Unmodified Rice Straw for The Lead Removal Approach from Synthetic Lead Solution

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ABSTRACT

Lead, is the most common toxic metal that causing damage to the environment and human health due to its accumulation and persistence in the environment. The possibility of using biosorption unmodified rice straw (RS) was studied to eliminate lead ions from synthetic industrial wastewater. The impact of the initial concentration for lead (5–200 mg/l) and the contact time (10–120 min) were studied at 25°C and pH 5 of stirring solutions. Different analytical techniques such as Scanning Electron Microscope (SEM), Energy dispersive X-ray (EDX), and Fourier-transform infrared spectroscopy (FTIR) were used to figure out the change of rice straw adsorption capacity Moreover, The kinetic models pseudo-first and second-order and both of Freundlich and Langmuir isotherm models have been tested. The results obtained show a significant removal of the lead content by approximately 94 % using unmodified rice straw (RS). Adsorption data was fitted with Freundlich isotherm model and pseudo - second order kinetics for unmodified rice straw

Keywords: Lead removal, Biosorption, Industrial Wastewater, Rice Straw

INTRODUCTION

Heavy metals are among the major important contaminants in source and treated water, and they are becoming a serious public health concern. Industrial and local wastewater also contains ions of metal, nonetheless, industrial waste is the main source of different types of metal contamination in natural water, (Demirbas2008). Lead is one of the heavy metals that is most poisonous Lead is one of the heavy metals most poisonous that produced from many industrial applications. such as battery manufacturing, electroplating, petrochemical processes, printing pigments, fuels, photographic materials manufacturing, (Jeyakumar2014).

According to the Hazardous Substances Directive of the European Union, the use of lead, cadmium and mercury in the production of electrical and electronic devices is banned as they have had a harmful impact on our safety, and due to their non-biodegradable nature, these metal accumulate in the environment resulting in contaminants of food and water, (Saxena *et al.*, 2017).

Environmental Protection Agency (EPA) and the World Health Organization (WHO), published that maximum permissible lead limit (MPL) in drinking water and wastewater is 0.05 mg/l. In industrial wastewater, the concentrations of lead ions are close to 200–500 mg/l; which is very high in relation to water quality standards that allowed 0.1 mg Pb/l as the maximum concentration (according to the Industrial Waste Disposal Law No. 44/2000) before discharging to drainage systems or water ways, (Arbabi *et al.* 2015). These metal pollutants accumulate in the environment due to their non-biodegradable and persistency in nature that causing the sever contamination of food and water, (Sud *et al.* 2008).

Rice straw (RS) is considered a residual of agricultural product, it is burned to dispose of its large quantities at harvest time. It causes the stuffy smoke the "black cloud" phenomenon which impacts the visibility, human health and global climate by emitting greenhouse gasses, (Park *et al.* 2017).

In this study, we aimed to study the efficiency of RS on the removal of lead from water by using untreated RS powder. The adsorption behavior was investigated using high and low lead concentrations. Different analytical techniques such as Fourier-transform infrared spectroscopy (FTIR spectra), energy dispersive X-ray (EDX) and scanning electron microscope (SEM) were used to figure out the changes of the absorbate up on the adsorption process.

MATERIALS AND METHODS

Materials

Rice straw (RS) was collected from the 10th village, Abis- Alexandria -Egypt in harvest season October 2016. Analytical grade of lead nitrate (99%) was obtained from LOBA Chemie®, India), sodium hydroxide (96%) was obtained from SIGMA-ALDRICH® - Germany and hydrochloric acid (36.5-38%) was obtained from Scharlau® - Spain.

Absorption Atomic Spectroscopy (AAS) Analysis.

The lead concentrations were determinate by Atomic Absorption Spectroscopy (Zeenet700 analytic Jena, flame type) with standard curve range 0.4-15 ppm. These analyses were carried out at the Central Laboratory at City of the Scientific Research and Technology Applications (SRTA-City), New Borg El-Arab, Alexandria. Egypt.

Scanning Electron Microscopy Imaging.

