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Characterization of Some Synthesis Irradiated and Non-Irradiated Sorbent Materials

Omar S. Khalifa[1], Hassan H. Abbas[2], Magdy A. Rizk[1], Mostafa A. Zahran[1], Mohamed A. AbdelSalam[2], Maha M. Ali[2], Ibrahim A. Mousa[1] and Mamdouh F. Abdelsabour[1*]

(1) Soil and water researches Department, Nuclear Researches Center, Atomic Energy Authority, Egypt
(2) Soil and Water Department., Faculty of Agriculture, Benha University, Egypt

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ABSTRACT

Different synthesized materials either irradiated or non-irradiated were examined in the laboratory to identify its characteristics as efficient absorbers in removing Pb, Cd, and Cu from contaminated water. Bio-based polymers are gaining overwhelming interest and recognition worldwide due to the health, safety, and environmental concerns associated with the conventional synthetic polymers. These bio-based polymers are renewable, biodegradable, and environmentally-friendly. The characterization analysis confirmed the successful synthesis of the investigated materials. The processing of rice straw was effective in removing silica and hemicellulose and leaving microcrystalline cellulose with an accepted level of purification. Vulcanized used fried oil was efficient to produce polysulphide polymer. Results gained from this laboratory work indicated that irradiated sorbents were more effective on the sorption of Pb, Cd and Cu especially those exposed to lower doses compared to the non-irradiated sorbents. At a high gamma ray dose (50 KGy), some sorbents showed inhabitation effect on sorption behavior and also removal efficiencies. It can be concluded that rice straw and fried oil can be used to synthesize suitable sorbents to remove contaminants from the water, and gamma radiation treatment was effective to improve contaminants removal.

INTRODUCTION

Fresh water scarcity in addition to simultaneous increase in the amount of wastewater is an alarming issue. The gap between demand and availability of water is accounted for about 13.5 Bm^3/yr. Recycling and Reuse of treated wastewater in the agriculture sector can reduce this gap and minimize the dependent on freshwater resources [1-2].

Potentially toxic elements (PTEs) abundant in industrial discharged water, pesticides and nutrient elements (N, P, K, and others) are found in agricultural drainage water [3-5]. Tested water samples collected from greater Cairo water bodies (near to industrial zones including Shoubra El-Khima and Mostorod) had a high level of PTEs, especially Pb, Cd, Cu, and Ni [6]. Long-term irrigated soils with domestic and industrial wastewater in Elgabal Elasfar area recorded a high accumulation of PTEs such as Pb and Cu [1].

Biomass and biomaterials from agricultural byproducts are being widely used for adsorption with or without modification. Their natural adsorption abilities without modification can sometimes be quite efficient. New bio-sorbents are being studied which would offer metal recovery, low costs, and minimum requirements of secondary waste treatment arising from the adsorption process [7]. In this respect, agricultural wastes are promising materials for the removal of PTEs from their aqueous solutions [8].

Searching for sustainable materials that can decontaminate PTEs from wastewater became a necessity. In this regard, some researchers reported that cellulose can be modified physically and chemically to form different materials, which can be used for waste treatment. Cellulose-based materials have natural binding capacity for metal ions [9-10]. Cellulose is a crystallization material with an abundance of hydroxyl groups [11]. The rice husk could be used for the adsorption of lead and copper ions as reported by Vieira et al. [12] while other researchers [13] used rice husk and rice straw for the adsorption of Hg(II) after treatment with NaOH. In a comparative study [14], two cellulose based materials, rice hull and sawdust, were used to adsorb heavy metals including Pb(II), Cu(II), Cd(II),

Corresponding author: wise2007egy@yahoo.com
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Zn(II), and Ni(II) from artificial solutions and wastewater samples. The results showed that the alkali treatment to cellulosic materials have the highest adsorption capacity.

Microcrystalline cellulose (MCC), as described in a previous study [15] is purified white cellulose that can be produced from different cellulosic sources by different techniques such as acid hydrolysis. MCC has unique colloidal properties that assist its usage in many industrial applications. The polymerization degree of MCC is less than 400 and about 10 % of MCC has a particle size of less than 5 µm. Microcrystalline cellulose usage for water and wastewater treatment was previously discussed, for example, Garba et al. [16] investigated the use of rice straw as an agricultural waste to synthesize microcrystalline cellulose as a cheap adsorbent for removing Pb, Cd, and Cu from contaminated waters.

Unsaturated oils from rapeseed, sunflower, canola, and olive plants are attractive as chemical building blocks because they are renewable and can be produced on all inhabited continents [17]. The alkene functional groups in these triglycerides also provide the requisite points for cross-linking during inverse vulcanisation. Vegetable oils used in professional fryer machines are heated several times in temperatures ranging from 90°C to 175°C. Prolonged oil heating in the presence of moisture, salts, and air can result in oil degradation and alteration of food quality [18]. The management of waste oils and fats pose a significant challenge because of problems related to their disposal and their possible contamination of water and land resources. The recovery of waste cooking oil for new uses and applications after suitable physico-chemical treatments is a challenging goal in relation to economic and environmental issues [19]. The polymers derived from oils , with and without spongy shape, were used for potentially toxic elements removal from aqueous solutions. The removal of iron using spongy sulfur-canola polysulfide polymer at 50 mg Fe L^{-1} was studied by Lundquist et al. [20]. Removal of iron using sulfur canola polymer reached 80% and 95% for non-spongy and spongy sulfur-canola polymer, respectively.

Spongy polysulfide polymer (also named low-density polymer) was prepared from sustainable fatty acid sources by Worthington et al. [21]. The hydrophobic spongy polymer could be prepare by a reverse vulcanization reaction between oils (such as sunflower oil, canola oil, lemonine, oil spill, and sulfur).

High energies particularly gamma-rays (1.17 and 1.33 MeV from ^{60}Co) can be used to initiate free radical based reactions in solids, liquids or gases. This method of synthesis represents a clean alternative to chemical methods and it has shown a huge potential in morphological control and particle size by means of parameters that include absorbed dose, dose rate, stabilizing agents, and concentration of metal ion, polymer/monomer, and protein precursors [22].

Degradation of rice straw and sugarcane bagasse was increased as affected by a gamma irradiation dose of 30 keV for 2 hrs[23]. Similar results were reported for cellulose [24] and carboxy methyl cellulose (CMC) [25].

