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Comparative study for Pb2+ adsorption from simulated wastewater of battery manufacture on activated carbon prepared from rice husk with different activation agents

Murtadha Hamid Khashan* and Abbas Khalaf Mohammad

Department of Chemical Engineering, College of Engineering, University of Al-Qadisiyah, Iraq

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ABSTRACT

This study contains a lab-scale investigation into the feasibility of applying an adsorption technique to treat wastewater polluted with lead metal simulated to that exhaust from a battery manufactory. Rice husks have been prepared for use in three forms; natural rice husks without any activation, activated carbon of rice husks pretreated with Sulfuric acid H2SO4, and the other pretreated with Potassium hydroxide KOH. Activated carbon using KOH provided the best condition for the removal of lead than carbon activated using H2SO4 and natural rice husks. The BET surface area and surface morphology by FESEM analysis were characterized. The values of surface area were (20, 561, 722) m2/g for natural rice husk, activated carbon pretreated with sulfuric acid, and potassium hydroxide respectively. The adsorption process was studied using Pb2+ at temperatures of (25-40)° C and for a concentration range of (20-100) mg/l. The maximum removal ratios of lead Pb2+ for natural rice husks and sulfuric acid or hydroxide potassium activation after 1h were 51.21%, 92.3%, and 97.25%, respectively. The maximum adsorption capacity of lead Pb2+ onto natural rice husks, rice husks pretreated with H2SO4 or KOH, and into activated carbons was (37, 86.2 and 94.6) mg/g respectively, at a concentration of 100 ppm and at 25oC. The equilibrium adsorption curves were obtained for Pb2+ and the data was fairly well fitted with Freundlich-isotherm with a confidence level of 0.99. The kinetic of adsorption was studied by using two kinetic models, pseudo first order and pseudo second order. The results showed the rapid increase in the rate of adsorption at the initial until equilibrium achieved. Pseudo second order model was represented the data very well with confidence level 0.99.

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1. Introduction

Every year, large amounts of wastewater containing heavy metals like lead (Pb2+) are discharged into the environment [1]. Pb2+ has negative health and environmental consequences. When wastewater containing Pb2+ is discharged directly into surface water, seawater, or groundwater, it has a significant impact on the lives of organisms [2]. Heavy metals like lead, iron, zinc, nickel, mercury, silver, iron, chromium, gold, arsenic, cobalt, Molybdenum, aluminum, and manganese can be accumulate and absorbed in the human body, causing serious health issues [3]. Pb2+ has been linked to cancer, organ damage harm, and nervous system damage, among other health issues. Minerals such as lead, zinc, and iron are

* Corresponding author. E-mail address: mortadhahamid@gmail.com (Murtadha Hamid Khashan)

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regarded to be particularly poisonous. Lead (Pb2+) has been identified as a major source of damage to the nervous and immunological system [4]. Therefore, these pollutants must be reduced to an acceptable level before disposal to the environment and according to the limits permitted by the international regulations and laws of the World Health Organization (WHO) [5]. There are many industries responsible for the disposal of organic or non-organic pollutants through the discharge of wastewater directly into the environment, such as Battery manufacturing, pulp, food, paper, mining, fertilizer, pharmaceutical, and petrochemical, in addition to the refineries which considered as most used industry locally [6].

This study focuses on lead Pb2+ heavy metal that discharge from Battery manufacturing which causes harmful effects on the environment and people's health. There are many methods used for this purpose, including chemical precipitation, membrane filtration, ion exchange, and electrochemical treatment methods, in addition to adsorption (Bratby 2016). However, these methods have some limitations, and disadvantages such as low efficiency, high power consumption, and high quantity of sludge production[7]. Bio adsorbents have emerged as one of the promising heavy metal and metalloid removal solutions. Fungi, Plants, and algae are examples of biomass-derived adsorbents capable of adsorbing heavy metals from the solutions[8]. Due to functional groups such as imidazole, carboxyl, hydroxyl, amino, phosphate, sulfate, phenol, carbonyl, and amide, bio-adsorbents have an affinity for heavy metal ions to form metal complexes or chelates [9]. RH is a low-cost (agricultural by-product) bio adsorbent that has been extensively explored for the removal of numerous heavy metals from both groundwater and surface water, including Pb, Cd, Zn, Ni, and As [10].