The RS was imaged before and after lead adsorption by using (SEM - JEOL, JSM6010LV) at 150× and 5000× magnification. The samples were coated with platinum-palladium (PT- Pd) to be prepared before scanning. Elemental rice straw analysis was conducted before and after adsorption using the Energy-dispersive X-ray spectroscopy (EDX or EDS) analysis method as an integrated tool of the scanning electron microscope (SEM). (Detector Si Li, 10 mm^2 detection area, Shimadzu, Japan), central labs at SRTA-City (City of Scientific Research and Technological Applications).

Fourier Transform Infrared Spectroscopy (FTIR) Analysis.

FTIR spectrometer (JEOL, JSM6010LV, Japan) at Egyptian Japanese University for Scientific Technology (E-JUST) was used to characterize the RS. FTIR spectra recorded directly from the samples without any treatment. The IR spectrum was estimated in the 4000-400 cm-1 wavenumber range.

Biosorbent Preparation.

The RS was washed with distilled water four times to remove dust according to the method described by (Pengthamkeerati et al. 2008), then it was dried for 24 hours at 60 °C in an oven to get rid of moisture content. Afterwards, the dried RS was crushed with grinder using (Micro-mill®, USA) and the particles were sieved to obtain particle size of 450 μm with a standard sieve. The grained RS was dried in the oven for 1 h at 60° C to ensure they were free of moisture. The obtained RS was kept in desiccators for further experiments.

Effect of pH on the removal of Pb^{+2}

Different lead concentrations (5, 10, 50,100,150 and 200 mg/l) were treated with RS (1 g each, with particle size of 450 μm) and shaken for 2 h at 25∘C. The pH of the samples was initially adjusted using 1.0 N NaOH and 1.0 N HCl to the different equivalent studied pH values (3, 5) for each concentration.

Synthetic Lead Solution Preparation.

Stock solution of lead (II) was prepared by dissolving 1.59 g of lead nitrate in 1 L distilled water (Rice et al., 2017). Different concentrations of synthetic lead solution were prepared by using distilled water to dilute the stock solution. The pH has been maintained at 5 by using 0.1 N HCl and 0.1 N NaOH solutions.

Adsorption Test.

Synthetic solution (50 ml) of Pb^{+2} was poured into 100 ml bottle. One gram of raw RS was added to the solution then incubated on the shaker at 150 rpm up to the desired contact time (2 h) at room temperature (25°C). After the incubation time each sample was subsequently filtered using a filter paper whatman® No. 40 to separate the RS from the treated solution.

Determination of Lead Concentration.

The samples were acidified using nitric acid to a pH < 2 (Rice et al., 2017) and the equilibrium concentration of Pb^{+2} (C_e) was analyzed using atomic absorption spectrometer (AAS). At the beginning of each run, the operating conditions of the AAS were adjusted, and the device was calibrated using a calibration blank and calibration Pb^{+2} standards, prepared using the same lead stock solution used for the synthetic solution. The tests were performed in triplicates (Li et al. 2015).

Data Analysis

The percentage of Pb^{+2} ions removed was calculated using the below equation:

$$R\% = [(C_0-C_e)/C_0] \times 100 \qquad (1)$$

Where, R is the percentage of removal, C_0 is the initial Pb^{+2} ions concentration in liquid phase (mg/l) and C_e is the Pb^{+2} ions concentration at equilibrium in the liquid phase (mg/l).

The adsorption capacity or the amount of Pb^{+2} ions adsorbed on the RS surface layer was calculated as follows:

$$q_e = [(C_0 - C_e) \times V]/m \qquad (2)$$

Where q_e (mg/g) is the mass of metal ions adsorbed per unit mass of the sorbent, C_0 (mg/L) is the initial concentration of Pb^{+2} ions in liquid phase, C_e (mg /L) is

the concentration of Pb^{+2} ions at equilibrium in liquid phase, V (ml) is the Pb^{+2} solution volume (50 ml) and m (g) is the biosorbent mass used(Li *et al.* 2015).

Adsorption Isotherms.