This conduced work aims at: A) Synthesis of certain materials from agricultural processing of wastes and their composites, and identification of its main physical and chemical properties, B) Evaluating the sorption efficiency of certain PTEs i.e. lead, cadmium and copper using the adsorbents previously prepared from the agricultural wastes C) Follow up of the impact of irradiation doses on the removal capacities of chemically synthesized materials.

MATERIALS AND METHODS

Experimental trials were carried out in the laboratory of Soil and Water Research Department, Nuclear Research Center, Atomic Energy Authority, Egypt.

Chemical synthesis of absorbent materials from wastes

A) Rice straw

Rice (*Oryza sativa*) straw was washed with tab water twice to remove dirt and once with distilled water, then dried at 70°C for 24h, and preserved in a cool dry place. Dried rice straw (RS) have 70.1α-Cellulose, 1.34 Lignin, 17.8 Hemicelluloses, 13.8 Ash and 72.1% Silica in ash,

B) Used frying oil

Used frying sunflower oil collected from the local market (used for frying potato several times as a common practice in Egypt). This oil was purified with a sieve for removing any particles.

Microcrystalline Cellulose (MCC) synthesis

20 g of washed and dried rice straw (RS) was added to 1 L of 1% H_2SO_4 and boiled for 45 minutes. Then the straw separated and washed with abundant water till pH turned to neutral and the filtrate disposed of. To remove silica and lignin, the acid-treated RS from the last step was added to 1L of 1.5*M* of NaOH and 5% H_2O_2 and boiled for 30 minutes, then filtered and washed with abundant pure water.

Because of H_2O_2 addition, the filtrate color turned yellow, while the alkali treatment without an oxidation agent (H_2O_2) always produces Black liquor and the fibers produced with H_2O_2 addition was a lighter color than those produced without H_2O_2. To bleach fiber for the synthesis of white MCC, fibers derived from alkali

treatment were added to 0.5L of 0.5% NaClO and boiled for 30 minutes, filtrate and washed with abundant water, dried in air and ambient laboratory temperature for two or three days depending on ambient temperature and moisture in MCC, then ground with a household grinder. This preparation method was carried out according to Abbas [26].

Porous Polysulfide Polymer synthesis

This preparation was made according to a previous study [21]. In brief, 100 g of sulfur was mixed well with 100 g used frying oil and heated to 180° C then mixed well at this temperature for 20 minutes. This step produced a polysulfide polymer. Then, to make a spongy polymer, 700 g of ground NaCl was added to polysulfide polymer and mixed with more power because of increasing viscosity. After five minutes of mixing, this mixture was let to cool at room temperature for hours. This mixture was ground using a household grinder, and then washed with abundant water to remove salt granules. The dissolved salt from polymer let holes of these positions free, which make like Sponge Poly Sulfide Polymer. This porous polymer was dried and kept. The produced material will be known as Spongy PolySulfide Polymer (SPS).

Irradiated sorbent materials

To determine the effect of gamma rays on these materials, 1g of MCC and SPS was exposed to gamma irradiation at 1, 5, 10, and 50 k Gy. This samples has the following abbreviation: M-0, M-5, M-0 and M-50 for MCC irradiated with 0, 5, 10 and 50 KGy and S-0, S-5, S-10 and S-50 for SPS irradiated at 0, 5, 10 and 50 KGy.

Sorption experiment

50 ml of **Pb, Cd, and Cu at different** concentrations i.e. 0, 10, 20, 30, 50, 70, 100, 150 and 200 mg L^{-1} were added to 0.05 g portion of MCC, and SPS. Then agitated under; a constant equilibrium time (180 min), at constant temperature (298 K) and at constant pH (7); centrifuged at 7000 rpm, and then, the supernatants were used for determining the remaining element concentration and consequently the adsorbed one was included. The concentrations of Pb, Cd, and Cu were measured using a GBC, Type 902 atomic absorption spectrophotometer (AAS). The considered pH values of the solutions were adjusted using a dilute solution of either NaOH or HCl of 0.01*M*.

Calculation

In all experimental runs, the adsorbed amounts calculated as follows:

$$\text{Removal percentage (R\%)}=\frac{(C0-Ce)}{C0}*100 \quad \ldots.\textbf{(1)}$$

Where, C_0 and C_e are initial and equilibrium concentrations, respectively.

$$\text{Sorbent amount (mg g}^{-1}\text{) } q_e = (C_0-C_e)\frac{V}{W}\ldots \quad \ldots(\textbf{2})$$

Where, q_e is the amount of element sorbed by a unit mass of an adsorbent (mg g^{-1}), W is the weight (g) of the adsorbent and V is the volume of the equilibrium solution, and C_0 and C_e are the concentrations (mg L^{-1}) of each ion before and after adsorption, respectively.

Effect of the Irradiation dose on the removal of contaminants

A 0.05 g of irradiated synthesized sorbents materials, M-5, M-10, M-50; S-5, S-10 and S-50 were equilibrated with 50 mL of **Pb, Cd, and Cu** ions at fixed concentration of 20 mg L^{-1}, agitated for a constant equilibrium time (180 min), a constant temperature (298 K) and a constant pH (7), centrifuged at 7000 rpm and then, the supernatant were used for determining the considered studied metal ions.

Characterization of the synthesized sorbent materials

The evaluated sorbents used in this study were characterized by various techniques as following:

1) Forier Transmation infra-red (FT-IR) for recognizing functional groups,
2) X -ray diffraction (XRD) for degree of crystallization,
3) Scanning electron microscope (SEM) for the morphology of their surface, and,
4) Energy Dispersive X-ray (EDX) for the percentages of the elementary composition of these substances

RESULTS AND DISCUSSION

Characterization of the non-irradiated sorbent derived from agricultural wastes

The FT-IR spectrum for the MCC and SPS is graphically illustrated in Figure (1). The FT-IR spectrum of the MCC materials was identified the presence of a number of surface features such as the 1055 cm^{-1} wavelength peak for hydroxyl groups. The peak at 1729 cm^{-1} refers to the presence of carboxylic groups and 3352 cm^{-1} that constitute phenol vibration. These functional groups may be active in ionic contaminant sorption, including PTEs.

FT-IR spectrum of the SPS materials has identified the presence of several surface functionality such as: 1) the peak wave number 672 and 1454 cm^{-1} for aromatic compounds 2) 1741 and 2865 cm^{-1} for aldehydes 3) the peaks of frequencies 1161, 2923, and 3705 cm^{-1} for different hydroxyl groups. The peak of 3705 cm^{-1} represents phenol vibration. These functional groups can help in the sorption of contaminant ions, including PTEs and organic substances.