The success of these techniques depends on the development of an efficient adsorbent. According to literature, these adsorbents may be natural, industrial, agricultural, domestic waste, or by-products [11]. Decontamination of lead Pb2+ heavy metal from wastewater by the pretreated and carbonation rice husk is comparatively better than untreated one because untreated adsorbent can further generate more problems like high chemical oxygen demand, low adsorption capacity, biological oxygen demand, total organic carbon due to release of organic compounds in the plant (Rice husk) [12]. In this work, rice husks were investigated as a lowcost bio-sorbent alternative to prepare two forms of activated carbon using H2SO4 or KOH as an activating agent for Pb2+ metal removal from aqueous solutions, and the three husk forms were compared to adjust any form that has higher adsorption capacity and best removal percentage. The research also contains equilibrium isotherms of adsorption and the kinetics that could be useful in the design and future operations.

2. Experimental work

2.1. Materials

The nitrogen inert gas with a purity of 96% was used as inert medium gas in the process. Sulfuric acid (H2SO4) and Potassium hydroxide (KOH) with 97% of purity were used as a reagent. The polluted water Pb2+ was simulated according to the heavy metals residues from the battery facto. The lead Pb2+ ion aqueous stock solution was prepared by dissolving 0.16 g of Lead (II) nitrate (Pb (NO_3)₂), MW: 331.2 g/mol) in de-ionized water according to Eq. 1, [13].

$$W = \frac{C*M.wt*V}{A.wt*10^6} \tag{1}$$

Then the stock solution with h concentration 100 ppm of Pb2+ diluted to 20,40,60 and 80 ppm according to Eq. 2:

$$C1 * V1 = C2 * V2 \tag{2}$$

2.2. Rice husks preparation

Rice husks (RH) were gathered from a mill in the Iraqi city of Al-Shamiya. RH is cleaned with de-ionized water and dried at 100 oC to guarantee that all volatile compounds and water were vaporized. Then, 1kg of dried and cleaned rice husks was ground using the electrical grinder for 40 minutes. The powder produced sieve for 88 µm using apparatus of sieve analysis. Fig.1 shows a diagram of the experimental installation of a system for the production of activated carbon (AC). The vessel (reactor) as shown in fig.1 that was installed inside the combustion furnace it was made from a cylinder of carbon steel with a diameter of 10 cm and a height of 15 cm. It features a cover that can be opened and closed with two nuts. Several turns of stainless steel tube (1/4) inch in diameter are wrapped around the vessel, the lower end of the tube which is open inside the vessel, and the upper end is connected to the tube coming from the nitrogen cylinder. The regulator controlled the flow rate of nitrogen gas from the gas cylinder. The gas is then passed through a (1/4) inch pipe that is coiled around the vessel to ensure that the gas is preheated before flowing inside. The thermocouple and temperature recorder were attached to the effluent N2 gas line upside the furans [14].



Figure 1. A diagram for the activation system.

60 grams of RH powder was put inside a crucible which is made from graphite, mineral wool is fixed on top of it and this was used to keep the powder from getting out with the gas during the burning process. Then the graphite crucible is placed inner side of the vessel, which is located within the furnace. In the presence of nitrogen flow, the sample was precarbonized for 100 minutes at 280 °C in the furnace.

The produced carbons were cool at 25 °C, and then impregnate with (4Molar) of H2SO4 or KOH for one day. Then the mixture was filtered to separate the solid material from the activation liquid by using filter paper and dried the sample to 100°C for three hours in the oven. The filtrate material was placed inside a crucible that was put in a vessel for activation. The vessel's temperature was kept at 650 °C for 140 minutes under nitrogen flow. After that, the activated carbon was allowed to cool to 25°C. The product of AC was cleaned by repeatedly washing with de-ionized water until the pH reached 7, and then dried in an oven at 105°C for 10 hours.

