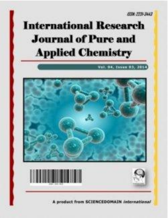

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A Remediation Study Using *Moringa oliefera* Seed (Uncarbonised and Carbonised) for the Adsorption of Chromium, Cadmium and Lead from Synthetic Water

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Authors' contributions

This work was carried out in collaboration between the three authors. Authors AM and JK designed the study and wrote the protocol. Author AM performed the studies, statistical analysis, managed the literature search and wrote the first draft of the manuscript with assistance from authors JK and TS. All authors read and approved the final manuscript.

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ABSTRACT

Moringa oleifera seed was treated and activated to prepare low-cost adsorbents whose adsorption properties would be compared. The uptake for lead, chromium, and cadmium from aqueous solution using the *Moringa oleifera* seeds biomass (MOS) and *Moringa oleifera* seed charcoal (CMOS) was studied. CMOS was prepared from MOS in order to make better use of this biomass material. Batch experiments were conducted under varying MOS/CMOS adsorbent dosages (0.5-1.5 g), contact time (30 min–6 hr) and metal ion concentrations (1-50 ppm) for chromium, lead and cadmium. The data for MOS fit well with Langmuir isotherm model for lead (maximum metal uptake

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(Q_m) = 1.281 mg/g) and Cadmium (maximum metal uptake (Q_m) = 0.168 mg/g) whereas CMOS can be modelled by both Freundlich and Langmuir isotherms for chromium (adsorption capacity = 0.2202 mg/g) and Lead (maximum metal uptake (Q_m) = 0.552 mg/g) respectively. However, the biomass of MOS was found to be more suitable than CMOS for the development of an efficient adsorbent for the removal of chromium, lead and cadmium from aqueous solutions. This study demonstrated that both the charcoal and uncarbonised biomass could be used as adsorbents for the treatment of Cr, Pb and Cd from aqueous solution.

Keywords: Moringa oleifera; heavy metals; biosorption; charcoal; adsorption isotherm.

1. INTRODUCTION

Heavy metals have come to be one of the main contaminants of water in recent years. They arise from different sources/activities carried out by industries [1]. Industries which neglect the implementation of environmentally friendly industrial waste disposal protocols tend to be the main agents for the contamination of water when they discharge the wastes with disregard to the future consequences thereof [2]. The contamination of water resources owing to negligent disposal of heavy metals has caused global worry for the last few decades [3]. Unlike organic pollutants, heavy metals are not degradable to form harmless end products and tend to be toxic to aquatic flora and fauna even in relatively low concentrations [4,5]. Metals, which are significantly toxic to human beings and ecological environments, include cadmium (Cd), arsenic (As), chromium (Cr), manganese (Mn), copper (Cu), mercury (Hg), nickel (Ni), iron (Fe), zinc (Zn) and lead (Pb) etc.[6] Some of these have a potential of being assimilated, stored and concentrated in the human body, inducing chronic pulmonary problems, erythrocyte destruction [7], nausea, diarrhoea, muscular cramps, renal degradation[8], salivation and skeletal deformity [9,10,11,12,13].

There is a need for developing a means of purification of water which is efficient and also cost effective since heavy metals are still toxic to humans even at very low concentrations. Other methods that have been utilised are ultra-filtration, reverse osmosis, ion exchange, solvent extraction and chemical precipitation, to mention but a few [2,14].

Knowledge that *Moringa oleifera* seeds can purify water is not new as it has been used as coagulant for turbid water with simultaneous treatment for bacterial faecal coliforms. Nations like India and Sudan have employed the seed for water treatment for generations. For example, Sudanese have utilised the seeds for water treatment since the beginning of the 20th century through a technique that comprehended the swirling of seeds in cloth bags with water for a few minutes and let it settle for an hour. [15]

Charcoal has also been widely used in its raw and activated state for the removal of heavy metals from aqueous solution. Several papers have been published in which charcoal from various sources has been used. Most of the sources are agricultural wastes which are formed as by-products of agricultural produce [16,17].

In this paper, adsorption of heavy metals from synthetic wastewater is investigated using shelled de-oiled *Moringa oleifera* seeds biomass (MOS) and *Moringa oleifera* seed charcoal (CMOS). A comparison between the MOS and CMOS is conducted with write to their metal uptake. The efficiency of the biosorbent materials in heavy metal adsorption is compared and fitted to some adsorption isotherms. The heavy metal ions used in this study are chromium, lead and cadmium.

