Alginate Modified Graphene Oxide for Rapid and Effective Sorption of Some Heavy Metal Ions from Aqueous Solution

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Abstract

Herein, we investigated a facile strategy to modify the graphene oxide (GO) with sodium alginate (SA) through using tetraethylorthosilicate (TEOS) as binding agent. The GO-SA was highly loaded with carboxylated ($-\text{COO}^-\text{Na}^+$) groups which permitted for fast and efficient interaction with the metal ions. Therefore, the prepared composite was employed as an efficient adsorbent to uptake of some heavy metal from aqueous solution. The as-synthesized GO-SA was characterized with various advanced techniques before and after the removal process. The analysis of the experimental data showed that, the Langmuir model excellent fit the adsorption data with maximum adsorption capacities of 887.21, 161.25 and 139.62 mg g^{-1} for Pb^{2+} , Zn^{2+} and Cd^{2+} , respectively. Moreover, the GO-SA presented a good regeneration and reuse ability with enhancing in the removal percent for all the studied metal ions. In addition, the prepared composite showed a suitable selectivity for Pb^{2+} from Zn^{2+} and Cd^{2+} co-existed solution.

1. Introduction

Nuclear fuel cycle creates extensive amounts of aqueous waste solutions that is polluted with different heavy metals. Some of the toxic heavy metals like zinc, lead, and cadmium create serious and toxic effects to the living environment. Cadmium, zinc, and lead ions exist in both industrial and radioactive waste. Cadmium has various radioisotopes among which ^{109}Cd with a half-life of 462.9 days. It is applied in nuclear reactors as control rods and shields to absorb neutrons (Aglan et al., 2014). It also has an important role in the production of nickel-cadmium (Ni-Cd) rechargeable batteries and as corrosion-protection coating for iron and steel. General industrial utilizes for cadmium are in alloys, batteries, solar cells, pigments, coatings (electroplating), and plastic stabilizers (Carolin et al., 2017). Zinc present in waste water streams of many industries such as steel works, in electroplating process and in intermediate level active waste (Ali & Ache, 1985). The addition of zinc to nuclear reactors greatly reduces corrosion processes, including stress corrosion cracking. Lead is highly toxic and can harm the nervous system and kidneys. The several sources of lead include effluents from battery manufacturing, painting pigment, steel industries, fuels, coating industries, and explosive manufacturing. Since lead effectively absorbs electromagnetic radiation of short wavelengths, it is utilized as a protective shielding around nuclear reactors for gamma rays, particle accelerators and X-ray equipment. It is also used as containers for transporting and storing radioactive materials (Dodd, 2004). Numerous trials were carried out to remove various heavy ions from the aqueous solution using adsorption approach. Rodríguez et al., 2020, studied the removal of aluminum and copper from acid mine drainage wastewater. They found that the decoration of GO with zinc oxide nanoparticles (ZnO) can remove the aluminum (Al) and copper (Cu) from acidic waters ($\text{pH} = 4$) with adsorption capacity 19.9 mg/g for Al and 33.5 mg/g for Cu over 30 min. Katubi et al., 2021, succeeded in adsorption of 636.94 mg g^{-1} of Pb-ion onto $\text{MnFe}_2\text{O}_4/\text{GO}$ composite at $\text{pH} = 7$ and the optimum contact time was 30 min. Li et al., 2020, investigated the preparation of graphene oxide/chitosan complex and its behavior for adsorption of copper, lead, and cadmium ions from simulated wastewater. They showed that the one gram of GO/CS composite can adsorb 60.7 mg of

copper, 48.7 mg of lead, and 32.3mg of cadmium at time 90 min and (pH = 6). Pourbeyram, 2016, studied the modified the graphene oxide surface with zirconium and phosphate to synthesis graphene oxide–zirconium phosphate (GO–Zr-P) nanocomposite. He observed that, the prepared composite (GO–Zr-P) can achieve maximum adsorption capacity for Pb(II) (363.42 mg g^{-1}), Cd(II) (232.36 mg g^{-1}), Cu(II) (328.56 mg g^{-1}), and Zn(II) (251.58 mg g^{-1}) at pH = 6 and after 20 min contact time. Removal of lead from aqueous solution by using nanocomposite of zinc oxide/graphene oxide (ZnO/GO) have been investigated by Ahmad et al., 2020. They reported that at pH = 5 after 160min mixing time the ZnO/GO possess maximum adsorption capacity of 418.78 mg/g for Pb(II). Bagheri et al., 2020, evaluated the prepared rGO-Fe⁰/Fe₃O₄-PEI nanocomposite for lead and cadmium removal. Their work showed that the maximum adsorption capacity 60.24 mg g^{-1} for lead and 54.47 mg g^{-1} for cadmium at pH = 6, and 60 min of contact time. Kong et al., 2020, studied the adsorption of Pb²⁺ and Cu²⁺ on graphene oxide-terminated hyperbranched amino polymer-carboxymethyl cellulose ternary nanocomposite (GO-HBP-NH₂-CMC). They reported that the maximum adsorption capacity of GO-HBP-NH₂-CMC towards Pb²⁺ is 152.86 mg g^{-1} and Cu²⁺ 137.48 mg/g at pH = 5 and mixing time 240min. Removal of Pb(II) has been studied by partially reduced graphene oxide-Fe₃O₄ composite from aqueous solutions by Guo et al., 2018. They found that one gram of the nano adsorbent can interact with 373.14 mg of Pb²⁺ at pH = 6 within 10 min. Yang et al., 2020, employed the oily sludge pyrolysis residue for decontaminated the heavy metal from synthetic flotation wastewater. The experimental results showed that the maximum uptake of the used adsorbent for Cd²⁺ (106.16 mg g^{-1}), Pb²⁺ (140.65 mg g^{-1}) and Cu²⁺ (128.04 mg g^{-1}) at pH < 7.5 and contact time 45min. Ge and Ma, 2015, used the microwave irradiation to fabricate triethylenetetramine-modified graphene oxide/CS (TGOCS). The affinity of TGOCS towards Cr(IV) from aqueous solution was 219.5 mg g^{-1} .