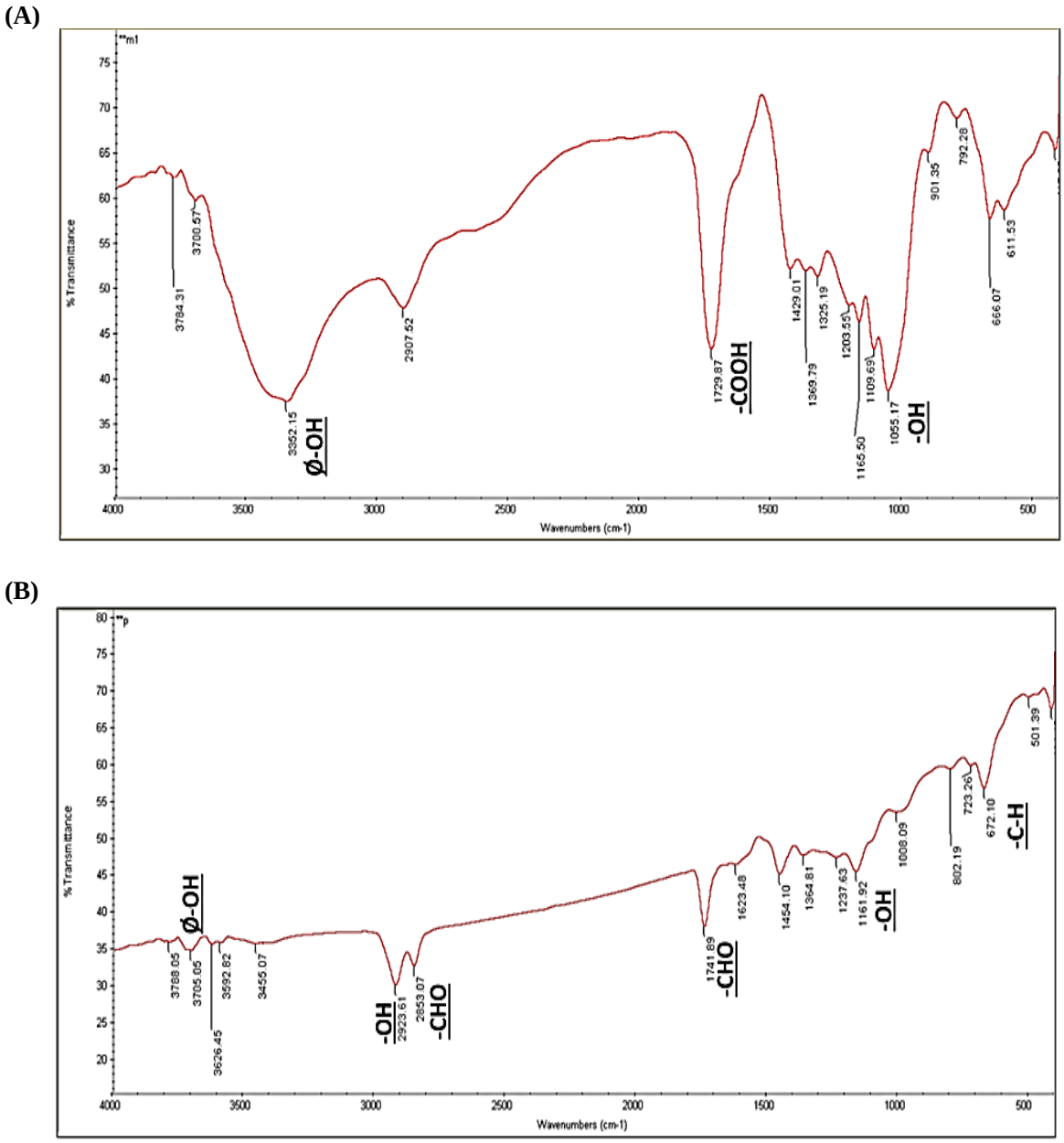


Fig. (1): FT-IR characterization of (A) :MCC and (B): SPS for functional groups

The crystalline type of cellulose and its crystallinity degree can be determined from the interpretation of the XRD diffraction patterns of MCC and SPS shown in Figure (2). The XRD pattern of MCC denotes the amorphous nature of the material, which has no crystallinity. The characteristic peaks which occur around 16.1º and 22.4º are ascribed to typical cellulose I crystal lattice structure. A narrower and more intense peak was shown at 22.4º in the diffraction pattern of MCC indicating that the removal of the hemicellulose and lignin through chemical treatments had caused a significant improvement in the crystallinity index of MCC. Additionally, this phenomenon implies that MCC was able to form a better and more organized structure at the crystalline interfaces.

The XRD pattern of SPS indicates identification peaks of polysulfide found at 22.9º, 27.6º, and 15.4º. This data are nearly closed to those published by Kosa and Hegazy [27] for cobalt sulfide polymer and that by Lundquist et al. [20] for canola polysulfide polymer.