2. METHODS AND MATERIALS

Moringa oleifera seeds were obtained from the Crop Science Department, University of Zimbabwe. 1000 ppm Standard solutions for the Cr, Cd and Pb were purchased from P & N Scientific Solutions. The metals were analyzed using a Shimadzu AAS6701F spectrophotometer with an air-acetylene flame.

2.1 Pre-treatment of Adsorbents

Dry MO seeds were obtained and stored at room temperature prior to use. The seeds were unshelled by hand and the kernels were washed with copious amounts of distilled water to remove any adhering dirt before drying them in the oven at 65ºC for 24 hrs. The seed kernels were ground to powder using mortar and pestle and the oil was extracted by soxhlet extraction using hexane as the extracting solvent. The powder was ground to a finer powder using mortar and pestle. A portion of the powder was then covered in a crucible and slowly heated until it had been turned to charcoal (CMOS) over a Bunsen burner at about 400^{0}C. Synthetic heavy metal solutions containing Cr, Pb and Cd metal ions were prepared for batch experiments at various concentrations from the 1000 ppm standard solutions. A solution of known metal ion concentration was prepared and a known quantity of adsorbent (MOS or CMOS) was added. The mixtures were shaken and the concentration of the unabsorbed metal ions in solutions was determined after separation of the sorbent by centrifugation and filtration using Whatman No. 1 filter paper. Cd was determined at 228.8 nm with a slit width of 0.5 nm. Pb was determined at 216.9 nm (slit width = 1.0 nm) and 283.3 nm (slit width = 0.2 nm). Cr was determined at 358 nm, 425.7 nm and 429 nm all with slit width of 0.2 nm. The two adsorbents were analysed using Analytical Technologies® INFRA 3000A FT-IR spectrometer by the KBr disc/pellet technique.

2.2 Effect of Contact Time

A 0.5 g mass of adsorbent (MOS or CMOS) was measured into 50 ml of a 25 ppm solution of Cr metal ions and was left to stand for 30 min before it was filtered. The filtrate was then determined by AAS for residual Cr ions in solution. This was repeated at varying times of 1 hr, 2 hr, 4 hr and 6 hr. This process was also done using Pb and Cd solutions.

2.3 Effect of Initial Metal Ion Concentration

A 0.5 g mass of adsorbent (MOS or CMOS) was added to a 50ml volume of the Cr metal solution whose concentration was 1 ppm. The mixture was allowed to stand for 1hr before it was filtered. The filtrate was then determined by AAS for residual Cr ions in solution. This was repeated for Cr metal solutions of concentration 5 ppm, 10 ppm, 25 ppm and 50 ppm each. This process was also done using Pb and Cd solutions.

2.4 Effect of Dosage

A 0.5 g mass of adsorbent (MOS or CMOS) was added to a 50 ml Cr metal solution of 15ppm and the mixture was left for 1 hr. The mixture was then filtered and the metal ion concentration of the filtrate was determined using AAS. This was repeated with 1 g and 1.5 g masses of the respective adsorbent. The process was then repeated using Pb and Cd solutions.

2.5 Calculation of Removal Percentage and Adsorption Capacity and Adsorption Isotherm Studies

The amount of metal ion adsorbed onto the MOS/CMOS, Q_e, was computed by the following equation:

$$Q_e = \frac{v}{m}(C_o - C_e) \tag{1}$$

where C_0 and C_e are the initial and equilibrium concentrations of lead ion in solution, whereas v and m are solution volume and mass of adsorbent, respectively. Langmuir and Freundlich isotherms were then applied to the experimentally obtained data (i.e. plot of C_e/Q_e vs C_e for Langmuir and plot of $\log Q_e$ vs C_e for Freundlich) [18,19,3]

3. RESULTS AND DISCUSSION

3.1 Functional Group Characterization of Adsorbents

The MOS spectra showed a broad peak at ~3350 cm^{-1} for O–H, sharp peaks at 2920 cm^{-1} for C-H stretch, 1660 for C=C and 1550 cm^{-1} for C=O. The O–H bands are known to occur at higher frequencies, usually as a sharp absorption at about 3600 cm^{-1}.Generally, you will observe a broad absorption at anywhere from 3500 to 2900 cm^{-1} since OH groups form strong hydrogen bonds that vary in length and strength. The other peaks observed were at ~1055, ~1115 for C–O single bond stretch and/or SO_2 symmetrical S=O stretch, ~1240 for P=O double bond stretch and ~1410-1450 for the deformation of the CH_2 [20].

