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Fe_3O_4/rice husk/ polypyrrole nanocomposite as a sorbent for efficient lead removal

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Abstract
Heavy metal pollution, particularly lead (Pb^{2+}), poses a significant threat to the environment and human health. In this study, Fe_3O_4/rice husk/ polypyrrole (Fe_3O_4/Rh/PPy) nanocomposite was synthesized and used for removal of lead from aqueous solutions. In the first, Fe_3O_4 magnetic nanoparticle was prepared through a simple and one step method and then polypyrrole (PPy) was synthesised chemically on the surface of nanoparticle. $FeCl_3$ was used as chemical oxidants for oxidation of pyrrole to PPy. The new nanocomposite was characterized by FT-IR, SEM and Dynamic Light Scattering method. The effect of pH, contact time, adsorbent dose, initial concentration, ionic strength and the effect of temperature on the adsorption was checked out in a batch process. Optimal adsorption conditions were determined at pH~ 7, 0.04 g dosage, 45 min contact time, and 30 ppm initial lead concentration, yielding a maximum lead removal efficiency of 99.7 %. Using the equilibrium constants obtained at different temperatures, the thermodynamic parameters were calculated and showed that the uptake of lead is spontaneous and exothermic. The maximum adsorption capacity by using Langmuir equation was calculated 85.47 mg/g. The kinetic data followed by Pseudo second.

Keywords: Fe_3O_4/rice husk/ polypyrrole nanocomposite, Removal, Lead

1. INTRODUCTION

Water is the most essential resource for sustaining life on Earth. As a result, providing clean water is crucial. Due to rapid urbanization, population growth, economic expansion, and industrialization, water contamination is increasing globally. Industrial wastewater has emerged as a significant environmental concern. Heavy metal contamination is one of the most serious environmental problems caused by inappropriate waste disposal from untreated industries. Lead is of particular concern, as metal finishing, electroplating, battery manufacturing, painting, pigments, and other processes are the primary sources of lead [1].

Lead ions are specifically utilized in many industrial processes, including the manufacturing of lead-acid batteries, ammunition, and industrial and medical uses such textile dyeing and metal mining and processing [2,3]. However, a number of health issues, including as cancer, neurological damage, anemia, angioedema, stomachaches, and muscle weakness, can be brought on by high amounts of lead ions in drinking water [4-6]. The World Health Organization (WHO) suggests that drinking water has a maximum lead ions content of 0.01 mg/L[7-10]. The result demonstrated that any trace of lead ion in water greater than 0.01 mg/L needs to be removed [11-13]. Several technologies are in use for lead removal. These technologies include ion exchange, chemical precipitation, electrolysis, membrane separation and adsorption. Amongst these, adsorption is more reliable and promising owing to its efficiency, local availability of adsorbents, operational simplicity, cost effectiveness and regeneration potential of the adsorbents [14-17]. Nanoparticles are a new group of materials that emphasiszed for the environmental treatment due to their unique properties such as large surface area and pore size, small particle size, surface charge, magnetic property, thermal stability, chemical inertness and biocompatibility [18].

Nanotechnology has emerged as a powerful tool for heavy metal remediation, offering efficient and sustainable solutions for removing toxic metals. Several studies have reported the removal of lead from water and wastewater using nanosorbents [19-26]. All of these studies revealed that novel applications of nanomaterials for the removal of lead from water. However, so far, there are no reports showing whether magnetic (Fe_3O_4/Rh/PPy)

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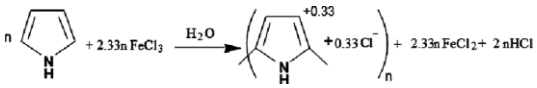
nanocomposite can be used for removal of lead. PPy and its derivatives can be readily synthesized chemically on the surface of different solid substrates such as fibers, glass, cloth, paper as powders, disperse or colloid. Iron (III) chloride has been found to be the best chemical oxidant and water is the best solvent for chemical polymerization [27]. Polymerisation of PPy with ferric chloride as oxidant is given in Fig. 1.