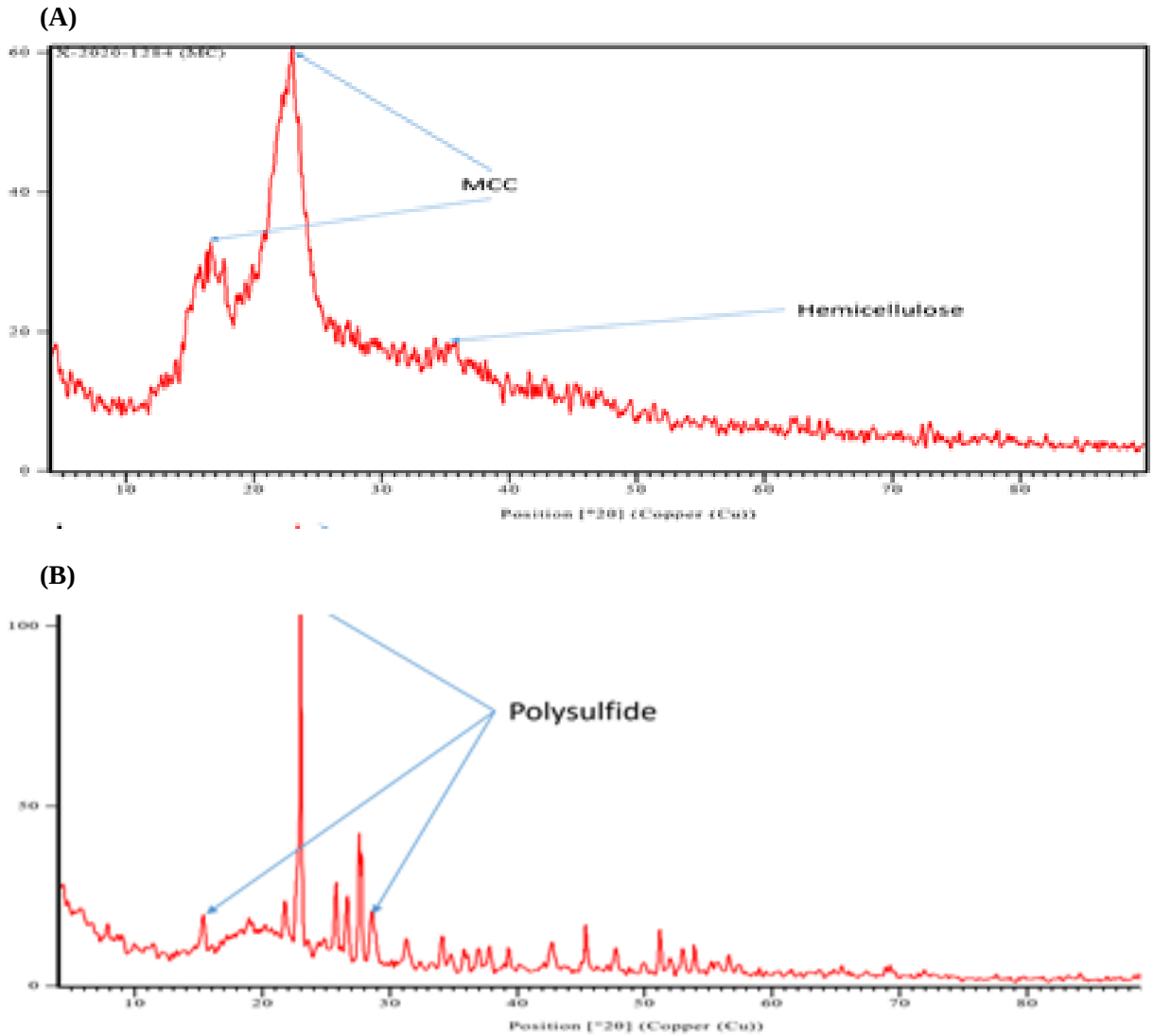


Fig. (2): XRD characterization (degree of crystallization) of (A) MCC and (B) SPS

The results of the EDX analysis for MCC and SPS graphically illustrated in Figure (3. Carbon and oxygen were the primary components, but silica and calcium were at lower levels. These data was similar to that presented by chin et al. [28] who investigated extraction of MCC from RS. In addition, Kunusa et al.[29] reported similar results when studying the extraction of MCC from corn cobs with 6% NaOH alkaline treatment.

The results of the EDX analysis for SPS, showed that carbon, oxygen and sulfur are the primary component, which confirmed the success of the SPS preparation method.

SEM images of the synthesized MCC and SPS are shown in Figure (4). It can be seen that the morphology of the untreated rice straw fiber was contrastive with the morphology of the MCC. This is due to the success of the removal of amorphous hemicellulose and lignin, which enveloped the untreated rice straw fiber through chemical treatments, leaving only cellulose behind.

It can be seen that the morphology of the SPS showed a cavity and many tiny holes, which make a polysulfide such as a sponge. In addition, SEM image showed that many sizes of SPS formed due to the method of synthesis. These results are similar to those found by [20-21] who illustrated a near image for SPS prepared with the same method using unsaturated cooking oils (canola oil).

Characterization of the irradiated sorbents

Sorption and removal data concluded that the gamma irradiation dose of 10 KGy was most effective in improving sorption and elimination behavior. Figure (5) shows FT-IR analysis of sorbent composites M-10 and S-10, respectively. Figures (6) shows XRD analysis of sorbent composites M-10 and S-10, respectively. These Figures show that there were some shifts toward the peaks, which referred to that irradiation.

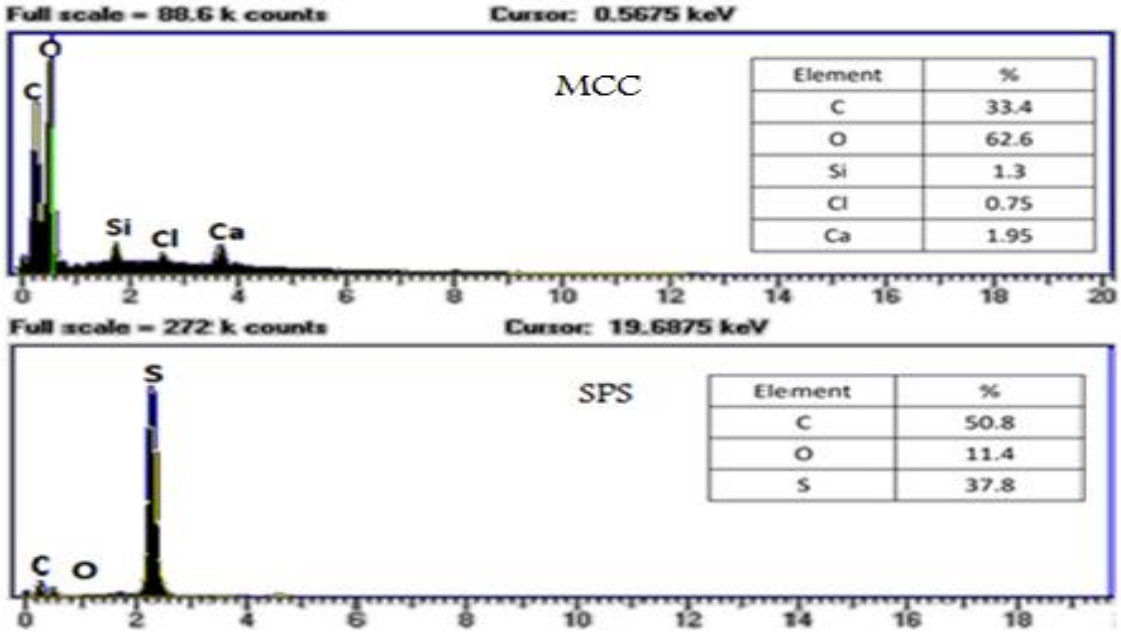


Fig. (3): EDX analysis (% of elementary analysis) for the synthesized MCC and SPS