The spectra of the adsorbents in Fig. 1, shows that there is a reduction in the intensity of the absorption peaks from MOS to CMOS since most of the functional groups present in the MOS were lost as result of conversion to form CMOS. This loss of functional groups infers that the two adsorbents should have distinct sorption characteristics for the metals under study as the adsorption mechanisms are different. That is, MOS should predominantly exhibit complexation phenomenon whilst CMOS would be that of physisorption.

3.2 Effect of Initial Concentration

CMOS was found to have better adsorption of Chromium from solution over a wide concentration range than MOS (Fig. 2). MOS exhibited more effective in removal Lead from solution as compared to CMOS (Fig. 3). The effectiveness of both sorbents was however comparable with cadmium though CMOS showed better removal up to about 10 ppm (Fig. 4). Both sorbents are very effective in removing ions in low concentrations. The efficiency is then reduced as the concentration increases. As the concentration is increased the percentage removal of both curves decreases for both curves. The binding sites which are available are constant for a fixed amount of the biomass added, thus the same amount of metal ions will be bound. This leads to a fall in percentage removal of the metal ions as the initial metal ion concentration is increased [21].

3.3 Effect of Contact Time

The curves in Figs. 5, 6 and 7 generally show that there is rapid uptake in the first hour which is followed by a slow release of the ions back into the solution. This behaviour may be attributed to saturation of the available adsorption sites present on the MOS/CMOS. That is, the decrease in concentration seized due to the saturation of adsorption sites. After all the sites were occupied, the ions were then slowly released back into the solution. The relative high charge density and small radii of the heavy metal ions results in strong hydrolysis by water. Thus the solution will compete with the adsorption sites for the metal ions resulting in the slow release of ions back into solution with time until an equilibrium is attained [21]. Uptake of lead by MOS continued with increasing time thus implying that lead is less hydrolysed by water as compared to the other metals under study.

MOS showed that it was more effective in the first hour than CMOS for lead and cadmium, whilst CMOS was more effective with chromium in the first hour.

3.4 Effect of Sorbent Dosage

Increasing the amount of MOS/CMOS increases the extent of ion removal from the solution as shown in the Figs. 8, 9 and 10 below and in other similar studies [22,23,1,18,24]. The increase is attributed to fact that more adsorption sites are made available when the mass of the adsorbent is increased [24]. We find that MOS is once again more powerful when removing metal ions from solution than CMOS is. This phenomenon is clearly seen especially with lead where all the lead ions are removed.