Sodium alginate (SA) is an anionic linear polysaccharide based on alternating blocks of β-(1→4)-linked d-mannuronic acid (M) and α-(1→4)-linked l-guluronic (G) residues. With a high density of free hydroxyl and carboxyl groups located over the alginate backbone, these are making the (SA) highly reactive and amenable for chemical modifications and complexation. On the other hand, the GO found as nanosheets with one C-atom thickness and various O-function groups distributed over GO nanosheet surface. Several studies were investigated in order to combine the GO and sodium alginate; Yu et al., 2019, were prepared hydrogel beads composed of polyvinyl alcohol (PVA)/graphene oxide (GO)-sodium alginate (SA) via crosslinking in saturated solution of solution of saturated boric acid and CaCl₂. They observed that PVA and SA molecules were bonded with GO layers via H-bond which cause significant disorder of the layered structure of GO. In addition, the increasing in the GO dose the hydrogel beads lose its ballbility and network structure, leading to enhance its permeability and adsorption capacity towards Pb²⁺ (279.43 mg g^{-1}). Yu et al., 2021, were employed the vacuum filtration technique to fabricate self-support composite membrane formed from GO/ SA/1, 2-Propanediamine (PDA) for adsorption of Pb²⁺. The adsorption experiments revealed that the equilibrium adsorption was reached after 20 min. And according to Langmiur, the maximum adsorption capacity for Pb²⁺ was 189.25 mg g^{-1} . He et al., 2020, investigated a novel hydrogel adsorbent consisted of yttrium-immobilized-graphene oxide-alginate (Y-GO-SA) for

removal of adsorption of As and tetracycline (TC). The adsorption process was pH-dependent. The maximum adsorption capacities were obtained 273.39 mg g⁻¹ (As(V)), and 477.9 mg g⁻¹ (TC). Jiao et al., 2016, were prepared porous aerogel constructure from sodium alginate/graphene oxide (SAGO) using in-situ crosslinking and freeze-drying strategy. The addition of GO, as reinforcing filler, enhance various properties of the obtained SAGO aerogel such as; porous structure, mechanical strength, elasticity, and the compression strength. Referring to Langmuir isotherm, the maximum removing capacities for Cu²⁺ (98.0 mg g⁻¹) and Pb²⁺ (267.4 mg g⁻¹). Zhou et al., 2018, were employed the hydrothermal polymerization of lignin, sodium alginate and GO in an aqueous solution to form 3D porous composite (PG/L/SA). The results obtained from the adsorption experiments showed that the PG/L/SA achieved maximum adsorption ability of Cd(II) and Pb(II) of 79.88 and 226.24 mg/g, respectively. Pan et al., 2018, were used sacrificial template of polystyrene particles to form porous calcium alginate/graphene oxide composite aerogel (mp-CA/GO). They observed that, the as (mp-CA/GO) possess suitable adsorption capacities towards Pb²⁺ (368.2 mg g⁻¹), Cu²⁺ (98.1 mg g⁻¹) and Cd²⁺ (183.6 mg g⁻¹) and adsorption equilibrium achieved after 40 min. Yang et al., 2014, was employed a facile preparation method for synthesis CS/GO via H-bonding interaction between CS and GO. They found that the CS/GO possessed adsorption capacity 202.5 mg/g to Cu(II).

As we can see before, the scientists used various strategies for preparation of GO/SA hydrogel (porous structure, beads, layer, aerogel, etc). The preparation of these composites was required multi-steps. Moreover, a part of the bind sites was consumed by crosslinking or by shieling interior in the bulk gel. As a result, the time for reaching equilibrium will be elongated and the adsorption capacity will be reduced.