The adsorption isotherms study of Lead (Pb^{+2}) was conducted with different initial lead concentrations and a fixed adsorbent concentration at room temperature (25˚C). One gram of the RS was loaded in a 100 mL bottles, and 50 mL of lead solutions were added. Six levels (5, 10, 50, 100,150, 200 mg/L) of initial lead concentrations were used. The solution's pH was maintained at 5 by use 0.1 N HCl and 0.1 NaOH solutions. The bottles were capped and shaken at 150 rpm for 2 h. The solution was filtered through a filter paper whatman® No. 40 at the end of the adsorption period and then analyzed. The amount of adsorbed lead (adsorption capacity) was calculated and the lead adsorption isotherm data by RS was computed by Langmuir linear form as following:

$$C_e/q_e = (1/K_L.q_m) + C_e/q_m$$
(3)

Where C_e (mg/l) is the equilibrium adsorbate concentration in a liquid phase, q_e (mg/g) is the amount of adsorbate per unit mass of adsorbent at equilibrium, q_m (mg/g) is the maximum adsorption capacity (monolayer capacity), and K_L (l/mg) is the Langmuir constant related to the bonding energy of adsorption, Mohsen-(Nia *et al.* 2007).

Freundlich linear form equations is as follows:

$$Log\ q_e = (1/n*Log\ C_e) + Log\ K_f$$
(4)

where q_e (mg/g) is the amount of adsorbate per unit mass of adsorbent at equilibrium, K_f (mg/g) (l/mg) $^{1/n}$ is the Freundlich capacity factor, K_f is an indicator of the adsorption capacity when metal equilibrium concentration equals to one, 1/n is the Freundlich intensity parameter, C_e (mg/l) is the equilibrium adsorbate concentration in a liquid phase, (Amer *et al.* 2017).

Adsorption kinetic measurements.

Adsorption kinetics for lead was assessed at room temperature (25°C) and an initial 50 and 200 mg/L lead concentration (equivalent to initial loading 50 and 200 mg lead /g RS). 3 g of RS was added in two Erlenmeyer flask 500 ml before starting of each kinetic experiment, and then 150 mL of lead solution was added into the flasks with concentrations (50 and 200 mg/L). The flasks were covered and shacked at 150 rpm. The solution's pH was maintained at 5. Samples were collected at 5-180

min intervals by collection of 15 mL of the reaction solution. Instantly, the sampled solution was filtered through a whatman ® No. 40 filter paper and then analyzed for Pb^{+2} concentration as mentioned previously.

The pseudo-first order kinetic model is given using the equation below:

$$Log\ (q_e\text{-}q_t) = log\ q_e - (K_1/\ 2.303)\ t$$
(5)

q_t and q_e are the amount of solute sorbed per mass of sorbent (mg/g) at time t and at equilibrium, respectively, and k_1 is the rate constant of first-order sorption (min^{-1}). The straight-line plot of log ($q_e − q_t$) versus t gives log (q_e) as slope and intercept equal to $k_1/2.303$. So, the amount of solute sorbed per gram of sorbent at equilibrium (q_e) and the first-order sorption rate constant (k_1) can be evaluated from the slope and the intercept, respectively.

Pseudo-Second-Order Kinetic Model is given by applying below equation:

$$t/q_t = 1/K_2\ q_e^2 + (1/q_e)\ t$$
(6)

$K_2\ q_e^2$ describes the initial sorption rate in which K_2 is the constant rate q_t is the capacity for metal uptake at any time t.

RESULTS AND DISCUSSION

Lead adsorption with RS at different pHs

Lead adsorption with RS at pHs (3 and 5) as preliminary experiment to determine the common conditions should be used to study the adsorption of lead (II) by using untreated rice straw (RS), the lead (II) removed (mg) by 1 g rice straw was studied as a function of the pH as shown in Figure 1. The solution's pH has a major impact on the elimination of because it determines the degree of the charge on the surface of RS and ionization of lead ions in the solution, (Anayurt *et al.* 2009).

At pH values higher than 6, the lead (II) ions precipitate as lead (II) hydroxide, (Amer *et al.* 2017). The adsorbed amount increased very slightly with increasing the pH, at the pH range of 3 - 5. Ion exchange has been mainly dominated on adsorption as H^+ ions are competitive to lead ions for ion exchange sites. The adsorption of lead is strongly enhances at pH 5. The highest lead removal was achieved at 10 ppm at pHs (3 and 5) showing 89.26, 89.95 % of lead removed, respectively (Figure 1).