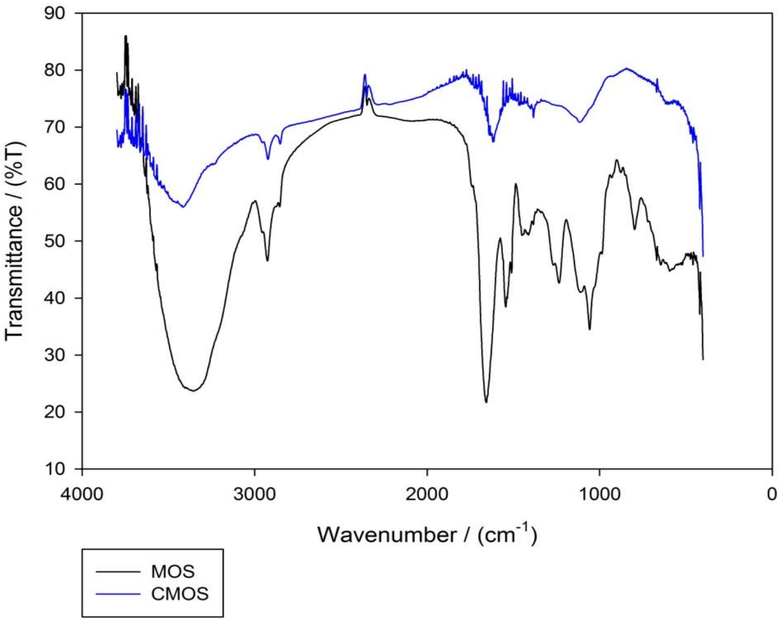

Fig. 1. FT-IR spectra of MOS and CMOS

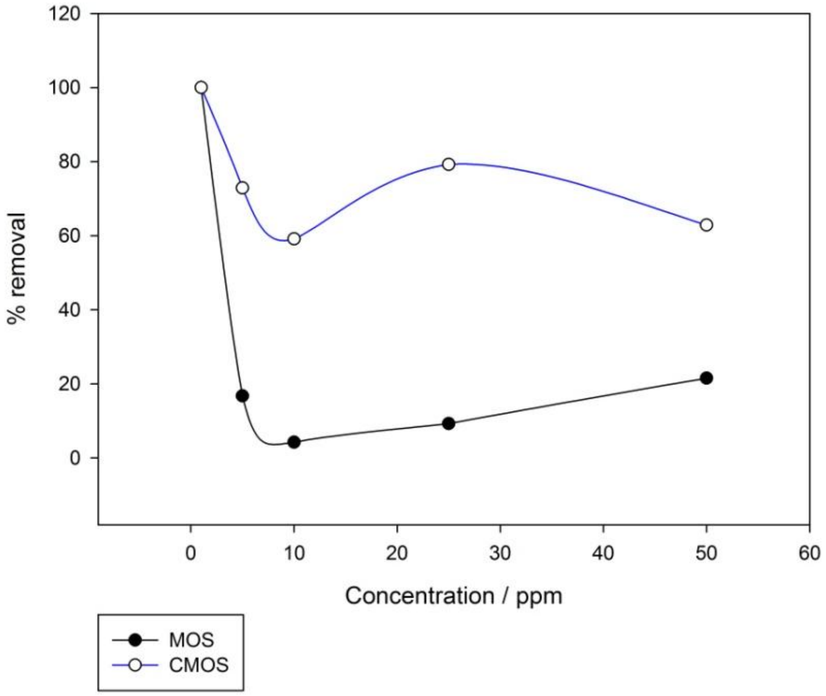

Fig. 2. Effect of initial concentration of chromium on metal removal using 0.5 g MOS AND CMOS powder

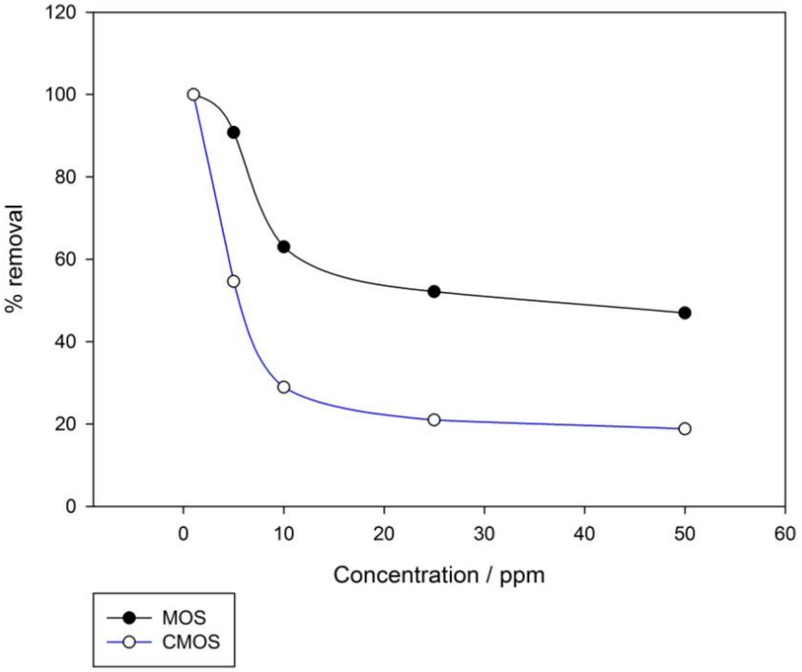

Fig. 3. Effect of initial concentration of lead on metal removal using 0.5 g MOS and CMOS powder