In this work, we suggest simple modification of the GO surface with SA through using TEOS as bridge to link the hydroxyl groups of both GO and SA. This manner will provide extra and efficient binding site (-COO⁻Na⁺) available for highly rapid chelating the heavy metal ion. The GO-SA composite was fabricated using mild and green conditions, characterized with different techniques and applied for removal of Pb²⁺, Zn²⁺ and Cd²⁺ from aqueous solution using batch technique.

2. Experimental

2.1. Materials and instrumentation

The chemicals required were of analytical grade and utilized without further purification. H₂SO₄ (95–97%, Riedel deHaen), H₂O₂ (36%, Pharaohs Trading and Import), HCl (30%, El Salam for Chemical Industries), KMnO₄ (99%, Long live), and graphite (200mesh, 99.99%, Alpha Aesar), sodium alginate (Alpha Chemika, analytical reagent), TEOS (99%, Across), ethanol absolute (Sigma-Aldrich), Pb(NO₃)₂ (99%, Sigma-Aldrich), Cd(NO₃)₂ · 4H₂O (99%, Sigma-Aldrich), ZnSO₄ · 5 H₂O (99%, Loba Chemie).

Analytical balance (CP 2245, Sartorius, USA.), Hot plate stirrer (IKA, C-MAG HS7, IKA®-Werke GmbH & Co. KG, Germany), pH meter (3510, Genway), Hot plate stirrer (SB 162, Stuart, UK.), Centrifuge, (Mikro 220R,

Hettich, UK.).

2.2. Preparation of GO and GO-SA composite

GO was synthesized according to our previous strategy (Abd-Elhamid et al., 2018). The GO-SA was fabricated via simple mixing approach. In detail, (4.0 g) of sodium alginate was added to 1000 mL distilled water under stirring until solution become clear. Thereafter, GO (0.15 g) were appended to the alginate solution at constant stirring to form homogeneous GO-alginate suspension solution (a). On another beaker, alcoholic solution of TEOS was prepared separately, by adding 5.0 ml TEOS to 25 ml absolute ethanol solution (b). Afterwards, solution (b) drop-wisely added to the solution (a), The final solution was kept stirring for 48 hr at 60 °C. The resulted viscous solid was separated by filtration washed several times with distilled water and stored for further use.

2.3. Batch Adsorption of heavy metals

A stock solution of 1000 mg/L was prepared by dissolving proper amount of every metal ion in double distilled water. A batch sorption experiments were conducted in order to study the parameters that affect the sorption of Pb(II), Cd(II) and Zn(II) such as shaking time, pH, v/m, initial metal concentration and temperature, where the optimized conditions were determined. The metal concentration was spectrophotometrically determined with a Shimadzu double beam spectrophotometer by using 4-(pyridyl-2-azo) resorcinol (PAR) (Marczenko, 1976). Batch experiments were done by shaking 2.8 mg of the prepared graphene oxide alginate with 5.0 ml of the solution of metal ions in a thermo-stated shaker bath adjusted at 25°C to achieve an adsorption equilibrium condition. The percentage removal, R%, of the Pb(II), Cd(II) and Zn(II) at equilibrium was calculated as given in Eq. 1.

$$R\% = \left(\frac{C_o - C_e}{C_o} \right) * 100$$

1

Where C_o and C_e are the initial and equilibrium concentrations (mg/L) of metals ions in solution, respectively.

2.4. Characterization

The prepared material was characterized by Transition Electron Microscope (TEM) analysis was carried out by a JEM-1010 unit (JEOL Ltd., Tokyo, Japan). Scanning Electron Microscope (SEM) (JEOL GSM-6610LV. Japan) with additional EDS unit, Light Optical Microscopy (LOM) (BX61, Olympus, Japan), Thermo-Gravimetric Analysis (TGA, Shimadzu Thermal Gravimetric Analysis (TGA)–50, Japan). Elemental composition of Cu (II) and Co (II) sorption on GO-C was displayed by an Oxford energy-dispersive X-ray (EDX) spectrometer (Oxford Link ISIS, Japan). Fourier Transmission Infra-Red Spectroscopy (FT-IR) (8400s, Shimadzu, Japan) covered the range from 400 to 4000 cm^{-1} and Raman Spectroscopy (Bruker, Senterra II, Germany).

2.5. Adsorption Kinetics Study (see supplementary materials)

2.6. Adsorption isotherm (see supplementary materials)

2.7. Thermodynamic Study of Adsorption (see supplementary materials)

3. Results And Discussion

3.1. Characterization

3.1.1. TEM and SEM

The TEM images of the GO layer appear as a single highly transparent sheet (Abd-Elhamid et al., 2018). After modification of the GO surface with alginate, the GO-SA composite layer appears to be dusk and cloudy, see Fig. 1a-c, which may be due to the modification with alginate.