2. EXPERIMENTAL

2.1. Materials and methods

The chemicals used were all AR grade. $Fe(NO_3)_3.9H_2O$ (99% wt), rice husk (80-mesh), $Pb(NO_3)_2$ (99% wt), NaOH (99.5% wt), Na_2SO_4 (95% wt), NaCl (99% wt), Ammonia solution 25% , $FeCl_3$ and Pyrrole (all from Merck Co, Germany). Standard solutions of lead ions (1000 ppm) were prepared by dissolving of $Pb(NO_3)_2$ salt in the distilled water. A SensAA GBC (Dandenong, Australia) atomic absorption spectrometer was used for measurement of lead in air-acetylene flame. A mechanical shaker KS 130 basic (Deutschland, Germany) having speed control and timer was used for batch experiments. A Metrohm pH meter (Herisau, Switzerland) was employed for pH measurements. The concentrations of the lead solutions were obtained using data from a standard calibration curve.

2.2 Preparation of Fe_3O_4/Rh

Rice husk(Rh) was supplied from the local market in Kerman and were ground with a household blender until very smooth fine powder was obtained. The particles were passed through an 80-mesh sieve, washed with distilled water and dried in 25 °C. In order to prepare magnetic Fe_3O_4/Rh, 3.0 g rice husk as a reducing agent was added to 100 ml water containing 4.0 g $Fe_3O_4.9H_2O$ and the mixture is stirred vigorously for 1 h at 90 °C for reduction of Fe^{3+} ions to Fe^{2+} ions. Then the pH value of the solution was adjusted to about 10 by gradually addition of ammonia 25% solution with continuing vigorous stirring on the heater stirrer. The freshly formed Fe_3O_4/Rh NPs were decanted with the help of a handheld magnet. Subsequently, the material was washed with deionized water, dried in at 60 °C and stored for subsequent modification.

2.3. Preparation of Fe_3O_4/Rh/PPy nanocomposite

Pyrrole distilled before polymerisation. Polymerisation was carried out in aqueous solution. In order to prepare Fe_3O_4/Rh/PPy nanocomposite(sorbent), 3.0 g of Fe_3O_4/Rh was dispersed in 80 mL distilled water for 20 min. Then, 6.0 g $FeCl_3$ and 0.8 mL freshly distilled pyrrole solution was added to the suspension, with continuing stirring at room temperature on magnetic stirring. After 3 h, the suspension decanted with a magnet. Subsequently, the sorbent was washed with deionized water, dried in at 60 °C for 12 h and then saved for adsorption studies.

2.4. Characterization

The functional groups of Fe_3O_4/Rh/PPy nanocomposite were identified by FT-IR spectrum in the wave number range of 500–4000 cm^{-1} by using pressed KBr pellets. The microstructure of the prepared nanocomposite was observed by Scanning Electron Microscopy (SEM) (Cam Scan MV2300). The Dynamic Light Scattering (DLS) test was also employed to specify the average particle size, also.

2.5 Adsorption method

Adsorption experiments were conducted in batch system at room temperature by shaking 0.04 g of the sorbent with 25 ml of the aqueous solutions 30 ppm of lead in stoppered pyrex glass flask, at a fixed temperature. The initial pH of the solutions was adjusted with diluted HNO_3 or NaOH 0.1 M solution and the shaking speed was 200 rpm for 45 min. At the end of the adsorption period, the sorbent was separated from the suspension with magnetic field out and the lead concentration was determined using FAAS.

The amount of Pb(II) ions adsorbed and percentage of removal of Pb(II) ions (Re%) was calculated using Equations (1) and (2).

$$q_e = V(C_0 - C_e)/M \quad (1)$$

$$Re\% = [(C_0\text{-}Ce)/C_0] \times 100 \quad (2)$$

As q_e is the amount of adsorbed Pb(II) ion per gram of the adsorbent, C_0 is initial concentration and C_e is the equilibrium concentration of the Pb(II) (mg L^{-1}) that is obtained from calibration curve and M is the mass of nanocomposite (g).