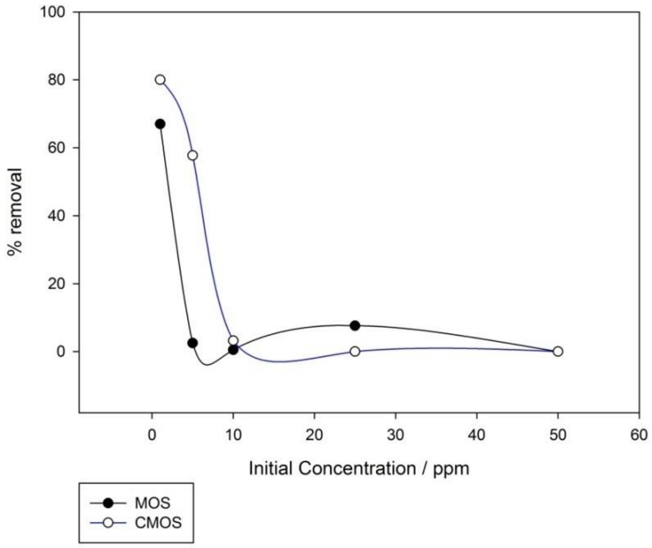

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Fig. 4. Effect of initial concentration of cadmium on metal removal using 0.5 g MOS and CMOS powder

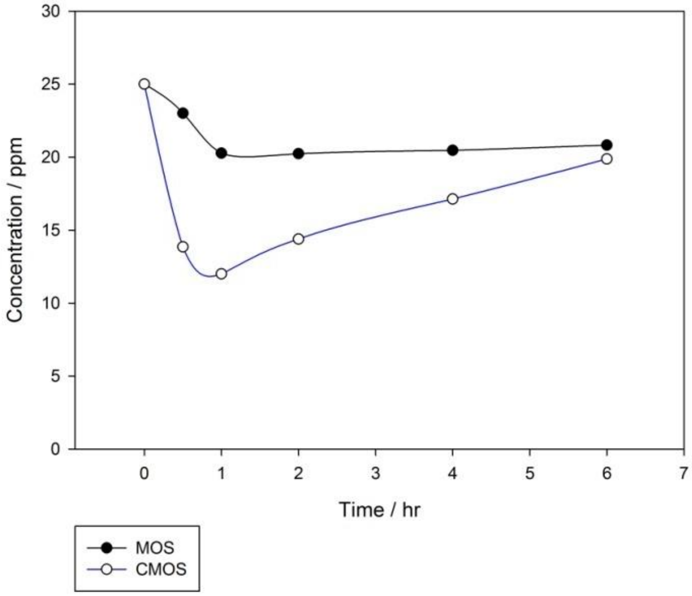

Fig. 5. Effect of contact time on chromium removal using 0.5 g MOS and CMOS powders at initial chromium concentration of 25 mg per L

The cadmium curves below (Fig. 10) show a very rapid uptake by MOS whilst that of CMOS is very gradual. This may be ascribed to the action of hydroxyl and carboxylic groups that are found in MOS that chelate the metal ions from solution. These groups are thus more efficient in removing metal ions from solution than the adsorptive forces found in the CMOS [2]. The FT-IR spectra (Fig. 1) of the two adsorbent confirms this, since there is a decrease functional groups present in CMOS as compared to MOS. Thus, the complexation phenomenon by the functional groups in MOS is generally more effective than physisorption. There is however minimal complexation due to the residual functional groups in CMOS as observed in the weak broad O-H peak and C=O peak as shown in Fig. 1.

3.5 Adsorption Isotherms

Adsorption isotherms are important criteria in optimizing the use of adsorbents as they describe the nature of adsorbate-adsorbent interaction. Thus, analysis of experimentally obtained equilibrium data by either theoretical or empirical equations is effective for practical design and operation of adsorption systems. The Langmuir and Freundlich adsorption isotherms were applied to each metal under study.