The morphological structure of GO-SA composite was determined with SEM technique. Fig. 1d-h, presented the SEM photographs of the GO-SA at various magnifications. At low magnifications the composite appears as fully separated distributed particles (Fig. 1d) with increase in the magnification power the layered structure become clearer and cracks appears at the composite surface, Fig. 1e & f. At higher magnification, the cracks could be detected clearly into separated bulky structure which may be refers to the dried sodium alginate that coated the GO surface Fig. 1g & h. However, the morphology of the composite was different after the adsorption of the metal ions, see Fig. S1. Moreover, the resulted composite-metal (GO-SA-M) morphological structure depend on the type of the adsorbed metal ions. Where, after adsorption of Pb-ions the GO-SA-Pb complex seems as layers with particles deposited on its surface. While in case of adsorption of Cd-ions the structure of GO-SA-Cd complex not greatly alter than the original composite. Moreover, at adsorbing of Zn-ions the yielded GO-SA-Zn complex looks alike great agglomerated bulky structure. The alteration of the morphological structure of the composite after the adsorption process may indicate that the adsorption of the selected metal ions is by crosslinking adsorption mechanism.

3.1.2. FTIR

The chemical structure of the GO and modified GO (GO-SA) were illustrated employing the FTIR spectra, Fig. 2a. The GO possess bands at 3433 cm^{-1} , 1635 cm^{-1} , 1398 cm^{-1} and 1033 cm^{-1} referring to O-H stretching, O-H bending, COOH and C-O stretching, respectively (Febrianastuti et al., 2019). After modification of GO by the alginate and TEOS as crosslinker, the GO-SA shows bands at 3434 cm^{-1} (adsorbed water and O-H stretching), 2923 cm^{-1} (CH_2 group), bands at 1740 and 1347 cm^{-1} belongs to vibration modes of C-O bond related to the COOH groups. The peaks at 1625 and 1413 cm^{-1} crossponding to the asymmetric and symmetric stretching modes of carboxylate salt groups ($-\text{COONa}$), respectively (Nigiz, 2020).

The peaks in rang 1100–1000 cm^{-1} assigned to the (C-O, Si-O and C-O-C stretching) of the GO, TEOS and glycoside bonds in the polysaccharide, respectively (Fig. 2a). After adsorption of metal ions, FTIR spectra of GO-SA-M complexes was differ from the GO-SA spectrum. Where, the intensity of the bands in the region 1740-1100 highly weaken (Allouss et al., 2020). These observations may be due to the complexation interaction between the various function groups of the GO-SA composite and the M-ions (Cui et al., 2015), see Fig. S3.

3.1.3. TGA

Thermogravimetric analysis of the GO and GO-SA composite was investigated to detect the influence of the modification step on the thermal properties of the prepared composite. The TGA graph of the GO and GO-SA composite were presented in Fig. 2b. As obtained, four degradation steps were required for GO decomposition namely, Fig. 2b, (i) liberation the surface moisture evaporation (19.6%) in the range of 28-88 °C, (ii) releasing of adsorbed water (4.07%), in the range of 88-158 °C, (iii) decomposition of oxygenated function groups –OH and C-O-C (20.50%) in the range of 158-215 °C, and (iv) pyrolysis of –COOH in the range of 215-320 °C.

On the other hand, the thermograph for GO-SA showed seven pyrolysis stages, as illustrated in Fig. 2b. The existence the alginate decorated the GO-surface, will minimize the quantity of the adsorbed moisture and bounded surface water. This is due to the hydroxyl (-OH) on the GO surface. This is accounted on the possible bounding moisture and water molecule via H-bonding involved in the functionalization process. Upon linking of the GO with alginate via silane bonding, the thermal characters of the synthesized composite (GO-SA) extremely differ from that related to the GO. Where, the GO-SA release 8.00% moisture at temperature (78 °C) which is lower than that for the GO, see Fig. 2b. This may be due to partial ionic bond formation between the Na-ion of the alginate and the water molecule. Which resulted in attract low amounts of moisture molecules. On the other hand, the amount of intermolecular water by the GO-SA (7.22%) which evaporated at $T = 181$ °C, is larger than that showed for GO, refer Fig. 2b. This may be attributed to the properties of the alginate hydrogel to retain extra water molecules within its chain, which require higher temperature for release it (Ablouh et al., 2019; Gopalakannan et al., 2016).

Moreover, Liu et al., 2015, demonstrated that sodium alginate started to decomposes from 202 to 289 °C, this might be referred with the break of glycosidic bonds, dehydration, decarboxylation and decarbonylation of the alginate. Herein, GO-SA composite smoothy degrade and its weight reduced by 25.20% at 181-268 °C, 14.20% at 268-335 °C, 11.24% at 335-472 °C and 28.00% at 472-603 °C. This behavior may be ascribed as, the alginate chains modified the GO freely extend in the space and these chains gradually pyrolysis. Moreover, the alginate chains may be crosslinked with silane linker which leads to enhance the thermal properties of the prepared composite.