The pH 5 was chosen to be used for the adsorption experiments of different initial lead concentrations by

using RS, because it is much closer to the pH of industrial effluents than pH 3.

The adsorbent dosage effect

The lead (II) ions adsorption by RS was done by using different dosages of adsorbent (0.05–1.0 g) at 25°C and with initial concentration of 200 mg/L metal solution. The percentage of adsorption increased from 51.63 to 86.43%. When the adsorbent dosage was increased from 0.05 to 1.0 g (Figure 2), the increasing of adsorption percentage can be correlated with the increase in the active sites on the adsorbent and thus facilitate easier penetration of the ions of metal into adsorption sites (Mohamad *et al*., 2010).

Although, it seemed no significant change obtained by the gradual increases of the RS concentrations from 0.5 to 1 g, it was decided to use 1 g RS to ignore the effect of limited used RS or available adsorption sites on the used RS.

RS exposed to different lead concentrations

Adsorption of different concertation of lead by RS where studied. Results depicted that the increase of the initial lead concentration resulted in an increase in the amount of the removed lead (Figure 3). Whereas, the highest lead removal was 94.6 % at 150 mg/L initial lead concentration.
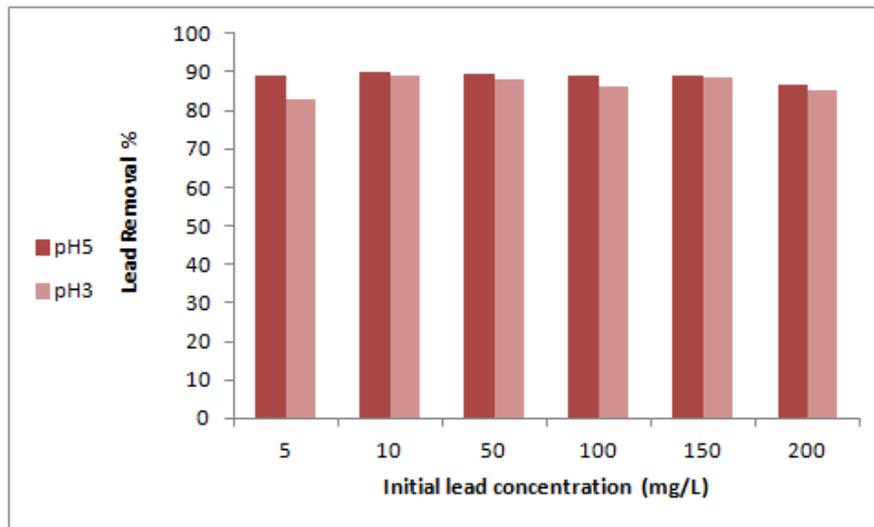

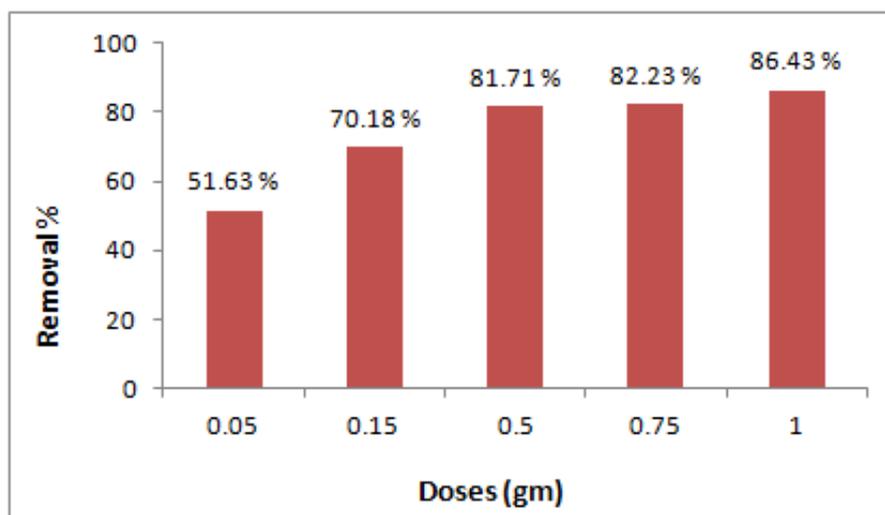**Fig.1. Lead (Pb^{+2}) removal from aqueous solution at pH 3 and pH 5 using RS**