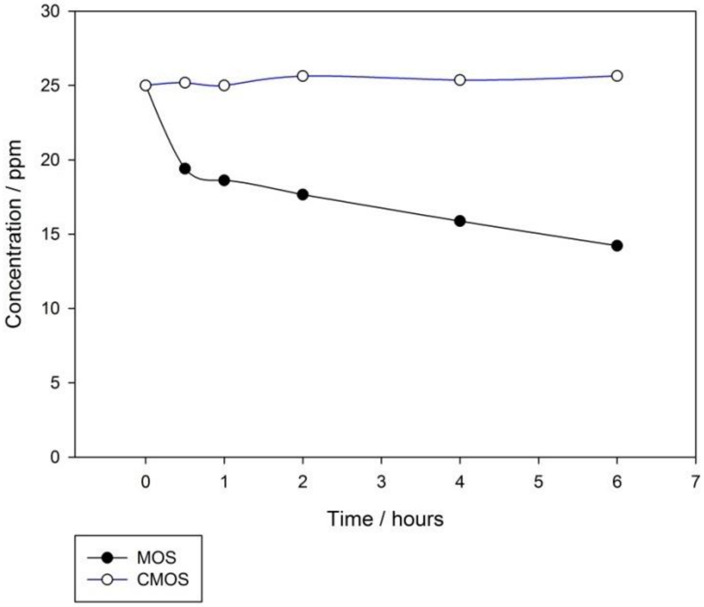

Fig. 6. Effect of contact time on Lead removal using 0.5 g MOS and CMOS powders at initial lead concentration of 25 mg per L*

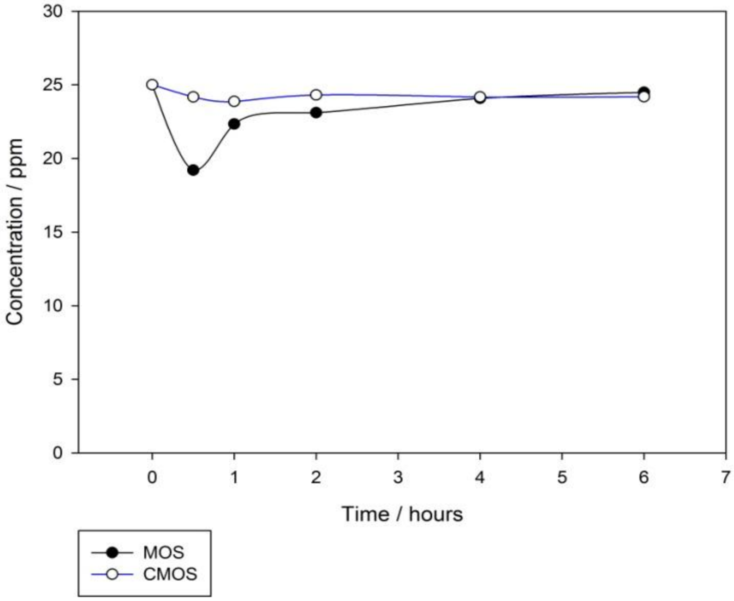

Fig. 7. Effect of contact time on cadmium removal using 0.5 g MOS and CMOS powders at initial cadmium concentration of 25 mg per L

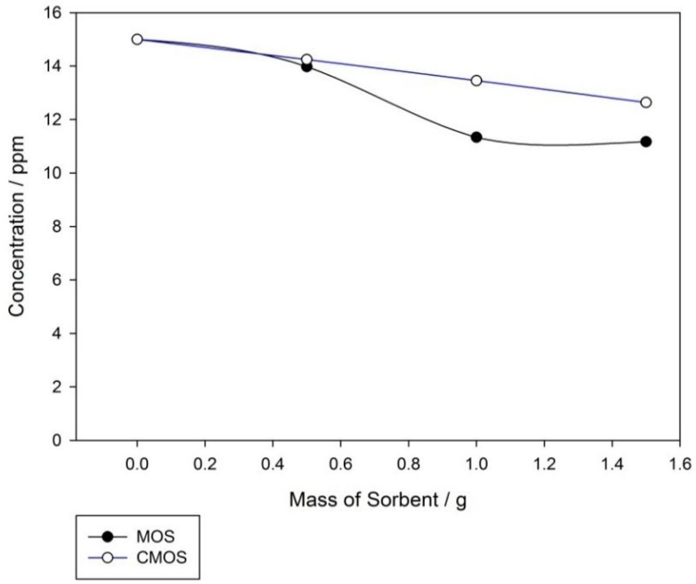

Fig. 8. Effect of dosage of MOS and CMOS powders on removal of chromium ions at initial chromium concentration of 15 mg per L