3.1.4. EDS analysis

The EDS is an important device that required to detect the elements related the prepared sample. In this research the composite (GO-SA) prepared employing GO, SA and TEOS, therefore, the EDS analysis detect

the presence of C, O, Si and Na atoms in the analyzed sample. By blending the composite materials with the M-ion (Pb, Cd and Zn) solutions. The EDS analysis detect the appearance of Pb, Cd and Zn and absence of Na in the analysis data of the analyzed samples. These results confirm that the adsorption process proceeded through cation exchange mechanism, Fig. 3.

3.2. Adsorption Experiments

3.2.1 Effect of contact time

In figure 4a the relation between removal percent (%R) against contact time is illustrated. From this figure it's clear that the removal percent (%R) is quite fast and reach equilibrium within 1.0 min shaking. The highly rapid sorption kinetics of the studied heavy metal ions may be referee to either surface complexation or strong chemisorption of these heavy metals on the GO-SA surface (Liu et al., 2015) and the separated layer structure of the GO-SA as appeared in the TEM analysis, see Fig. 1a-c.

To assess these results kinetically, the second order kinetic equation was applied (see supplementary materials). The results obtained are given in Fig. 4b. The relation coefficient and other various parameters were calculated from the linear relation between t and t/q_e and summarized in Table S2. We noted that, the $R^2 \geq 0.995$ (close to unit), moreover, the calculated (q_{cal} , mg g⁻¹) for all tested metal ion were greatly close to the adsorption capacity values measured from experiments (q_{exp} , mg g⁻¹). Therefore, we suggested that, the adsorption kinetics follow the pseudo-second-order kinetics model and the adsorption rate was mainly described via the chemical adsorption process.

3.2.2. Effect of solution pH

In sorption process the pH values of the aqueous solution consider an important factor. Where, the variation in the solution pH will induce on the behavior of adsorbent surface-active groups and the nature of the adsorbate species. Here, we adjusted the pH values according to the nature of heavy metal to avoid precipitation. It was found that, the removal efficiency of the GO-SA is highly dependence on the pH value of the contaminated solution, see Fig. 4c. At low pH values of the removal efficiency (%R) relatively low and sharply increase with further increase in the solution pH. This behavior may be attributed to, the GO-SA composite possess carboxylated (COO⁻Na⁺) groups on its surface and these groups were the responsible on interaction with the contaminated species. Therefore, at low pH the concentration of H⁺ is high and will block the adsorption active site against the heavy metal. On the other hand, by increasing the pH value the number of the H⁺ ion will be minimized and the surface-active groups (COO⁻) become highly ionized which provide a favorable sorption site for the heavy metal ion (Wang et al., 2016; Huang et al., 2018).

3.3.3. Effect of initial metal ion concentration

The metal ion concentration displays a vital role on retarding the mass transfer resistance among the aqueous and solid phases (Nurhajawarsi et al., 2018). The influence of the initial metal ion concentration was studied using batch approach in rang (200-1000 mg L⁻¹) for removal percent (%R) of Pb²⁺ and (25-

150 mg L⁻¹) for Cd²⁺ and Zn²⁺, Fig. 4d. It is clearly obvious that, the GO-SA uptake percent decrease with further increase in the initial concentration of the metal ion. This manner referee to at low initial concentration of the metal ions, the number of metal ions is relatively low compared with the number of available active surface function groups. While, at high initial concentration of metal ions, the ratio of the number of metal ions available binding sites at the composite surface is higher. Therefore, the adsorption percent (%R) tend to decrease (Taamneh et al., 2017).

Numerous isotherm models have been employed. Among these Langmuir and Freundlich models are the most applied (see supplementary materials) to fit the experimental results in order to describe the interaction behavior between the metal ions and the used composite GO-SA (Fig. 4e & f). The calculated parameters and correlation coefficient, R², revealed to the used models were recorded in Table 1. The R² values related to Langmuir model was > 0.98 which is higher than that of Freundlich isotherm for all studied metal ions. This clarify that Langmuir model is more suitable to represent the experimental data. Moreover, according to Langmuir model the GO-SA composite presents the maximum adsorption capacities (mg g⁻¹); Pb (884.96), Cd (134.23) and Zn (155.04), see Table 1.

Table 1

Langmuir and Freundlich constants for adsorption of Pb²⁺(t = 1 min, Dose = 2.8 mg, V= 5 ml, pH = 4, T = 25 °C) Cd²⁺(t = 1 min, Dose = 2.8 mg, V= 5 ml, pH = 7, T = 25 °C) and Zn²⁺(t = 1 min, Dose = 2.8 mg, V= 5 ml, pH = 5, T = 25 °C) form aqueous solution.