3. RESULTS AND DISCUSSION

3.1 Characterization of the sorbent

Fe_3O_4/Rh/PPy nanocomposite was characterized employing various techniques including FT-IR, SEM and DLS. FT-IR spectrum of the sorbent is shown in Fig. 2. The peaks around 1531 and 1455 cm^{-1} correspond to the symmetric and asymmetric C-N and C-C ring-stretching modes of polypyrrole, respectively. Absorption band at 3406 cm^{-1} is due to the N-H group of polypyrrole. These peaks indicate the doping state of PPy on the nano particle magnetic. SEM analysis was done to identify

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the surface and morphology of Fe_3O_4/Rh/PPy nanocomposite· SEM image of Fe_3O_4/Rh showed (Fig. 3) that Fe_3O_4 nanoparticles incorporate on the surface of rice husk as agglomerated form and other places also. These Fe_3O_4 nps are spherical in shape and prepared using rice husk powder as reducing agent.

The Dynamic Light Scattering (DLS) test was also employed to specify the average particle size where its results are available in Fig. 4. Accordingly, the average particles size after incorporating of the PPy was less than 100 nm.

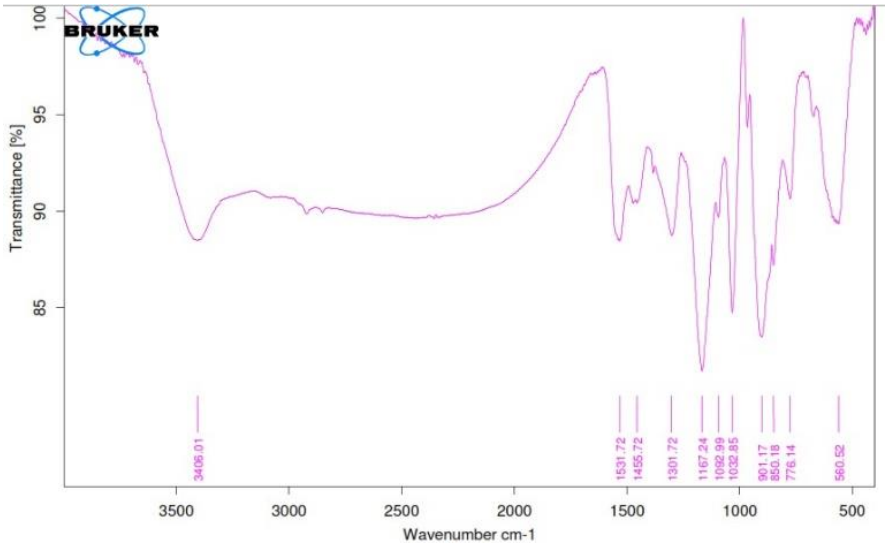

Fig. 2. FT-IR spectrum of Fe_3O_4/Rh/PPy nanocomposite.

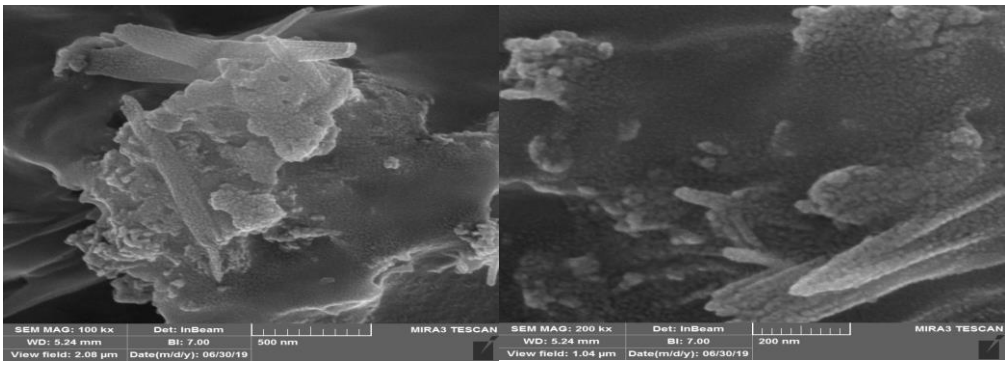

Fig. 3. SEM images of Fe_3O_4/Rh/PPy nanocomposite.