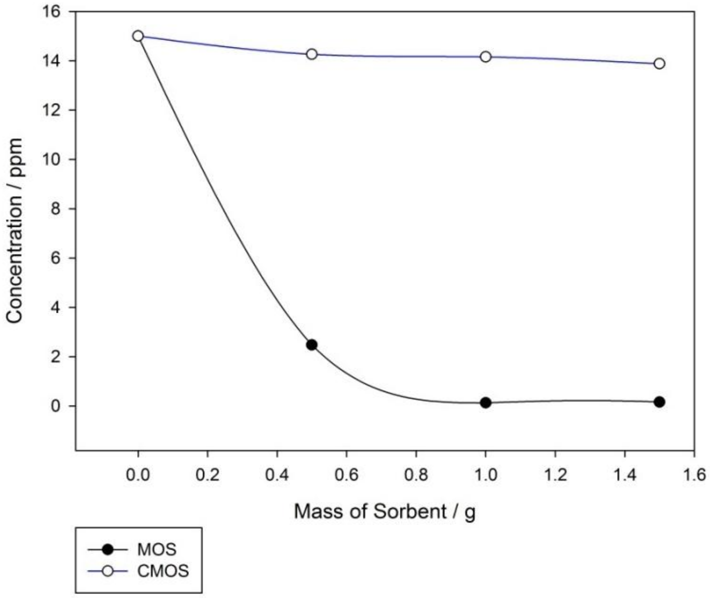

Fig. 9. Effect of dosage of MOS and CMOS powders on removal of lead ions at initial concentration of 15 mg per L

3.5.1 Langmuir Isotherm

The Langmuir equation relates the coverage of particles on a solid surface to the concentration of a medium above the solid surface at a fixed temperature. This isotherm founded on the following assumptions, that is to say, adsorption is limited to monolayer coverage, all surface adsorption sites are the same with each site accommodating one adsorbed particle and the propensity of a particle to be adsorbed on a given site is independent of its neighbouring sites occupancy. By employing these assumptions and a kinetic principle (rate of adsorption and desorption from the surface is equal), the Langmuir equation can be written in the following form:

$$Q_e = \frac{Q_m.b.C_e}{1+b.C_e} \qquad (2)$$

Where:

- C_e – equilibrium concentration of metal ions in the liquid
- Q_e – equilibrium concentration of metal ions in the solid phase
- Q_m – Langmuir constant for maximum metal uptake
- b – Langmuir sorption equilibrium constant

The above Langmuir equation is often written in the linear form as follows:

$$\frac{C_e}{Q_e} = \frac{1}{Q_m} C_e + \frac{1}{b.Q_m} \qquad (3)$$

Q_m and b can be calculated from the slope and intercept of the linear plot, C_e/Q_e versus C_e. That is, Q_m = 1/slope and b = 1/(intercept).(Q_m) .

3.5.2 Freundlich isotherm

Freundlich isotherm is an equation that is derived from experiment and observation rather than theory. This equation is one amidst the most widely utilised isotherms for the detailing of adsorption equilibrium. The Freundlich isotherm is able to describe the surface assimilation of organic and inorganic compounds on widely diverse adsorbents including biosorbent. This equation has the following form:

$$Q_e = K_f C_e^{1/n} \qquad (4)$$

Where:

- Q_e and C_e are the equilibrium concentrations of metal ions in the adsorbed and liquid phases
- K_f is the Freundlich constant characteristic of the system, indicating the capacity
- n is the Freundlich constant characteristic of the system, indicating the adsorption intensity

This equation can also be expressed in the linearized logarithmic form as follows:

$$\log Q_e = \log K_f + \frac{1}{n} \log C_e \qquad (5)$$

The plot of log Q_e versus log C_e has a slope with the value of 1/*n* and an intercept magnitude of log K_f. Thus, the Freundlich constants K_f and n can be worked out from the slope and intercept of the linear plot of log Q_e against log C_e. log K_f is equivalent to log Q_e when C_e equals unity. However, in other case when $1/n \neq 1$, the K_f value depends on the units upon which *Q*e and C_e are expressed. On average, a favourable adsorption tends to have Freundlich constant *n* between 1 and 10. Larger value of *n* (smaller value of 1/*n*) implies stronger interaction between biosorbent and heavy metal while 1/*n* equal to 1 indicates linear adsorption leading to identical adsorption energies for all sites. This isotherm attempts to incorporate the role of substrate-substrate interactions on the surface [25,3].