M ^{z+}	q _{exp} , mg g ⁻¹	Freundlich Parameters			Langmuir parameters			
		K _f , mg g ⁻¹	n	R ²	Q _o , mg g ⁻¹	b, mL mg ⁻¹	R _L	R ²
Pb ²⁺	887.21	184.17	4.26	0.977	884.96	0.018	0.217	0.983
Cd ²⁺	139.62	54.79	4.91	0.906	134.23	0.204	0.089	0.999
Zn ²⁺	161.25	29.27	2.81	0.919	155.04	0.067	0.229	0.995

3.3.4. Effect of (v/m) ratio

The effect of changing v/m (L/g) ratio on the sorption of Pb(II), Cd(II) and Zn(II) from aqueous solution on GO-SA is performed out at a v/m range of 1.79– 0.357 L/g. The obtained results are given in Fig. 5a, from which it is apparent that the sorption efficiency increases as the v/m ratio decreases. Accordingly, for Cd (II) and Zn (II), the sorption percent was increased from about 67.24to 89.45% for (100 mg/L) Cd (II) and increased from 63.72–86.90% for (100 mg/L) Zn (III) as v/m decreased from 1.79 to 0.357 L/g. While for Pb (II), the sorption efficiency was decreased from about 57.31–87.62% for (600 mg/L) Pb (III) as v/m decreased from 1.79 to 0.357 L/g. Therefore, the v/m ratio 0.446 L/g was selected for the removal process in the following experiments. This manner is attributed to, at low v/m ratio the number of the available active sites will be sufficient to adsorb the most of adsorbate species. As the v/m ratio

increase the number of the metal ion in the solution increase while the number of the binding sites maintain constant. Therefore, large number of the adsorbate species residue in the aqueous solution after the adsorption process, hence, the uptake efficiency will be reduced (Abu Elgoud et al., 2019).

3.3.5. Effect of solution Temperature

The impact of aqueous solution temperature in rang (25-65°C) on the removal percent (%R) of the examined heavy metal ion on the GO-SA composite was presented in Fig. 5b. It was noted that, the sorption efficiency of the all studied metals was increased with increase in the solution temperature from 25 to 65°C. This is due to, the increasing in the solution temperature will enhance the movement of the metal ion species from the bulk of solution to the adsorbent surface, leading to increase the complexation chance between the metal ion and the surface function groups.

For exploring the thermodynamic parameters, (see supplementary materials) of the adsorption process of the selected metal ions on the GO-SA composite, free energy, entropy and enthalpy were calculated referring to Fig. 5c, the values of parameters were calculated and listed in Table 2. The negative values of ΔG indicates that the all metal studied were spontaneously sorbed on GO-SA surface. The positive values corresponding to ΔH and ΔS (Table 2) implies that the endothermic adsorption process as well as the affinity of the composite to adsorb the metal ion with increase in the randomness of the system.

Table 2
Thermodynamic parameters for sorption of Pb^{2+} , Cd^{2+} and Zn^{2+} ions.

T (K)	ΔG (kJ/mole)			ΔH (kJ/mole)			ΔS (J/mole/K)		
	Pb^{2+}	Cd^{2+}	Zn^{2+}	Pb^{2+}	Cd^{2+}	Zn^{2+}	Pb^{2+}	Cd^{2+}	Zn^{2+}
298	-25.70	-24.41	-22.68	11.52	24.27	10.18	124.89	163.35	110.26
308	-26.95	-26.04	-23.78						
318	-28.20	-27.68	-24.88						
328	-29.44	-29.31	-25.99						
338	-30.69	-30.94	-27.09						

3.4. Regeneration- Reuse

The adsorption-desorption behavior of the adsorbent material is a key index to estimate the suitability of the adsorbent employment in actual sewage management. In this regard, a definite amount of the adsorbent was utilized for adsorption of the studied metals ions. After achieving equilibrium, the supernatants were separated and the %R were calculated. The GO-SA-M material was washed by solutions of 5.0 mL (0.5 M HCl) to remove all sorbed metal ions and regenerated. The composite was treated by as 5.0 mL (0.5 M NaOH) and final 5 mL (distilled H₂O) to perform the next use. As presented in

Fig. 5d, GO-SA showed excellent reproducibility for adsorbing of (Pb^{2+} , Cd^{2+} , Zn^{2+}) over five adsorption/desorption experimental runs. However, we observed that the removal efficiency of the regenerated composite of all studied metal ions was enhanced compared to the original composite. This is may be attributed to the regeneration solutions activated extra binding site and make them available for adsorption more metal ions.