2.3. Adsorption Experiment

The adsorption isotherms for lead Pb2+ metal on the different kinds of RH prepared forms were examined at (25, 30, 35, and 40) °C. (100) ml of Pb2+ solution at a specified concentration was mixed with (0.1) grams of different RH forms in a Florence flask. Then, the flask was put in a constant temperature hot plate magnetic stirrer. The tests of the adsorption equilibrium were done in two parts:

Firstly, the time of equilibration was determined by adding 0.1 grams of RH or its derivative to 100 ml of Pb2+ solutions at a concentration of 100 ppm. The temperature and concentration of the solutions that were selected as the lowest temperature (25°C) and the greatest concentration (100 milligrams per liter), respectively. These settings indicate the greatest adsorption quantity that results in the highest length of time required for equilibrium to be reached under all of the variables that will be tested in the experiments. The mixture was vigorously stirred under the influence of reflux at a constant temperature of 25 °C. The test was repeated at different time intervals of 10, 20, 60, 80, and 100 minutes, after that, the filtrate solution was tested using Atomic Absorption Spectroscopy AAS to measure the concentration after the adsorption process. After 1 hour, solution concentrations had stabilized to within a little range. For the adsorption process, this duration is indicative of the equilibrium period.

Secondly, a series of tests were carried out to investigate the impact that temperature and initial concentration had on the adsorption of Pb2+ from an aqueous solution onto the RH that had been previously prepared. To do this, a variety of concentrations were taken (20, 40, 60, 80, 100) ppm of Pb2+ at different temperatures (25, 30, 35, 40) \circ C with a contact time of 1 hour. Every single experiment was carried out by putting 0.1 gram of the adsorbent material (RH and its derivative) in a conical flask with (100 ml) of the solution of the heavy metal. After that, the adsorption procedure was carried out at the temperature that had been specified before, with continuous stirring for an hour. This procedure was repeated for each concentration at each temperature. At the end of the experiment's period, the solution was filtrated by the filtration apparatus and the filtrate was taken for analysis by AAS. Finally, the adsorbed amount QE (mg/g) of metals on the prepared rice husks was calculated by applying the material balance as in equation Eq. 3, [15]:

$$\mathbf{q}\mathbf{e} = ((\mathbf{C}\mathbf{0} - \mathbf{C}\mathbf{e}) * \mathbf{V})/\mathbf{W}$$
(3)

And the removal percentage was calculated from the following Eq. 4:

$$\mathbf{Removal} \% = (\mathbf{Co} - \mathbf{Ce}) / \mathbf{Co} * \mathbf{100}$$
(4)

2.4. Kinetics of Adsorption

The study of kinetics is very important to explain the rate of adsorption. 0.1 gram of the adsorbents (RH and its derivatives of activated carbon) was put in the adsorption flask with (100 ml) of the highest concentration range (100 ppm) of the Pb2+. The mixture was mixed under reflux at different temperatures (25, 30, 35, 40) °C. In each experiment, 5 ml samples of the solution were taken at different periods. These samples were analyzed for Pb2+ concentration. This routine was continued until the equilibrium state was reached. The equilibrium state was attended when the concentration of two successive samples are approximately equal.

3. Result and discussion

3.1 Characterization of Adsorbents (FESEM and BET)

The RH and its derivatives were identified by several test techniques. These checkup procedures include FESEM and BET.

3.1.1 Characterization of RH

FESEM was used to characterize the morphology of the prepared RH in the form of powder by obtaining structural photos and micrographs. Fig.(2) shows FESEM images of the prepared (RH) without any activation.



Figure 2. FESEM images for prepared RH, at 10 KX.

The analysis revealed an almost smooth surface with a small number of pores and cavities. It could be concluded from these that the content has a small surface area available for the adsorption process. These findings are in line with those found by several other studies [16].

The BET test of the prepared RH without activation was found to be with a BET surface area of $(20.5025 \text{ m}^2/\text{g})$ and an average pore diameter of (4V/A by BET) 8 .90682 nm. It could be expected that this low surface area has a significant diverse effect on the adsorption. The low surface area could be contributed to the high pore sizes. The large size of the pores provides a low surface area while the small size does the reverse. Furthermore, there is a large number of cellulose materials that close pores of small diameters.

3.1.2 Characterization of (RHAC)

Fig. (3) shows the FESEM result of the RH that was pre-treated with acidic solution H2SO4.



Figure 3. FESEM images for prepared RHAC, at 10 KX.

It is clearly shown in this figure that the pre-treatment with H2SO4 and carbonization causes the rice husks to become more amorphous and porous. RHAC has a porous structure with a random distribution of distinct pore diameters.