Fig.2. The removal% of lead (Pb^{+2}) from aqueous solution using different doses of RS
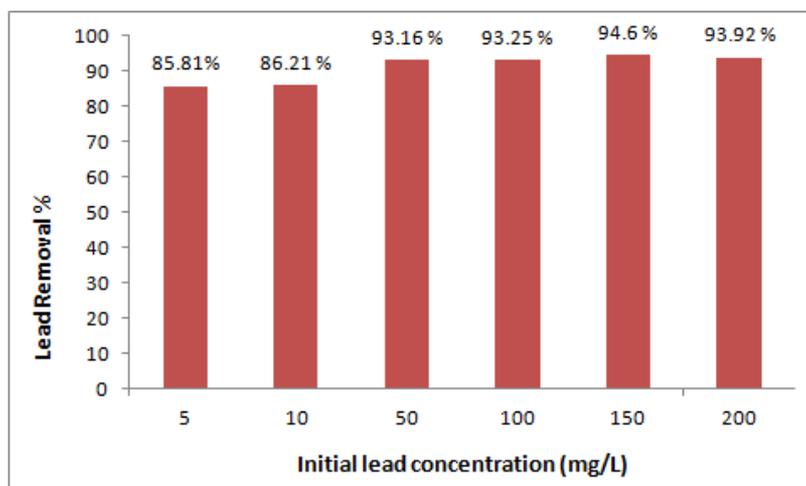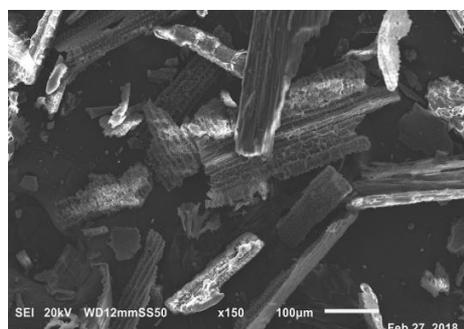
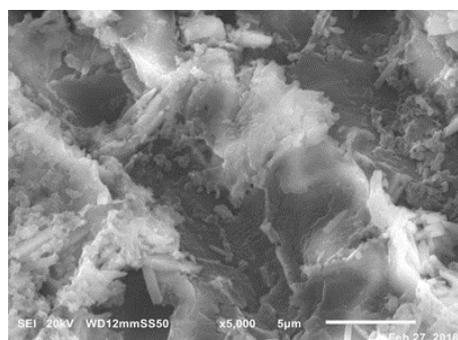**Figure 3. Removal of Lead (%) versus the initial concentration of lead (mg/l) by Using the RS powder**

Characterization of RS

Figure 4 shows the images for rice straw pre and post of lead adsorption were carried out using SEM (at x100, 150 x and x5000). It was noticed the RS had an irregular a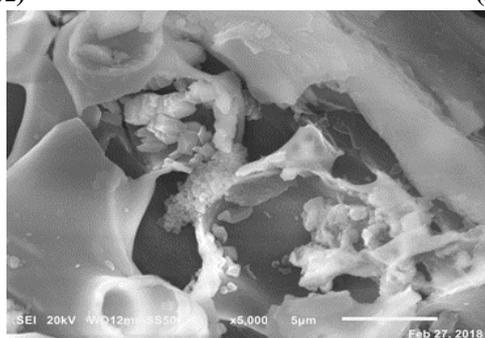nd coarse nonporous surface (Figure 4A and 4B), and after lead adsorption, the accumulated lead showed aggregates/flukes on the surface of the RS (Figure 4C).