3.5. Selectivity behavior towards Pb^{2+}

Based on the adsorption experimental results, the selectivity of Pb^{2+} adsorption by GO-SA was assessed relative to Cd^{2+} and Zn^{2+} . These metal ions could be considered as competitive species during the adsorption of Pb^{2+} from acidic aqueous solution at $\text{pH} = 1$ containing Cd^{2+} and Zn^{2+} , as present in Fig. 5e. The affinity of the GO-SA adsorbent towards the tested metal ion in order Pb^{2+} (57.21%) > Cd^{2+} (10.65%) > Zn^{2+} (8.21%) from initial metal ion concentration 600, 100 and 100 mg L^{-1} , respectively. The previous results implies that the GO-SA own an excellent ability to select removal of Pb^{2+} from metal ion mixture solution at $\text{pH} = 1$.

3.6. Comparison with other adsorbent material

The efficiency of sodium alginate modified graphene oxide as an adsorbent for removal of Pb^{2+} , Cd^{2+} and Zn^{2+} from aqueous solution was estimated by comparing the adsorption capacity with other adsorbent materials in the literature presented in Table 3. The data showed that sodium alginate modified graphene oxide have a highly feasible capacity for the sorption of studied metal ions compared with other adsorbent materials. Therefore, sodium alginate modified graphene oxide could be used as a highly effective adsorbent for removal these metals from aqueous solutions.

Table 3
Adsorption capacities of different adsorbents of Pb²⁺, Cd²⁺ and Zn²⁺

Adsorbate	Adsorbent	q _o (mg/g)	Ref.
Pb ²⁺	(GO–Zr-P) nanocomposite	363.42	(Pourbeyram, 2016)
	zinc oxide/graphene oxide nanocomposite	418.78	(Ahmed et al., 2020)
	rGO-Fe0/Fe3O4-PEI nanocomposite	60.24	(Bagheri et al., 2020)
	graphene oxide-terminated hyperbranched amino polymer-carboxymethyl cellulose ternary nanocomposite	152.86	(Kong et al., 2020)
	oily sludge pyrolysis residue	140.65	(Yang et al., 2020)
	Graphene Oxide/Sodium Alginate/1, 2-Propanediamine composite membrane	189.25	(Yu et al., 2021)
	Sodium alginate/graphene oxide aerogel	267.4	(Jiao et al., 2016)
	3D porous graphene/lignin/sodium alginate composite	226.24	(Zhou et al., 2018)
	a porous calcium alginate/graphene oxide composite aerogel	368.2	(Pan et al., 2018)
	EDTA-GO	508.4	(Cui et al., 2015)
	Functionalized graphene (GNSPF6)	406.4	(Deng et al., 2010)
	GO	35.6	(Lee & Yang, 2012)
	GO–TiO ₂	65.6	(Lee & Yang, 2012)
	GO-SA	887.21	This work
Cd ²⁺	rGO-Fe0/Fe3O4-PEI nanocomposite	54.47	(Bagheri et al., 2020)
	oily sludge pyrolysis residue	106.16	(Yang et al., 2020)
	3D porous graphene/lignin/sodium alginate composite	79.88	(Zhou et al., 2018)
	Functionalized graphene (GNSPF6)	73.42	(Deng et al., 2010)
	GO	14.9	(Lee & Yang, 2012)
	GO–TiO ₂	72.8	(Lee & Yang, 2012)

Adsorbate	Adsorbent	q_0 (mg/g)	Ref.
Zn^{2+}	RGO–Fe(0)/Fe ₃ O ₄	1.91	(Bhunia et al., 2012)
	GO-SA	139.62	This work
	GO	30.1	(Lee & Yang, 2012)
	GO–TiO ₂	88.9	(Lee & Yang, 2012)
	Mango peel	28.2	(Iqbal et al., 2009)
	magnetite (MG)	33.95	(Abdus Salam and Adekola,2018)
	baobab fruit shell (BB)	29.20	(Abdus Salam and Adekola,2018)
	magnetite–baobab composite (MB)	38.25	(Abdus Salam and Adekola,2018)
	GO-SA	161.25	This work

4. Conclusion

In this paper we succeed to modify the GO with highly active biopolymer (SA) through mild condition. The as-synthesised composite (GO-SA) was characterized by TEM, SEM, FTIR, Raman, TGA and EDX. The activity of the GO-SA was evaluated using batch adsorption process of different heavy metal. According to Langmuir isotherm model the GO-SA achieve maximum adsorption capacity 887.21, 161.25 and 139.62 mg g⁻¹ for Pb²⁺, Zn²⁺ and Cd²⁺, respectively. Moreover, the GO-SA represent excellent regeneration ability with enhancing the removal efficiency over five reuse runs. In addition, the GO-SA composite can selective adsorb Pb (II) from low acid of pH=1 in presence of Cd(II) and Zn (II).

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Figures

Figure 1

TEM (a-c) and SEM (d-h) images for alginate modified graphene oxide (GO-SA).

Figure 2

a) FT-IR spectrum of GO and GO-SA composite and b) Thermogravimetric diagram of GO and GO-SA composite.