Furthermore, this figure shows that activated carbons made from RH waste have a heterogeneous rough surface with a random distribution of cracks and cavities, as well as porosity and that there is a considerable chance that the pores will attract and absorb heavy metal molecules. These findings are in line with those of several other studies [17], with different raw materials. The BET test of the activated carbon of rice husks that were pretreated with H2SO4 was observed to be with BET surface area equal to (561.7827 m²/g) and the average adsorption pore diameter of (4V/A by BET) 2 .62941 nm and it could be noticed that a higher surface area was achieved by acid activation than the surface area of rice husk without activation. Furthermore, the observed reduction of pore size indicates that the cellulose material was degraded by acidic activation. Hence, more fine pores were opened and more surface area was available for adsorption. These surfaces enhance the adsorbent for adsorption of adsorbates on the active sites for the heavy metal to be captured and absorbed via these pores.

3.1.3 Characterization of (RHBC)

Fig. (4) shows the FESEM test of the activated carbon of rice husk pretreated with basic solution KOH



Figure 4. FESEM images for prepared RHBC, at 10 KX.

It is clearly shown that the pre-treatment with KOH and the carbonization make the rice husks more porous and amorphous. RHBC has a more good porous structure, random arrangement, and different sizes of pore.

The RHBC has a heterogeneous rough surface with a random pattern of cracks and cavities with porosity, as shown in fig. (4.8), and there is a considerable chance that heavy metal molecules will be trapped and adsorbed into these pores.

These findings are in line with those of several other studies (Patel, Asatkar, et al. 2019).

The BET test of the activated carbon which was prepared from rice husks and pretreated with hydroxide potassium (KOH) has a BET surface area equal to (721.7400 m²/g), in addition, the adsorption average pore diameter was (4V/A by BET) equals to 3 .60725 nm. It could be noticed that the activation with KOH gave the best result and a higher surface area for the heavy metal to be adsorbed and trapped in these pores.

3.1.4 Comparison between Adsorbents

It was noticed clearly that the activation procedure resulted of the opening of confined pores and the reduction of carbonaceous material aggregates. The activation process clearly shows that the carbonized waste materials (rice husks) become well developed and porous due to the extremely hightemperature treatment and the action of chemical activation agents. The activated carbons that are prepared from rice husks RHBC and pretreated with basic KOH agent have more uniform surfaces of homogeneously arranged pattern with high porosity that is responsible for high surface area because the KOH is a strong base agent. While natural rice husks (RH) and acidic activated carbon RHAC that are activated with acid agents have lower porosity and less heterogeneous rough surface with a random distribution of fractures and voids. Therefore, the activated carbon prepared from rice husks (RHBC) with KOH as an activation agent gave a good pore diameter (2.62941 nm) and BET surface area (720.8293) in comparison with RH and RHAC. The smallest diameter and the bigger surface area make the RHBC the best for the adsorption of molecules of the heavy metal to be sucked into and adsorbed.

3.2 Equilibrium of adsorption

The combination of isothermal information analysis and isothermal characteristic modeling has made tremendous progress in finding the most appropriate model for design.

Figures (5-7) demonstrate the adsorption equilibrium isotherms for lead Pb2+ various temperatures on RH, RHAC and RHBC. These curves plotted at different temperature (25, 30, 35, 40) °C for a range of concentration of Pb2+ (20-100) ppm.

These figures clearly show that all curves for Pb+2 on RH and its derivate forms identical in nature and have the classical adsorption isotherm type 1 shape. This indicates that when the concentration of Pb2+ ions in the solution rises, so does the concentration on the adsorbent's surface. These results also show that the concentration in the solution and the concentration in the adsorption phase have a proportionate connection. However, this proportional declines and the adsorbed phase concentration reaches a constant value when the adsorbent reaches its maximum capacity at a large concentration of Pb2+ ions in the solution.



Figure 5. Adsorption equilibrium isotherms for Pb2+ ion adsorbed on the RH at temperatures range (25-40) Co.



Figure 6. Adsorption equilibrium isotherms for Pb2+ ion adsorbed on the RHAC at temperatures range (25-40) Co.



Figure 7. Adsorption equilibrium isotherms for Pb2+ ion adsorbed on the RHBC at temperatures range (25-40) Co.

The previous figures and table show that the maximum value of QE (mg/g) at 25°C decreased as the temperature increased, while the best value of adsorption was found for RHBC then followed by RHAC and RH, respectively. This conclusion is consistent with a prior study conducted by[18].

The experimental results were correlated to the most well-known adsorption isotherms that describe the adsorption of solutes from aqueous solutions onto RH, RHAC, and RHBC. These equations are known as Langmuir and Freundlich isotherms [19].