(A)

(B)

(C)

Figure 4. SEM images of the rice straw (RS) at two magnifications (A; 150x and B; 5000x) before lead adsorption and the RS (C) after lead adsorption at 5000x magnification

In addition to large amounts of SiO_2, The rice straw consists mainly of lignin, hemicellulose, cellulose, extractives, mineral ash and water, (Li *et al*. 2017). The lignin can interact promptly with cations, idea exchanging with protons and chelating with the metal ion, (Rocha *et al*. 2009).

The rice straw pre and post of lead adsorption was analyzed by (FTIR Spectra), respectively, shows the bands of absorption around 3454 cm^{-1} that could be resulted from the OH stretching mode at macromolecular association of cellulose and lignin. The peaks at 1645, 1461and 2923, cm^{-1} describe lignin, aromatic C=O and C=C and alkane CH, respectively. A peak at 1388 cm^{-1} could be attributed to carboxyl carbonate and aromatic CH stretching bonds. At 1087 or 470 cm^{-1} the absorptions could be associated with SiO_2 stretching and SiO bending indicating the presence of silica as well as cellulose and hemicellulose. The FTIR spectrum of the rice straw wasn't significantly changed after lead adsorption except abrading the intensity reduction in the peak at 1087 cm^{-1} that propose the linkage between the lead and the silica content of the rice straw.

The composition of RS before and after lead adsorption was analyzed by EDX (Table 1) which represented the main composition of the RS before and after lead adsorption. Many inorganic elements such as Al, Cl, K, etc. were detected in the RS before and after lead adsorption. Lead was detected in the EDX spectrum of rice straw after adsorption that proved the Pb^{+2} biosorption on the RS surface was taken place. The presence of oxygen could be attributed to presence polar groups on the RS surface (S-type). The reduction in the oxygen after lead adsorption proposed the linkage of the lead.

The adsorption isotherms and kinetics of lead

The isotherm of lead adsorption by RS was applied by isotherm models for both Freundlich and Langmuir. From the slope and intercept of the linear plot of C_e/q_e against C_e, the Langmuir constants, K_L and q_e were calculated. The Langmuir isotherm parameters are shown in (Table 2) for both RS. Although the low value of R^2 in case of using RS illustrates that the Langmuir model does not well fit the lead adsorption.

The log q_e vs. log C_e plot from which the Freundlich parameters, K_F and n, were specified from the intercept and slopes of the line (Figure 6) for RS. The correlation coefficients of the models are shown in (Table 3).

Table 1. The element composition of RS before and after lead adsorption

Element	RS before Lead adsorption	RS after lead adsorption
	Mass% (At %)	Mass % (At %)
C	36.93 (47.40)	37.04 (49.13)
O	44.24 (42.63)	39.50 (39.33)
Si	15.80 (8.67)	16.45 (9.33)
Pb	------	4.98 (1.25)
Other element traces (Al, K, Cl, Ca)	3.03 mass%	2.03 mass %