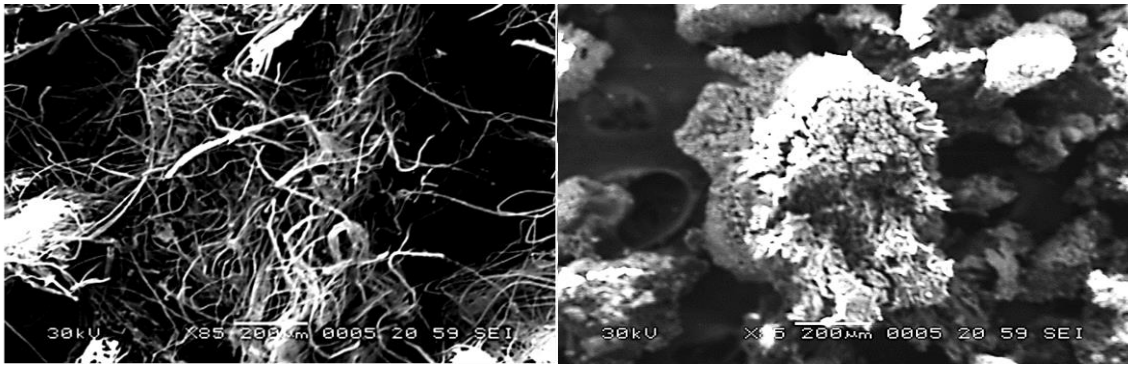
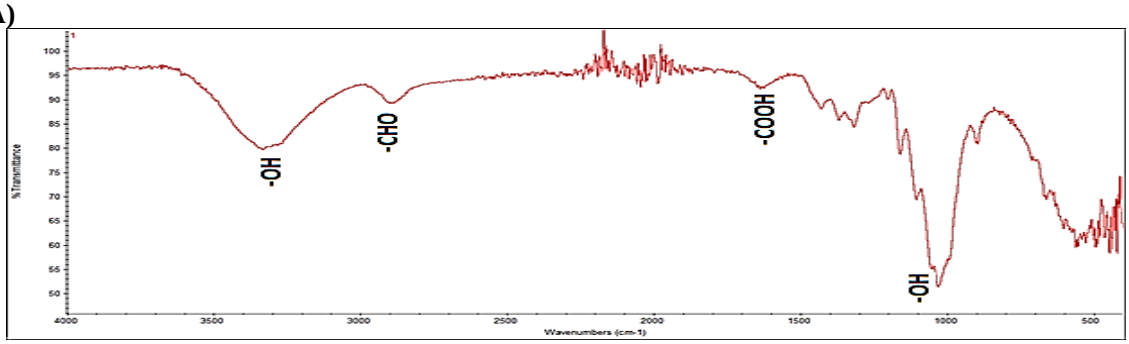


Fig. (4): SEM images of the synthesized MCC (left) and SPS (right) (magnification 85x)

(A)



(B)