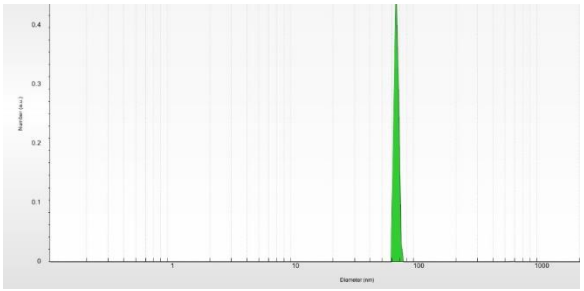

Fig. 4. DLS spectrum of nanocomposite.

3 2. Effect of pH

pH is a deciding factor for any kind of metal sorption on sorbents in an aqueous solution. The surface properties of sorbents, ionic state of functional groups and species of metals are dependent on pH condition. To find out the proper water condition with maximum metals adsorption pH dependent experiments are needed. The affinity of adsorbent functional sites for lead ions depends on the initial pH of the solution. Lead occurs as Pb^{2+}, $Pb(OH)^{+}$ and $Pb(OH)_2$ species in deionized water. The effect of pH on adsorption of

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Pb(II) ions was studied over a pH range of 1-8 at room temperature. According to the results depicted in Fig. 5, in acidic range, functional groups on the sorbent protonated and caused electrostatic repulsion to Pb^{2+} ions. The percentage of removal of lead increased with increasing pH, due to electrostatic attractions occur between cationic Pb^{2+} ions and anionic adsorbent surface. At high pHs, Precipitation occurs due to forming lead hydroxide, therefore, the experiments were performed at pH ~7.

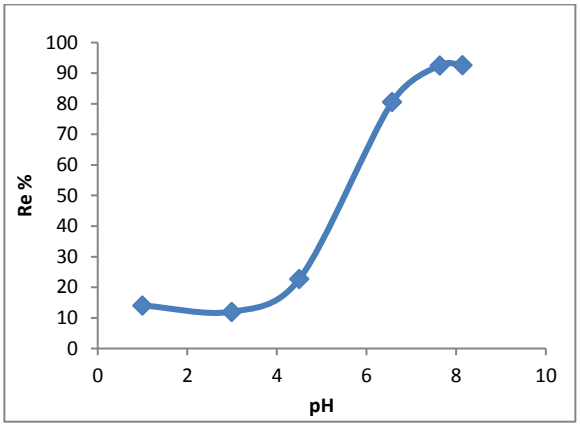

Fig. 5. Effect of pH on the removal of lead.

3.3. Effect of sorbent dose

Effect of adsorbent dosage on the uptake of lead was studied and is shown in Fig. 6. The percent removal of lead increased from 54.9% to 97.5% with an increase in adsorbent amount. The removal of lead at m> 0.04 g remains almost constant. An increase in the removal with the adsorbent dosage can be attributed to greater surface area and the availability of more adsorption sites. At m< 0.04 g, the adsorbent surface becomes saturated with lead and the residual concentration in the solution is large. Thus, the dose of the adsorbent was fixed to 0.04 g for the subsequent adsorption experiment.

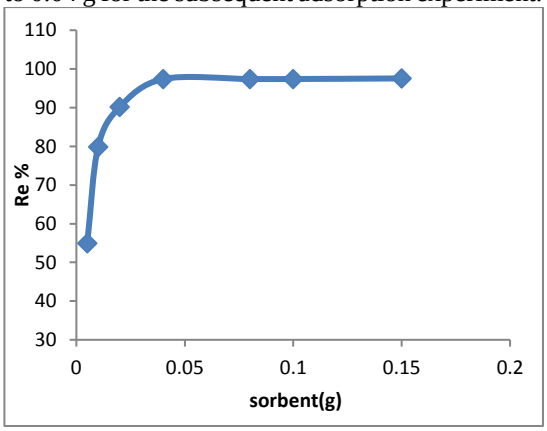

Fig. 6. Effect of adsorbent dosage on the removal of lead.