Table 2. Langmuir model's parameters (q_m, K_L, and R_2) using RS

RS		
q_m (mg/g)	K_L (L/mg)	R^2
5.15	0.07	0.6492

Table 3. Freundlich models' parameters using RS

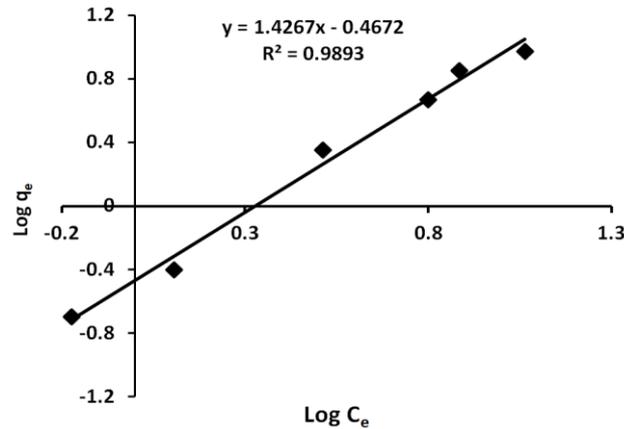| RS		
K_F	N	R^2
0.34	0.7	0.9893

Figure 6. Freundlich isotherm plots for using RS

Based on $R^2 = 0.9893$ coefficients value, well correlation with the adsorption data was observed. If $1/n$ values was more than 1 that indicative of S-type isotherms. These were comparatively uncommon, but are often found at low concentration levels for adsorbed surfaces with a polar functional group in which low concentrations are present. For the adsorptive, The RS surface has a low affinity, this affinity increases with higher concentrations of lead (II). At low concentrations of lead, it has been hypothesized that, the Lead (II) is in competition for adsorption sites with water. This isotherm is known by a decreasing slope as Lead (II) concentration increases so when the adsorbent (RS) becomes covered the unoccupied (free) adsorption sites decrease. This behaviour of adsorption could be clarified by the high affinity of the adsorbent for the adsorptive at low concentrations and then decreases with increases of the lead (II) concentration. (Amer 2015).

For investigation of the kinetics of biosorption, the pseudo first order and second-order kinetics models were applied. In Figure 7, the slope and intercept of the linear plots corresponding to the both kinetic models can be determined, the value of the adsorption capacity at equilibrium and the rate constant for the two-initial concentration of lead (50 and 200 ppm) adsorbed by RS at three different temperatures 15, 25 and 40°C for solution. The isotherm plots at 25°C by using low (50 ppm) and high (200 ppm) initial lead (II) concentrations, the kinetic parameters produced from the analysis of experimental data at the same temperature are shown in Table 4. (Brahmaiah *et al*. 2015)
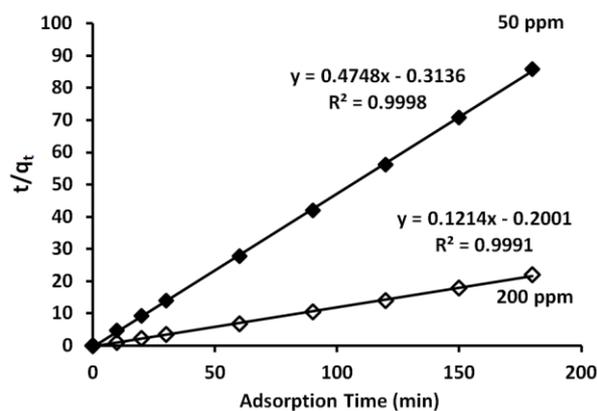

Figure 7. Representations of Biosorption kinetics of Pb (II) using pseudo-second-order model using RS at 25°C. Two initial concentrations of Pb (II) (50 and 200 ppm) were used

Table 4. Description of Pb (II) Kinetics biosorption by pseudo-first and pseudo-second order models. at 25°C on RS

C_0 (mg/L)	Pseudo-first order model			Pseudo-second order model		
	k_1 (1/min)	q_e (mg/g)	R^2	k_2 (mg/g min)	q_e (mg/g)	R^2
	Rice Straw (RS)					
50	0.0023	0.10	0.6810	0.72	2.11	0.9998
200	0.0039	0.59	0.9636	0.07	8.17	0.9991

Table 5. The different biosorbents and its sorption capacity for pb(II)

Sorbent	Pretreatment	Size/mm	pH	Sorption capacity (mg/g)	Literature sources
Sugarcane bagasse	Concentrated H_2SO_4	0.21	4-6	1.58	H.P.Lal et al,(2010)
Sawdust	Formaldehyde in sulfuric acid	0.1-0.25	5.5	9.78	V. C. Taty-costodes et al, (2003)
Rice husk	Unmodified	0.6	6	2.80	R. T. Prabha and T. H. Udayashankara , (2014)
Waste tea leaves	Conc. HCl	Dried biomass	5	8.6	B. S. S. Ahluwalia and D. Goyal (2005)
Almond shells	0.4 mol/L NaOH	1–5	5-6	9	M. R. Mehrasbi (2009)
Rice Straw	Unmodified	0.45	5	2.11	This study

The data derived from applying the aforementioned kinetic models shows that biosorption of Pb (II) on RS fitted with the pseudo-second-order kinetic model. In full agreement with previous studies, (Brahmaiah *et al.* 2015), the pseudo-first-order Lagergren equation is not appropriate according to lower coefficients of R^2 (<0.5).Whereas, the plots of pseudo-second order kinetic model shown in Figure 7, depicted a linear fit indicating second order kinetics with higher correlation factors (R^2>0.99) (Table 4). Thus, the model pseudo-second-order kinetic proposed that biosorption of lead ions on the RS is based on chemical reaction as supported by reduction in oxygen content after lead adsorption as shown in the EDX analysis (see Table 1), involving exchange of electrons or sharing between adsorbate and adsorbent, (Amer 2015).