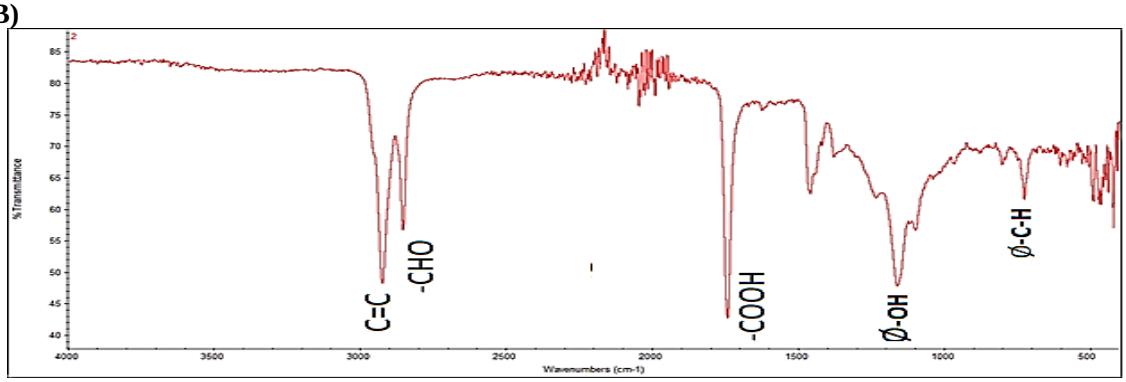


Fig. (5): FT-IR analysis for M-10 and S10 respectively

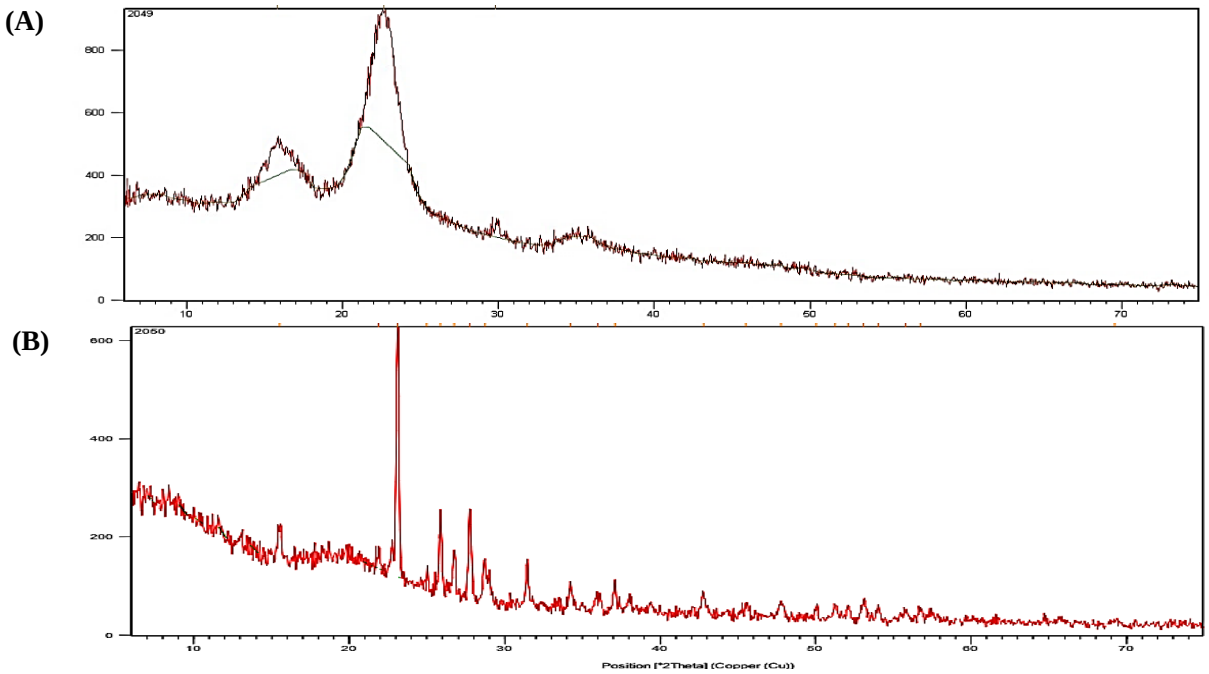


Fig. (6): XRD analysis for (A) M-10 and (B) S-10 respectively

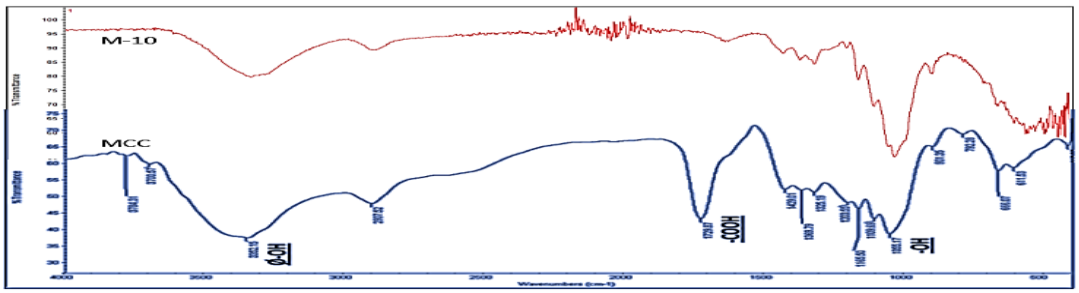


Fig. (7): FT-IR analysis for MCC and M-10

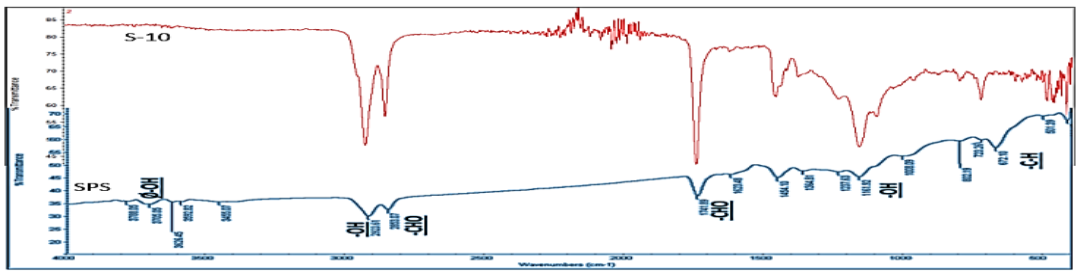


Fig. (8): FT-IR analysis for SPS and S-10

Irradiation using gamma ray at 10 KGy assigned the functional groups. The irradiation dose affected the carboxylic group (was at 1729 cm^{-1}), which sifted and decreased, while the hydroxyl groups (that was at 1055 cm^{-1}) was more sharper and it increased.

The effect of radiation dose on the polymer polysulfide was observed as the peak acute increase, especially between 400 cm^{-1} and 3000 cm^{-1}, which means that the effective functional groups (hydroxyl and aldehydes) can be increased and be more efficient for metal ion sorption. On the other hand, the hydroxyl groups at wavelengths of more than 3000 cm^{-1} were decreased.

These changes in functional groups will influence the sorption (elimination) behaviour. Paralleled Figures of FT-IR for each sorbent and its irradiated one are Figures (7 and 8) for MCC with M-10, SPS with S-10, respectively.

X-ray diffraction analysis for sorbents and their irradiated modifications are illustrated in Figures (9 and 10) for MCC with M-10 and SPS with S-10, respectively. In these Figures, no obvious changes can be detected. This may be due to the fact that irradiation did not affect the sorbents crystallinity.

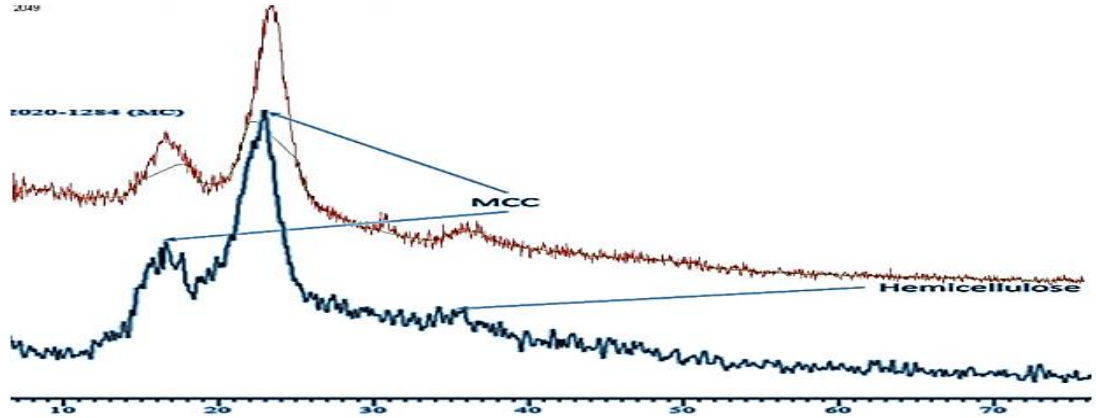


Fig. (9): XRD analysis for MCC and M-10

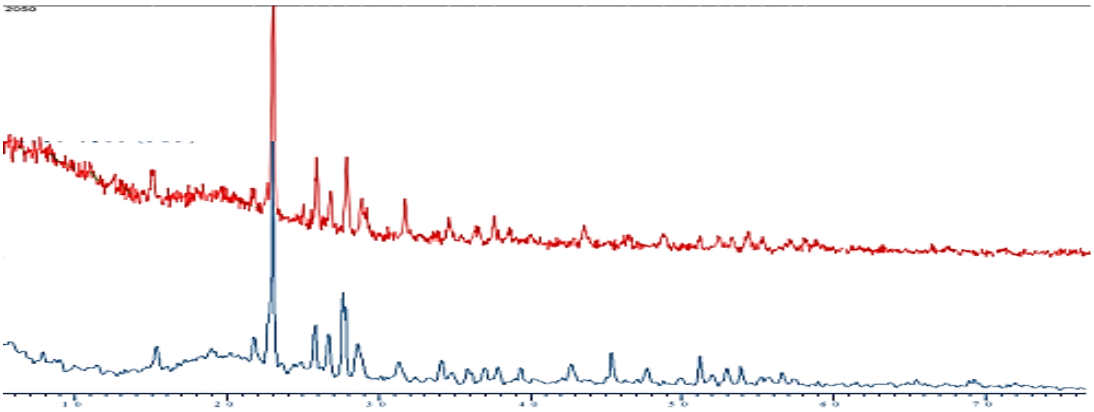


Fig. (10): XRD analysis for SPS and S-10

Effect of the initial concentration of the contaminant on sorption efficiency of sorbent materials

