montmorillonite/SBR with TiO polymer sheet has the highest values of HVL and TVL 22.35cm and 74.28cm, respectively. And their values of μ and mfp are 0.031and 32.26, respectively. As shown in table (4), the experimental mass attenuation coefficient (Exp.μ_m) and the theoretical mass attenuation coefficient (Tho.μ_m) are calculated for the studied samples. The values of Tho.μ_m of the samples were obtained using win XCOM computer program (Berger, *et al*, 1987). The theoretical calculations of mass attenuation aim to investigate the suitable type and thickness of these γ-

Table 4 Attenuation properties of the investigated shielding composite samples at sheet thickness equals 0.2cm and energy of ^{137}Cs γ-rays

composite samples	Properties					
	μ(cm)	Exp.μ_m (cm^2/g)	Tho.μ_m (cm^2/g)	mfp (cm^{-1})	HVL (cm^{-1})	TVL (cm^{-1})
Control	0.018	0.0020	0.0021	55.56	38.50	127.92
caly+SBR+ TiO	0.031	0.0258	0.0252	32.26	22.35	74.27
caly+SBR+ Fe_2O_3	0.042	0.0262	0.0270	23.81	16.50	54.82
caly+SBR+ ZnO	0.037	0.0284	0.0283	27.03	18.73	62.23
caly+SBR+ MoO	0.067	0.0304	0.0310	14.93	10.34	34.36

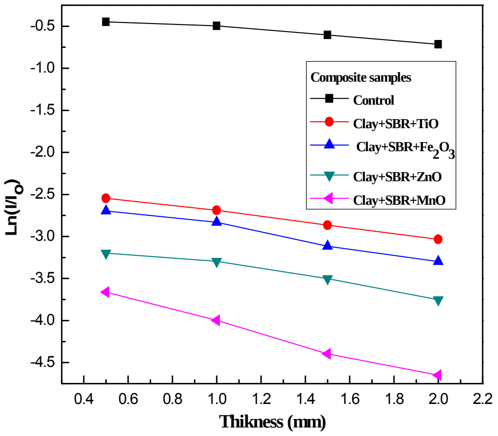


Fig. (4): The transmission of gamma ray through the investigated samples as function of their sheet thikness; E=662KeV.

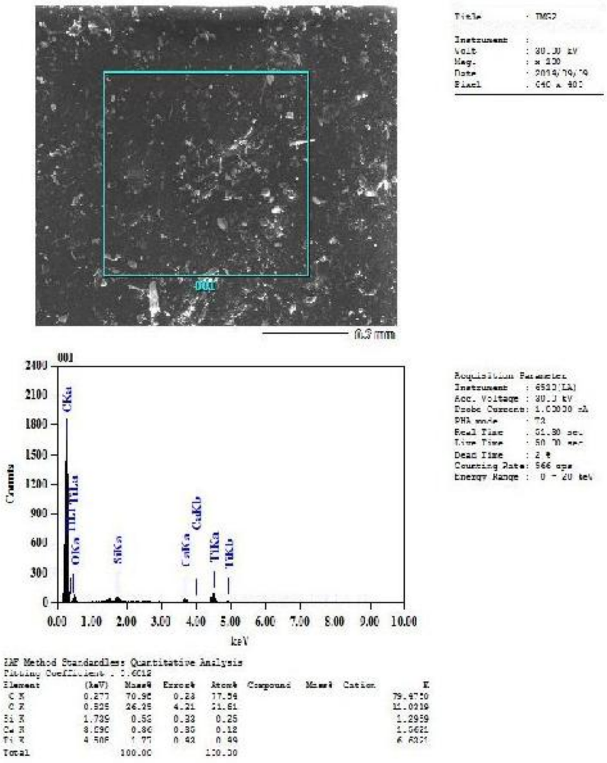
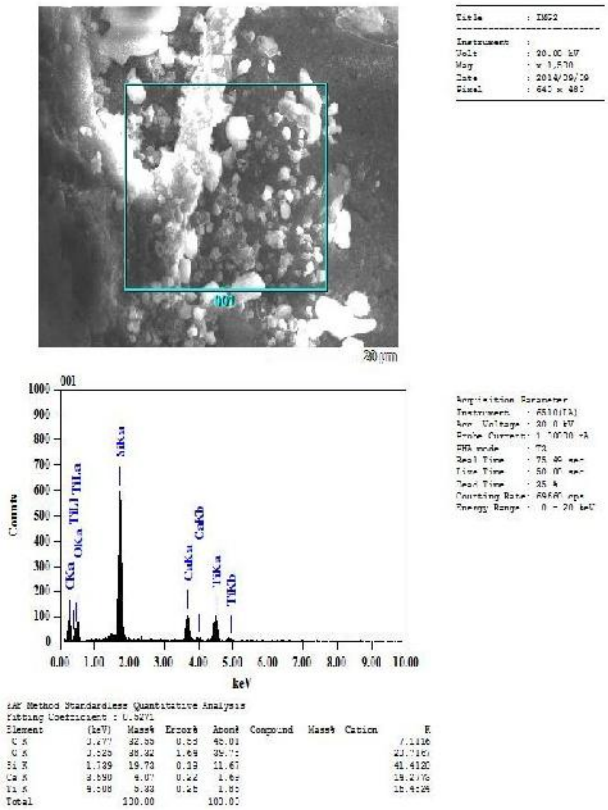