3.4. Effect of contact time

Another important characteristic determining whether or not the use of a given adsorbent is feasible for a particular task is the time. The effect of contact time is depicted in Fig. 7. The experiments were performed by varying contact time (1–120 min.) with initial Pb(II) concentration 30mg/L, keeping all other parameters constant. The percent removal increased from 60.7% to 99.1% onto nanocomposite with increasing time. Pb(II) removal increased continuously upto 45 min and then removal efficiency remains constant. This may be attributed that initially, the active sites for Pb(II) ions are available in excess, but after time equilibrium attained due to the saturation of the sorbent active sites. 45 min was selected for more experiments.

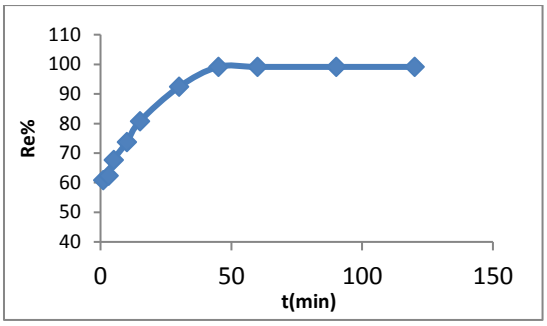

Fig. 7. Effect of contact time on lead adsorption.

3.5. Effect of temperature

The effect of temperature on the adsorption of lead was studied by varying temperature in the range of 0–45°C. The lead removal decreased with increase in temperature illustrating that adsorption of lead on the sorbent is exothermic. The Gibbs free energy change, ΔG^0, is fundamental criterion of spontaneity. The sorption process of lead can be summarized by the following reversible process. The equilibrium constant (K_c) is defined as:

$$K_c=(C_0- C_e)/C_e \quad (3)$$

where C_e is the concentration of lead at equilibrium. The K_c value is used in the following equation to determine the ΔG^0 of sorption.

$$\Delta G^0= -RT\ln K_c \quad (4)$$

The enthalpy (ΔH^0) and entropy (ΔS^0) can be calculated from the slope and intercept of Vant Hoff equation of ΔG^0 versus T.

$$\Delta G^0= \Delta H^0-T\,\Delta S^0 \quad (5)$$

R the gas constant (8.314 J $mol^{-1}K^{-1}$) and T is the absolute temperature (K). The obtained ΔG^0 values and the adsorption thermodynamic parameters are given in Table 1. The positive value ΔS^0 shows the increase in degree of freedom or increase the disorder of adsorption lead on the sorbent and the negative value ΔH^0 indicates that adsorption process is exothermic.

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Table 1. Thermodynamic Parameters of lead.

T (K)	ΔG^0 (J mol^{-1})	ΔS^0 (J mol^{-1} K^{-1})	ΔH^0 (kJ mol^{-1})
273	-11171.3		
283	-10723.6	56.15	-26.66
296	-10282.4		
303	-9828.41		
308	-9431.34		
313	-9114.12		
318	-8494.71		

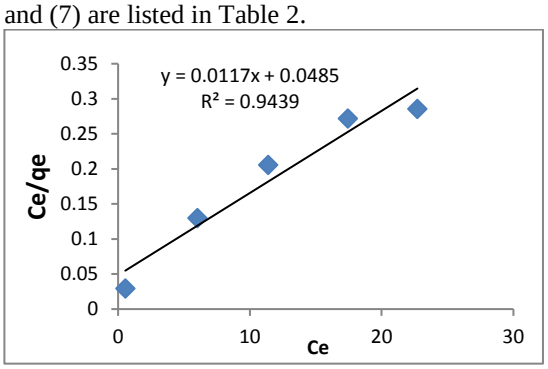
3.6. Equilibrium adsorption isotherms

The adsorption isotherms of lead on Fe_3O_4/Rh/PPy were examined in the concentration range 30-150 mg. L^{-1} using Langmuir and Freundlich models to understand the adsorption mechanism. Linear forms of Langmuir and Freundlich isotherms are given in Eqs. (6) and (7).