Figure 3

EDS and EDX analysis for GO-SA composite, GO-SA-Pb complex, GO-SA-Cd complex and GO-SA-Zn complex

Figure 4

Effect of a) contact time on removal percent (%R), b) 2nd order kinetic model of Pb^{2+} ($[Pb^{2+}] = 200 \text{ mg L}^{-1}$, Dose = 2.8 mg, $v = 5 \text{ ml}$, $pH = 5$, $T = 25 \text{ }^{\circ}\text{C}$) Cd^{2+} ($[Cd^{2+}] = 50 \text{ mg L}^{-1}$, Dose = 2.8 mg, $v = 5 \text{ ml}$, $pH = 5$, $T = 25 \text{ }^{\circ}\text{C}$) and Zn^{2+} ($[Zn^{2+}] = 50 \text{ mg L}^{-1}$, Dose = 2.8 mg, $v = 5 \text{ ml}$, $pH = 5$, $T = 25 \text{ }^{\circ}\text{C}$) from aqueous media. C) Effect of the initial solution pH on the removal percentage (% R) from aqueous solution of Pb^{2+} ($t = 1.0 \text{ min}$, $[Pb^{2+}] = 200 \text{ mg L}^{-1}$, Dose = 2.8 mg, $V = 5 \text{ ml}$, $T = 25 \text{ }^{\circ}\text{C}$) Cd^{2+} ($t = 1.0 \text{ min}$ $[Cu^{2+}] = 50 \text{ mg L}^{-1}$, Dose = 2.8 mg, $V = 5 \text{ ml}$, $T = 25 \text{ }^{\circ}\text{C}$) and Zn^{2+} ($t = 1 \text{ min}$, $[Zn^{2+}] = 50 \text{ mg L}^{-1}$, Dose = 2.8 mg, $V = 5 \text{ ml}$, $T = 25 \text{ }^{\circ}\text{C}$) from aqueous media.

d) metal ion concentration on removal percent, e) Langmuir isotherm plot, f) Freundlich isotherm plot of Pb^{2+} ($t = 1 \text{ min}$, Dose = 2.8 mg, $v = 5 \text{ ml}$, $pH = 4$, $T = 25 \text{ }^{\circ}\text{C}$) Cd^{2+} ($t = 1 \text{ min}$, Dose = 2.8 mg, $v = 5 \text{ ml}$, $pH = 7$, $T = 25 \text{ }^{\circ}\text{C}$) and Zn^{2+} ($t = 1 \text{ min}$, Dose = 2.8 mg, $v = 5 \text{ ml}$, $pH = 5$, $T = 25 \text{ }^{\circ}\text{C}$) from aqueous solution.

Figure 5

Effect of a) v/m ratio on sorption of Pb^{2+} ($t = 1$ min, $[Pb] = 600\ mg\ L^{-1}$, $v = 5$ ml, $pH = 4$, $T = 25^\circ C$), Cd^{2+} ($t = 1$ min, $[Cd] = 100\ mg\ L^{-1}$, $v = 5$ ml, $pH = 7$, $T = 25^\circ C$) and Zn^{2+} ($t = 1$ min, $[Zn] = 100\ mg\ L^{-1}$, $v = 5$ ml, $pH = 5$, $T = 25^\circ C$) from aqueous media.

b) temperature on the removal percent and c) thermodynamic parameters of Pb^{2+} ($t = 1$ min, $[Pb] = 600\ mg\ L^{-1}$, Dose = 11.2 mg, $v = 5$ ml, $pH = 4$, $T = 25^\circ C$) Cd^{2+} ($t = 1$ min, $[Cd] = 100\ mg\ L^{-1}$, Dose = 11.2 mg, $v = 5$ ml, $pH = 7$, $T = 25^\circ C$) and Zn^{2+} ($t = 1$ min, $[Zn] = 100\ mg\ L^{-1}$, Dose = 11.2 mg, $v = 5$ ml, $pH = 5$, $T = 25^\circ C$) from aqueous solution.

d) the number of the re-use cycles of the GO-C on the removal percentage of Pb^{2+} ($t = 1.0$ min, $[Pb] = 600\ mg/L$, Dose = 14 mg, $V = 5.0$ ml, $pH = 4$, $T = 25^\circ C$), Cd^{2+} ($[Cd] = 100\ mg/L$, Dose = 14 mg, $V = 5.0$ ml, $pH = 7$, $T = 25^\circ C$) and Zn^{2+} ($[Zn] = 100\ mg/L$, Dose = 14 mg, $V = 5.0$ ml, $pH = 5$, $T = 25^\circ C$) from aqueous media.

e) Selective removal of Pb (II) from metal ion mixture from aqueous solution. [Mz+] ([Pb] = 600.0 mg/L, [Cd] = 100.0 mg/L, [Zn] = 100.0 mg/L, v = 10.0 ml, m = 0.014 gm, pH = 1.0, t = 1.0 min, T = 25 °C

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