Adsorption experiments were performed with variable initial concentrations of metal ions from 0 to 200 mg L^{-1} using 2 g L^{-1} of MCC or SPS. Results illustrated in Figure (11) showed that sorbed amounts of the tested PTEs (Pb, Cd, and Cu) were increased with increasing initial PTEs concentrations. At the same initial concentration of metal ion, sorption of Pb^{+2} using MCC reflected the highest values which ranged from 1.9 to 28.5 mg g^{-1} with increasing initial Pb^{+2} concentration from 10 up to 150 mg L^{-1} then decreased to 25.3 mg g^{-1} at 200 mg Pb^{+2} L^{-1}. Generally, sorption of cadmium and copper by MCC was higher than SPS sorbent.

Cadmium sorption ranged from 1.8 to 12.2 mg g^{-1} for MCC and 1.6 to 9.2 mg g^{-1} for SPS. However, Copper sorption ranged from 1.9 to 13.1 mg g^{-1} for MCC and 1.9 to 11.4 mg g^{-1} for SPS. The obtained data of sorption were similar to those reported by wei et al. **[30]** and Garba et al. **[16]** and for sorption of Pb, Cd, and Cu ions using MCC. **S**imilar results were reported by other researchers**[31]** for sorption of Cd ions on rice straw (raw powder and citric acid modified RS). These results may be related with the nature of each studied sorbent, especially the surface nature. Functional groups identified by FT-IR analysis for MCC were sharper than that obtained for SPS.

Effect of irradiation

The effect of radiation doses on the MCC and SPS sorbents was followed up at a certain initial contaminant concentration (20 mg L^{-1}). Results of sorbed amounts per unit masses (mg g^{-1}) of the studied contaminants (Pb, Cd, and Cu) using modified sorbents at different radiation doses (0, 5, 10, and 50 kGy) are illustrated in Figure (12).

The obtained results showed that sorption of the studied water contaminants was slightly increased by increasing the irradiation doses from 0 to 10 kGy, then tended to decrease at 50 kGy. This means that radiation doses affected MCC sorption sites on the sorbent surface, and a high radiation dose (50 kGy) resulted in a negative effect on the MCC sorbent surface. The changes of the sorption behavior as affected

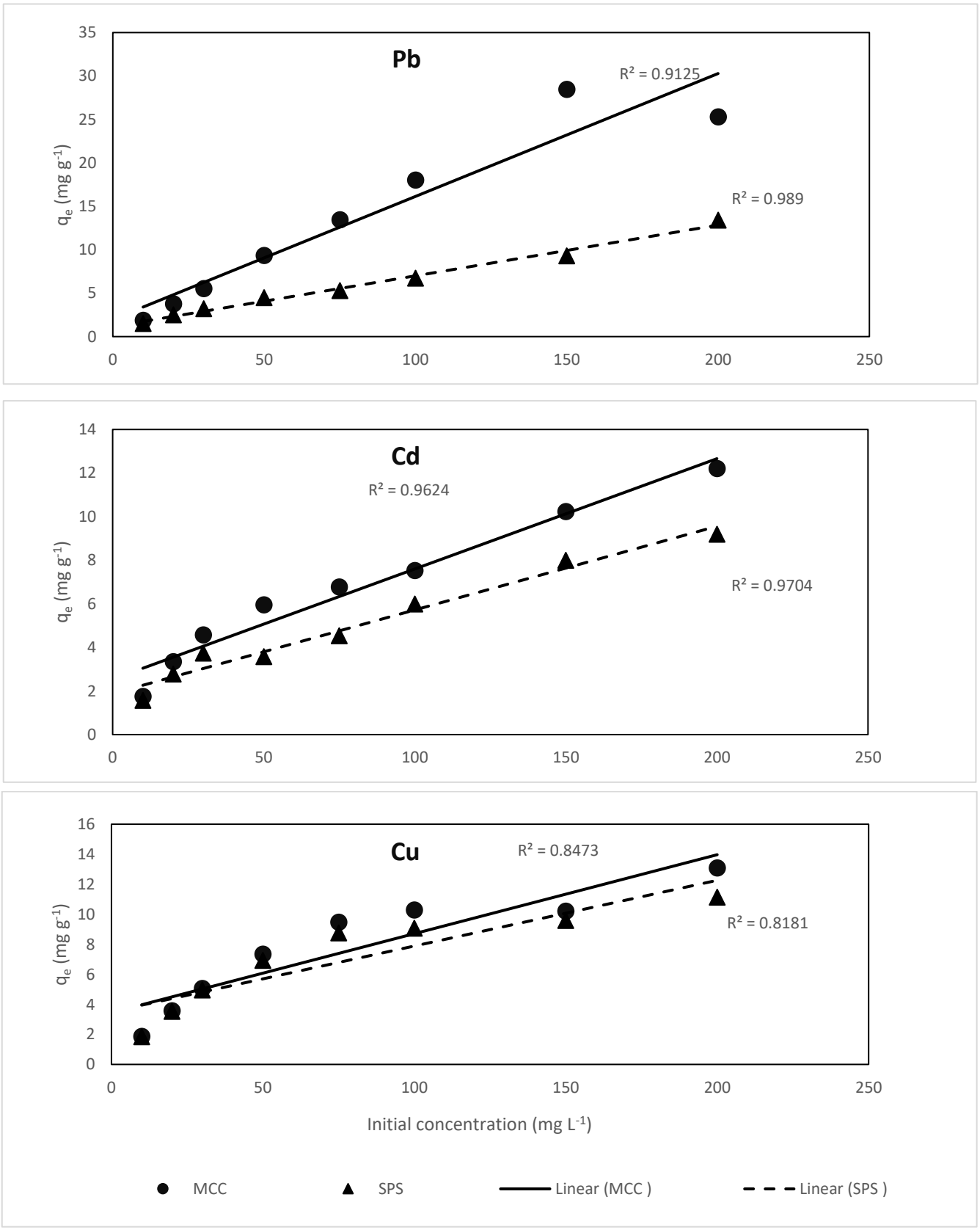


Fig. (11): Effect of solution initial concentration (mg L^{-1}) on sorbed amounts (mg g^{-1}) of PTEs (Pb, Cd and Cu) from their aqueous Solutions using sorbent materials