$$C_e/q_e = (1/Qb) + (1/Q)\, C_e \quad (6)$$

$$\text{Log } q_e = \log K_f + 1/n \log C_e \quad (7)$$

q_e is the amount of adsorbed ion per gram of the adsorbent, b is Langmuir constant and *Q* is adsorption capacity expressed in mg g^{-1}, n is the Freundlich constant, and K_f is the adsorption coefficient. The results showed the Freundlich model yields somewhat better fit for adsorption of lead (Fig. 8). All the calculated parameters from intercepted and slope of plots according to Eqs. (6) and (7) are listed in Table 2.



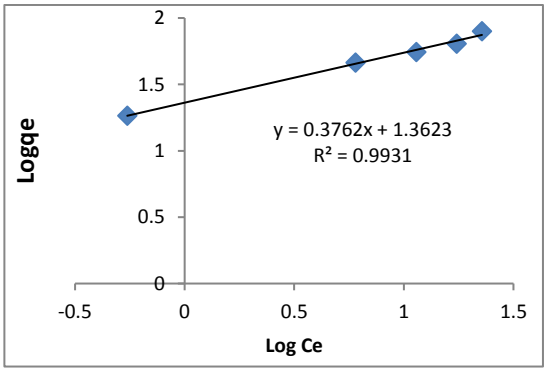


Fig. 8. Langmuir and freundlich isotherms of lead.

Table 2. Langmuir and freundlich isotherm constants for lead.

T (K)	Langmuir isotherm			Freundlich isotherm		
	Q(mg g^{-1})	b(L mg^{-1})	R^2	K_f	1/n	R^2
298	85.47	0.24	0.94	23	0.37	0.99

3.7. Effect of ionic strength

The effect of various amounts of NaCl and Na_2SO_4 on the sorption of lead on the nanocomposite was examined. It was seen that the there was no significant decrease in the percent removal efficiency. As, even at high concentration of the salt, Fe_3O_4/Rh/PPy nanocomposite still has big percent removal efficiency and can be used to efficiency remove lead from aqueous solutions.

3.8. Adsorption kinetics

The adsorption kinetics described by the relationship between lead uptake and contact time that is discussed by Pseudo-First order and Pseudo-Second order, Eqs. (8,9).

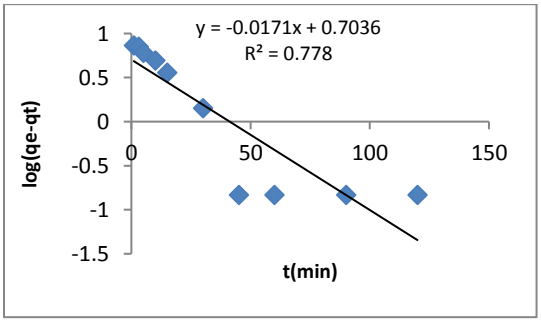
The Pseudo- First order equation is:

$$\log(q_e - q_t) = \log q_e - (k_1/2.303)\, t \quad (8)$$

And the Pseudo-Second order equation is:

$$(t/q_t) = (1/k_2 \,.\, q_e^2) + t/q_e \quad (9)$$

where q_t is the amount of lead adsorbed at 't' time (mg/g), t is the contact time (min), q_e is the amount of lead adsorbed at equilibrium (mg/g), k_1 is the rate constant of Pseudo-First order reaction (min^{-1}) and k_2 is the rate constant of Pseudo-Second-order reaction (g mg^{-1} min^{-1}). The slope and intercept of plot of log (q_e-q_t) against time (min) were used to determine the First order rate constant k_1. It was observed that correlation coefficient (R^2) was low and experimental data are not fitted well for Pseudo-First order reaction. Hence, the adsorption mechanism cannot be well described by Pseudo-First order kinetics. The Pseudo-Second order rate constant k_2 was calculated from the slope and intercept of the plot t/q_t against time (Fig. 9). The calculated q_e ($q_{e,cal}$) was in good agreement with experimental q_e ($q_{e,exp}$) with correlation coefficient high These results confirm the well fitting of Pseudo-Second order model for adsorption of lead on to the (Fe_3O_4/Rh/PPy) nanocomposite.