Table 1. Equilibrium model parameters for adsorption of Cr, Pb and Cd by MOS and CMOS

	Chromium		**Lead**		**Cadmium**	
	MOS	**CMOS**	**MOS**	**CMOS**	**MOS**	**CMOS**
Langmuir isotherm						
Q_m / mgg^{-1}	N/A	N/A	1.281	0.552	0.168	N/A
b / Lmg^{-1}	N/A	N/A	0.8757	2.5348	0.8704	N/A
Freundlich isotherm						
K_f / mgg^{-1}	N/A	0.2202	N/A	N/A	1.5435	N/A
n / Lg^{-1}	N/A	1.0304	N/A	N/A	0.3546	N/A

NB: Qm is the Langmuir constant for maximum metal uptake, b is the Langmuir sorption equilibrium constant, Kf is the Freundlich constant characteristic of the system, indicating the capacity, n is the Freundlich constant characteristic of the system, indicating the adsorption intensity ,N/A is when data cannot be explained by the model

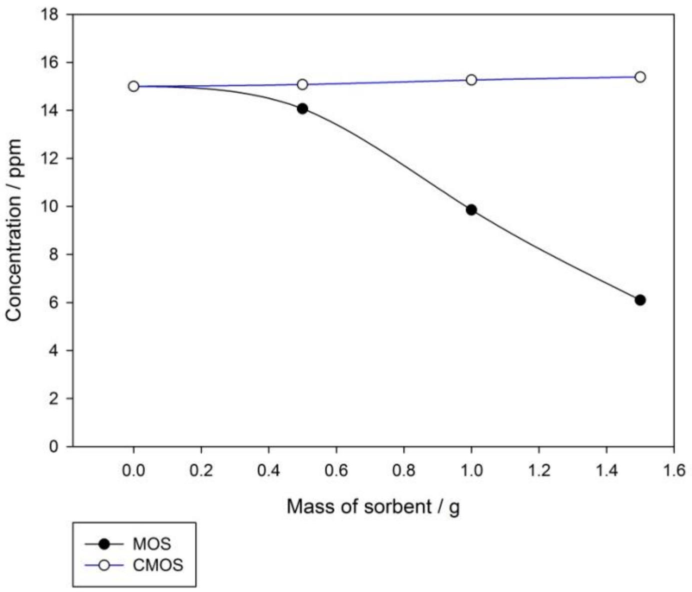

Fig. 10. Effect of dosage of MOS and CMOS powders on removal of cadmium ions at initial concentration of 15 mg per L

On average, a favourable adsorption tends to have Freundlich constant n between 1 and 10 [25]. Therefore the adsorption of chromium by CMOS was favourable since n = 1.0304 whilst that of cadmium by MOS was otherwise as shown in Table 1. The basic assumption of Langmuir adsorption isotherm is based on monolayer coverage of the adsorbate on the surface of adsorbent [18], which is an indication of the fact that the adsorption of Lead (II) ions onto MOS and CMOS generates monolayer formation as well as cadmium (II) ions for MOS.

4. CONCLUSION

In the present study, batch adsorption experiments for the adsorption of Cr, Pb and Cd ions from aqueous solutions have been carried out using MOS and CMOS as adsorbents. The adsorption characteristics have been examined at different contact times, initial Cr, Pb and Cd ion concentrations, and different adsorbent dosage levels. In conclusion the study has shown that MOS and CMOS powder can be used for chromium, lead and cadmium ion removal from aqueous solution. The increase in initial chromium, lead and cadmium concentration results in a decrease of percent removal of the chromium, lead and cadmium. The equilibrium time for the adsorption of chromium, lead and cadmium by MOS and CMOS is ~1 hr. The percent removal of chromium, lead and cadmium is increased with increase in the MOS and CMOS dosage up to 1.5 g of powder per 50 ml. The data for MOS fit well with Langmuir isotherm model for Lead and Cadmium whereas CMOS can be modelled by both Freundlich and Langmuir isotherms for chromium and Lead respectively. Overall, CMOS is a promising agent for removal of Chromium, Lead and Cadmium ions from aqueous solution though it is not as effective as MOS. The effectiveness of CMOS can be possibly improved by activation.

COMPETING INTERESTS

Authors have declared that no competing interests exist.

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