Fig 3 SEM with EDX for the control shielding material (nano montmorillonite /SBR).



Fig(4) SEM with EDX for shielding material of nano montmorillonite/SBR treated with TiO

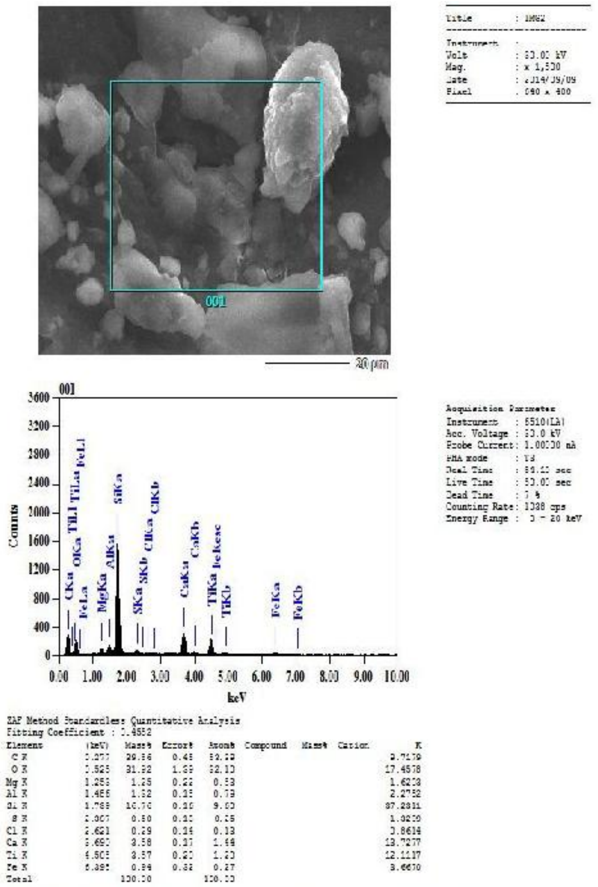


Fig 5 SEM with EDX for shielding material of nano montmorillonite/SBR treated with Fe_2O_3.

shielding material, which can be used to protect the people against direct and scattered γ-radiation (Nabil *et al,* 2009). It is clear that the Exp.μ_m and Tho.μ_m values are synergistic in all samples. The Exp.μ_m and Tho.μ_m of different composite samples have the following order: nano montmorillonite/SBR with MoO > nano montmorillonite/SBR with ZnO> nano montmorillonite/SBR with Fe_2O_3> nano montmorillonite/SBR with TiO; i.e. there is a clear improvement in the γ- attenuation coefficient in the case of molybdenum oxide than other investigated metallic oxides and this improved may be attributed to combined matrix materials nano montmorillonite/ SBR with MoO molecules. These results are agreement with results of Berger where developed Fortran program to determine the effectiveness of some γ- shielding materials such as lead, lead-rubber composites, lead-glass and rubber (Berger *et al* ,1987). The calculations of mean free path (mfp) of the investigated composite samples show that nano montmorillonite/SBR with MoO has the lowest mfp value and they have the following order: nano montmorillonite/SBR with TiO > nano montmorillonite/SBR with Fe_2O_3> nano montmorillonite/SBR with ZnO>; nano montmorillonite/SBR with MoO,i.e. 32.26> 23.81> 27.03> 14.93.