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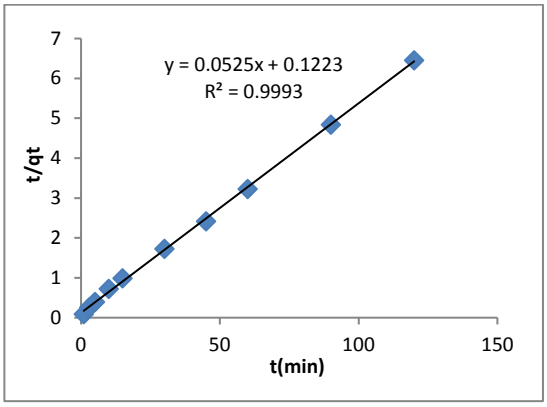

Fig. 9. Pseudo-First order and Pseudo-Second order plot for lead adsorption

4. CONCLUSION

In the present paper, a novel sorbent, magnetic Fe_3O_4/Rh/PPy nanocomposite, was synthesized and used to remove lead ions efficiently from aqueous solutions. In the first, magnetic nanoparticles were synthesized using rice husk as the reducing agent and then the nanocomposite prepared with incorporation PPy. The nanocomposite exhibited high adsorption and purification efficiency, achieving 99.7% removal of lead ions at pH~7. The results revealed that adsorption time and pH value are significant factors. The optimal conditions were: contact time of 45 min, a pH~7, dose of sorbent, 0.04g based on the results. The removal of lead ions using (Fe_3O_4/Rh/PPy) nanocomposite proved great potential as an effective sorbent for lead removal from aqueous solutions.

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نانوکامپوزیت پلی پیرول/ سبوس برنج/ Fe_3O_4 بعنوان یک جاذب برای حذف موثر سرب

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چکیده

آلودگی عناصر سنگین خصوصا سرب رفتار ویژه ای برروی سلامتی انسان و محیط زیست دارد. در این مطالعه نانوکامپوزیت پلی پیرول/ سبوس برنج/ Fe_3O_4 ($Fe_3O_4/Rh/ppy$) سنتز و برای حذف سرب از محیطهای آبی استفاده شد. در ابتدا نانوذره مغناطیسی Fe_3O_4 با یک روش ساده و یک مرحله ای آماده و سپس پلی پیرول برای کارایی بیشتر جاذب به طریق شیمیایی برروی سطح نانوذره قرار گرفت. $FeCl_3$ بعنوان اکسید کننده برای اکسیداسیون پیرول استفاده شد. نانوکامپوزیت جدید توسط FT-IR , SEM و DLS مورد بررسی قرار گرفت. اثر pH ، زمان تماس، مقدار جاذب، غلظت سرب، قدرت یونی و اثردما برروی جذب بروش ناپیوسته بررسی شد . بهترین شرایط در pH~ 7 ، مقدار جاذب ۰/۰۴ گرم ، زمان تماس ۴۵ دقیقه، غلظت اولیه ۳۰ ppm تعیین گردید که منجر به بیشترین جذب سرب با درصد حذف ۹۹/۷ گردید. با استفاده از ثابتهای تعادلی بدست آمده در دماهای مختلف، پارامترهای ترمودینامیکی محاسبه شدندکه بیانگر خودبخودی و گرمازا بودن فرآیند جذب سرب می باشد. حداکثر ظرفیت جذب با استفاده ازمعادله لانگمویر۸۵/۴۷ میلی گرم بر گرم محاسبه شد. داده های سینتیکی با شبه مرتبه دوم مطابقت داشتند.

کلید واژه ها: نانوکامپوزیت پلی پیرول/ سبوس برنج/ Fe_3O_4 ، حذف ، سرب

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