The Langmuir adsorption isotherm describes solute adsorption on a solid adsorbent that is limited to a single monolayer of solute molecules. This is based on the Langmuir adsorption assumption, which assumes that the solid adsorbent's surface has a homogeneous surface. On the other hand, the Langmuir adsorption isotherm equation could be represented mathematically as follows [20]:

$$qe = Qm KL * \frac{Ce}{1 + KL Ce}$$

Freundlich adsorption isotherm illustrates the multilayer adsorption of a solute onto the heterogeneous surface of a solid adsorbent. Mathematically, the equation for the Freundlich adsorption isotherm is as follows [21]:

$$qe = Kf Ce^{\frac{1}{n}}$$

The linear formulations of the Langmuir and Freundlich adsorption isotherm equations were as follows Eqs. 4 and 6, [22] :

Langmuir:
$$Ce/qe = Ce/qm + 1/(KL qm)$$
 (5)

Freundlich:
$$\log qe = \log Kf + \frac{1}{n} \log Ce$$
 (6)

According to equations (5) and (6), the Langmuir and Freundlich adsorption isotherms were plotted for adsorption of Pb2+ onto RH, RHAC RHBC in their linear forms in figures (8-13).



Figure 8. Langmuir adsorption isotherm model for Pb2+ ions adsorbed on RH .



Figure 9. Langmuir adsorption isotherm model for Pb2+ ions adsorbed on RHAC.



Figure 10. Langmuir adsorption isotherm model for Pb2+ ions adsorbed on RHAC



Figure 11. Freundlich adsorption isotherm model for Pb2+ ions adsorbed on RH at (25-40) Co.



Figure 12. Freundlich adsorption isotherm model for Pb2+ ions adsorbed on RHAC at (25-40) Co.



Figure 13. Freundlich adsorption isotherm model for Pb2+ ions adsorbed on RHBC at (25-40) Co.

The average values of confidence level (R2) are shown in table (1) for Langmuir and Freundlich isotherm models for the adsorption of Pb2+ on prepared RH and its activated forms. The comparison of (R2) values for the two isotherm models provide evidence that the Freundlich adsorption isotherm more accurately predicts the experimental results than the Langmuir adsorption isotherm. Therefore, it was concluded that the adsorption of Pb2+ on all forms of rice husks is limited to a multilayer and all forms of prepared rice husks have heterogeneous surface and every layer of these represent the adsorption as in a single layer. This conclusion is consistent with the findings of a prior study conducted by [23], who concluded that the adsorption of zinc heavy metals onto phosphoric acidactivated rice husk follows the Freundlich isotherm.

 Table 1. Average confidence interval levels (R2) for Langmuir and

 Freundlich models.

Adsorbents	R ² Langmuir model	R ² Freundlich model
RH	0.973	0.994
RHAC	0.979	0.993
RHBC	0.937	0.987

3.3 Kinetic of adsorption

Figures (14 to 16) represent the amount adsorbed Pb2+ on to different types of prepared RH and its activated carbon form as a function of the progression of time for the range of temperature (25-40) °C.



Figure 14. Rate of adsorption of Pb2+ on RH



Figure 15. Rate of adsorption of Pb2+ on RHAC



Figure 16. Rate of adsorption of Pb2+ on RHBC

These figures show that the amount adsorbed of the Pb2+ heavy metal increases rapidly in the first fifteen minutes. After that, the change of the amount adsorbed is not significant until reaching the equilibrium state. The large concentration of adsorbable solute molecules and the availability of empty active sites in pores on the adsorbent may explain the rapid rate of adsorption in the early stages.

The experimental adsorption data of the current investigation were correlated using pseudo 1st order and pseudo 2nd order kinetics adsorption models. The adsorption kinetic models could be rewritten in linear form as shown in the following formulas [24]:

1st order:
$$\log(qe - qt) = \log(qe) - K1/2.303 * t$$
 (7)

2nd order:
$$t/qt = 1/(K2 * qe^{(2)}) + 1/qe * t$$
 (8)

Figures (17 - 22) illustrate the kinetics experimental data fitting with linearized form of pseudo 1st order and pseudo 2nd order kinetics models, for iron, lead, and zinc heavy metals adsorption on various types of prepared rice husks.