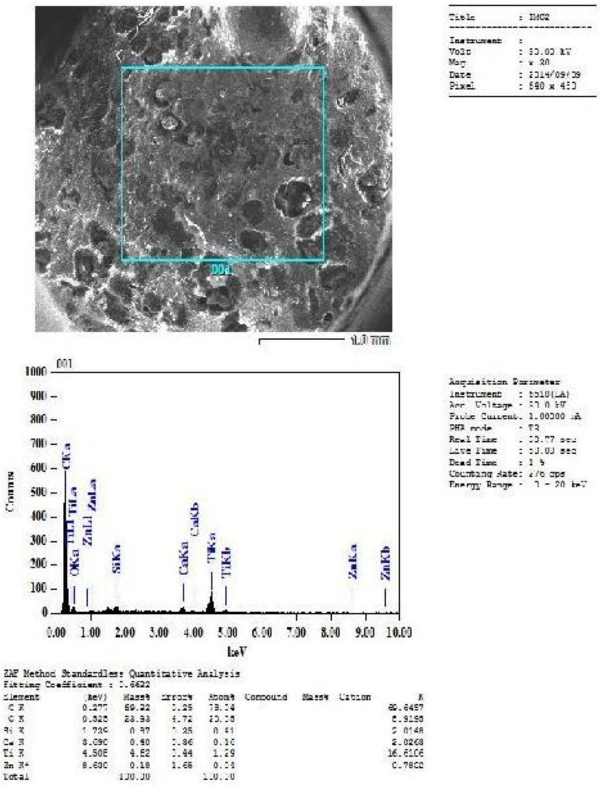


Fig 6 SEM with EDX for shielding material of nano montmorillonite/SBR treated with ZnO

The concepts of half-value layers (HVL) and tenth-value layers (TVL) are the simplest methods for determining the effectiveness of the shielding materials. One half-value layer is defined as the amount of shielding material required to reduce the radiation intensity to one-half of the unshielded value. One tenth-value layer is defined as the amount of shielding material required to reduce the radiation intensity to one-tenth of the unshielded value. A comparison between the calculated values of HVL and TVL for the investigated composite samples at the same energy of the photon radiation and the same cheat thickness are illustrated in table 4. It is clear that, nano montmorillonite/SBR with MoO, the shielding composite required reducing the radiation intensity to one-half of the unshielded value equals 10.34cm and the tenth-value layer equals 34.36cm.

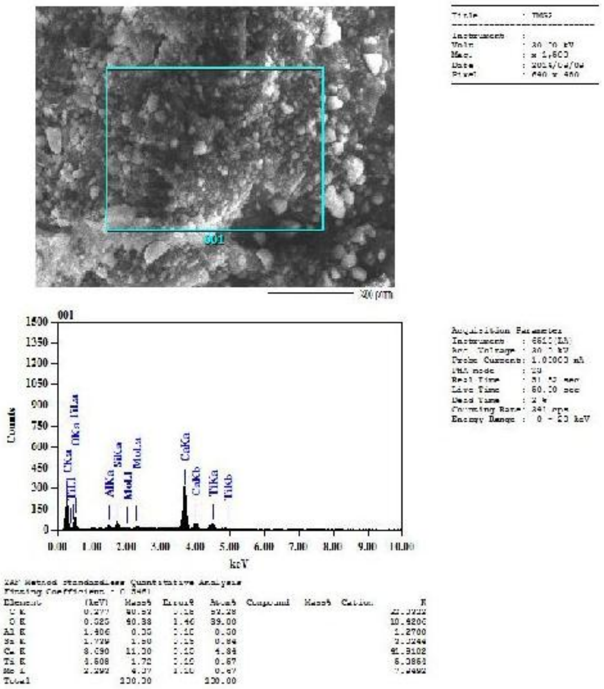


Fig 7 SEM with EDX for shielding material of nano montmorillonite/SBR treated with MoO

CONCLUSION

The experimental results had allowed the following conclusions:

The montmorillonite nanocomposites SBR polymer samples which treated with metallic oxides will open new possibility for γ-radiation protection. The sample that treated with MoO has the best attenuating shielding purposes for gamma ray than the other investigated samples.

The authors have successfully developed shielding material from the nanostructured-clay nanocomposite SBR polymer combined with metallic oxides, which was prepared from low cost materials.

The total mass attenuation coefficients of the investigated composites have the order: μ of composite with MoO > μ of composite with ZnO > μ of composite with Fe_2O_3> μ of composite with TiO.

The results of SEM-EDX analysis have provided new information about the total macroscopic cross sections, and distribution of metallic oxides in different sample sheets.

Montmorillonite SBR/MoO has potential for γ-ray protection through the absorption and/or reflection of gamma radiation. So these materials can be used in nuclear investigation centers and/or nuclear medicine departments as shielding materials at very low thickness to reduce the harmfulness and increase the economic feasibility.

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