Compared with other types of plant biomass, the absorption capacity of lead using untreated rice straw (this study) (2.11 mg/g) was compared to the unmodified rice husk (2.8 mg/g) and the modified sugarcane bagasse (1.58 mg/g). On other hand, the absorption capacity was high in case of other treated agriculture wastes such as treated waste tea leaves with conc. HCl (8.6 mg/g), almond shells treated with 0.4 mol/ 1 NaOH (9 mg/g), and sawdust treated with formaldehyde in sulfuric acid (9.78 mg/g). The treatments resulted in both degradation of hard lignin content and open new adsorption sites that lead to higher adsorption capacity than untreated ones. However, the use of natural adsorbents such as cheap and untreated rice straw is more environmentally friendly than the use of adsorbents that have been treated with expensive chemicals and are harmful to the environment (see Table 5).

CONCLUSION

In this study, the adsorption experiments for the removal of Pb (II) from aqueous solutions have been carried out using unmodified rice straw as low-cost, high efficiency, economic feasibility, and simplicity of preparation. The adsorption characteristics have been examined at different pH values, doses of rice straw and initial metal ion concentrations. Adsorption kinetics and isotherm were also examined. The obtained results can be summarized as follows:

1- The highest removal % of Pb^{+2} ions from aqueous solution was 94.6 and 93.92% at concentrations 150 and 200 ppm, respectively.

2- Using 1 gm rice straw, approximately, 94% of 200 ppm Pb^{+2} was removed at pH5.

3- Adsorption data was well fitted with Freundlich isotherm and adsorption equilibrium data was well fitted with Lagergren second order kinetics this indicates that the adsorption may be due to chemical adsorption.

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الملخص العربي

قش الأرز الغير معالج كطريقة لإزالة عنصر الرصاص من محلول يحتوى على الرصاص

عبد الفتاح سيد عبد الكريم سعد ، رانيا على حلمى عامر، السيد حسن محمد تايب ، نورهان نادى ، رحاب جمعة محمد

الرصاص، هو المعدن السام الأكثر شيوعًا الذي يتسبب في ضرر للبيئة وصحة الإنسان بسبب تراكمه و ثباته في البيئة. تمت دراسة إمكانية استخدام قش الأرز الحيوي (RS) لإزالة أيونات الرصاص من مياه الصرف الصناعي الصناعي. تمت دراسة تأثير التركيز الأولي للرصاص (٥ – ٢٠٠ مجم / لتر) ووقت التلامس (١٠–١٢٠ دقيقة) عند درجة حرارة ٢٥ درجة مئوية ودرجة الحموضة ٥.

كما تم استخدام تقنيات تحليلية مختلفة مثل المسح الإلكتروني بالمجهر (SEM) ، الأشعة السينية المشتتة للطاقة (EDX) ، فورييه – التحويل الطيفي بالأشعة تحت الحمراء (FTIR) لأكتشاف معدل تغيير قدرة قش الأرز على الامتصاص. بالإضافة الى ذلك تم أختبار النماذج الحركية من الدرجة الاولى والثانية الزائفة و تم اختبار كلا النموذجين Freundlich و Langmuir متساوي الحرارة .

أظهرت النتائج التي تم الحصول عليها انه تم ازالة نسبة كبيرة من محتوى الرصاص حوالى ٩٤٪ باستخدام قش الار الغير معالج. كما كانت بيانات الامتزاز متطابقة تماما مع نموذج الحركة من الدرجة الثانية الزائفة ونموذج Freundlich متساوى الحرارة بأستخدام قش الأرز الغير معدل.