Microcrystalline cellulose (MCC) and Spongy polysulfide (SPS), Experimental conditions: pH =7, Temperature= 298 K, Equilibrium time= 180 minutes, solution volume= 0.05 L, sorbent dose=5g L^{-1} and Aging=300K

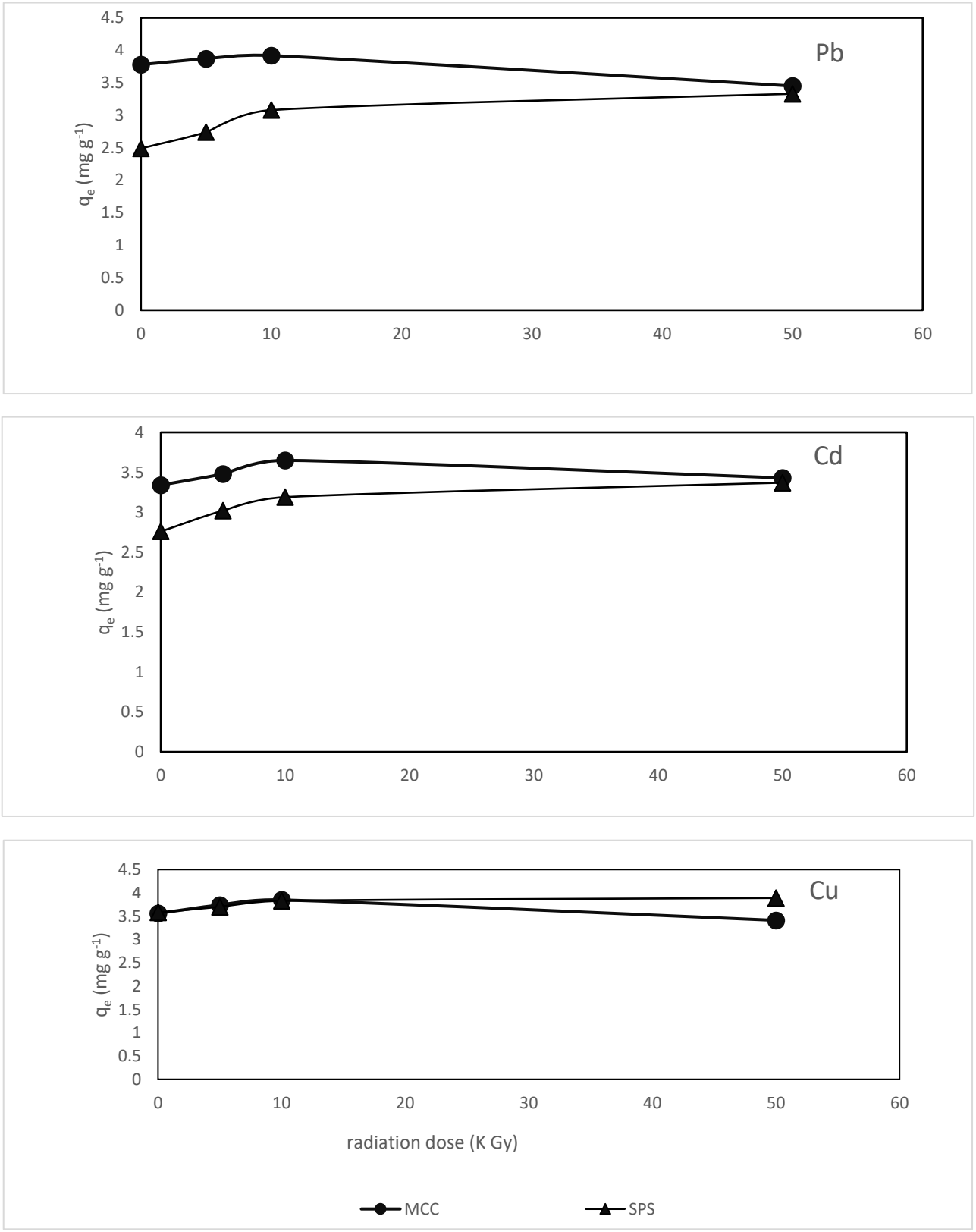


Fig. (12): Effect of Radiation Dose (K Gy) on sorption amount (mg g^{-1}) of water contaminants (Pb, Cd, and Cu) from their aqueous solutions using sorbent materials

Microcrystalline cellulose MCC, Spongy polysulfide SPS, and Spongy polysulfide polymer – Iron oxide nanoparticles (S-I)
Experimental conditions: initial concentration = 20 mg L^{-1}, Temperature= 298 K, Equilibrium time= 180 minutes, solution volume= 0.05 L, sorbent dose=5g L^{-1} and Aging=300K

by radiation doses can be explained by changes in functional groups. Increasing the radiation dose (more than 10 KGy) may have a negative effect on these functional groups.

CONCLUSION

Heavy metals removal from wastewater helps reuse of this water to minimizing the gap between available fresh water and demand, especially for agricultural sector. Using waste materials (rice straw and used frying oil) introduces low-cost and sustainable solutions for this problem. Synthesis of microcrystalline cellulose from rice straw and spongy polysulfide polymer conducted by the minimal chemical and thermal treatments, followed by irradiation by gamma rays (0-50 Kgy). Sorption of heavy metals (Pb, Cd and Cu) by these irradiated and non-irradiated materials was conducted.

Characterizations of the synthesized materials showed that applied treatments was successful and effective. Irradiation at 10 kGy gamma ray modified the functional groups on both of MCC and SPS. Sorption of Pb, Cd and Cu ions from aqueous solutions on MCC was higher than that for SPS. Irradiated MCC and SPS at 10 KGy was more effective for the removal of heavy metals than that for 50 KGy of gamma rays.

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