Figure 17. First order kinetic model for the adsorption of Pb2+ on RH at different temperature

2.5



Figure 20. Second order kinetic model for the adsorption of Pb2+ on RH at different temperature



2 5 5 1 0.5 0 0 10 20 30 40 50 Time

Figure 18: First order kinetic model for the adsorption of Pb2+ on RHAC at different temperature



Figure 19. First order kinetic model for the adsorption of Pb2+ on RHBC at different temperature

Figure 21. Second order kinetic model for the adsorption of Pb2+ on RHAC at different temperature



Figure 22. Second order kinetic model for the adsorption of Pb2+ on RHBC at different temperature

It's also possible to see how a pseudo-2nd-order model depicts experimental data by identifying the confidence level (R2) for the chosen equations. The confidence level for pseudo-second-order models has a high value of 0.998 while the confidence level of the pseudo-first-order is 0.987 this demonstrates that the pseudo 2nd order model accurately represents the experimental data. Table (2) show the pseudo 1st order & 2nd parameters of the adsorption of Pb2+ on the three types of prepared rice husks at different temperatures. These findings are consistent with prior studies. They applied pseudo 1st order and 2nd order models for metals removal from wastewater. They concluded that the 2nd order kinetic model fits the data well with an R2 of 0.993.

1	5	4

Adsorbents	R ² Pseudo First Order	R ² Pseudo second order
RH	0.981	0.994
RHAC	0.984	0.993
RHBC	0.987	0.998

Table 2. Average confidence interval levels (R2) for Pseud	o First
order and Pseudo second order.	

3.4 the effect of the initial concentration

The initial concentration of the adsorbate is a crucial factor for determining adsorption capacity and adsorbate removal percentage. The equation (4) was used to calculate the removal percentage:

The initial concentrations of lead and Pb2+ in the solutions are plotted as a function of removal percentage due to adsorption on the surfaces of RH, RHAC, and RHBC in Figures (23 to 25).



Figure 23 . Removal percentage of Pb+2 on RH versus initial concentration at temperature (25-40) C°



Figure 24. Removal percentage of Pb+2 on RHAC versus initial concentration at temperature (25-40) C°



Figure 25. Removal percentage of Pb+2 on RHBC versus initial concentration at temperature (25-40) °C

The increase of the initial concentration of the Pb2+ metal in the solution associated with a decrease in the removal percentage was demonstrated in these figures. However, increasing the initial concentration improves adsorption capacity while decreasing the percentage of Pb2+ removed.

These phenomena could be explained by monitoring the ratio of adsorbed molecules in the solution of the available active sites of the adsorbent. The removal percentage increases as this ratio decreases since the adsorbable molecule in the solution's chance to attach the active site on the adsorbent decreases. Therefore, for low Pb2+ ions concentrations in solutions, the initial number of metal molecules to available adsorbent surface area is small, while for large concentrations of heavy metals in solution, the number of accessible adsorption sites number decreases as the saturation state of the adsorption sites approaches.

In comparison with this result, the study done used activated carbon of rice husks pre-treated with a basic agent as an effective adsorbent for removing different metals Fe2+ and Pb2+ from aqueous solutions. For concentration range (5-100) ppm. They concluded that the removal percentage of Fe2+ and Pb2+ was 94.42% and 90.36% respectively.

4. Conclusion

In this research, activated carbon with a high surface area and high adsorption capacity was produced using no high temperature. The results demonstrate that RHAC or RHBC produces activated carbon with a high adsorption capacity, a porous structure, and a surface area of various shapes and sizes according to FESEM and BET tests. This study shows that the activated carbon RHBC has the highest rate for Pb2+ removal from wastewater compared with RH or RHAC. The Pb2+ metal shows good adsorption capacity at 25°C and as the temperature increases the adsorption efficiency decrease because increasing the temperature leads to an increase in the kinetic energy of the molecules adsorbed on the adsorbent surface, which leads to an increase in the possibility of their separation from the adsorbent surface and back into the solution. The adsorption isotherm data were well fitted by Freundlich isotherm, for Pb2+ ions. The results revealed a sharp rise in adsorption rate initially until equilibrium is achieved. With a confidence level of 0.99, the pseudo-second-order model accurately reflected the data. The results of this investigation will be useful for the removal of Pb2+ metal by rice husks from Battery manufacture wastewater.

Authors' contribution

All authors contributed equally to the preparation of this article.

Declaration of competing interest

The authors declare no conflicts of interest.

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