INTRODUCTION

Potentially toxic heavy metals occur naturally in low concentrations, but with the development of the human industrial activities such as mining and smelting operations, batteries production, metal plating, and fertilizer applications, the concentrations of these metals were significantly increased above the allowable ranges, which consequently threatened the environment due to their adverse toxic effect on humans and animals (Esmaeili & Aghababai Beni, 2015; Jauiberty et al., 2017; Qasim et al., 2021).

The heavy metals from the industrial wastes discharged result in water pollution which transforms the clean water to un-wanted wastewater which therefore affect the quality of natural resources of water and the water ecosystem due to their immunogenic, carcinogenic and mutagenic action (Witek-Krowiak, 2013). Therefore, the wastewater treatment became one of an important things that attracted the researcher’s interest.

In the recent years, various conventional methods have been applied for heavy metals removal from wastewater. These methods were mainly categorized as chemical, physical and biological methods (Crini & Lichtfouse, 2019; Peng et al., 2021; Shankor et al., 2021). These conventional methods included ion exchange, membrane filtration, precipitation, coagulation and flocculation (Dong et al., 2019; Karim et al., 2019). In fact, the above mentioned conventional methods suffered from some limitations in terms of operating cost, chemical consumption, producing of toxic sludge and environmental
impact. Among them, the adsorption of heavy metals using agricultural by-products such as saw dust, maize corn cob, wheat straw, rice husk as activated carbon is considered as a proficient technique due to its high adsorption capacity, cost-effectiveness, ease of use and also simple regeneration of adsorbent (Thompson et al., 2016; Hina Khatoon and Jai Prakash Narayan Rai, 2016; Wang et al., 2019). Therefore, the choice of appropriate adsorbents with low cost and highly adsorption capacity is necessary for removal of heavy metals. The peanut shell that is produced in huge quantities constitutes a substance rich in cellulose and polymer materials that contain reactive functional groups which make it good raw material source for activated carbon preparation. The activated carbon is characterized as a porous carbonaceous material with highly surface area, it considered as an effective adsorbent and a good chelating agent that forms stable complexes with heavy metals in aqueous solutions which are used to purify the wastewater (AL-Othman et al., 2012; Wang et al., 2020; Al-Jadir & Siperstein, 2018). The physical and chemical activation are the traditional methods for preparing the activated carbon. Various chemical activators such as H₃PO₄, H₂SO₄, Ca(OH)₂ have been already used for the preparation of activated carbon (Liu et al., 2020). The alkaline agent such as KOH is one of the most effective activators that enhance the peanut shell adsorption properties by: i) improving its swelling as well as its capacity for cationic pollutants adsorption, ii) improving its ability to disintegrate the lignocellulose internal structure which enhances its pore structure and subsequently increases its adsorption capacity, iii) removing the impurities from peanut shell surface thereby exposing chemically the reactive functional groups like hydroxyl (OH), and iv) increase the concentration of the oxygenated complexes on peanut shell surface (L. Huang et al., 2014; Ashrafi et al., 2016; H. Huang et al., 2017; Nam et al., 2018).

In the literature, the information concerning the efficiency of the KOH-activated carbon for heavy metals removal is still limited. For this reason, the aim of the current study was to: i) prepare KOH-activated peanut shells; ii) study its efficiency for Zn, Pb and Cd removal from wastewater; and iii) study the isotherm and the kinetic process to understand the adsorption process.

**MATERIALS AND METHODS**

**Activated peanut shell preparation**

The peanut shells used in current study were collected locally from farming markets. The collected material was firstly washed with tap water for two hours to remove the dirt and coloration, and then washed several times with distilled water. To reduce the water content, the washed peanut shells were dried using a laboratory oven set at 110 °C for 24 h. The dried material was powdered using a laboratory grinder, and sieved using a mesh sieve to obtain 100 µm particles size. The sieved peanut shells were dried at 110 °C for 24 h using a laboratory oven. A suitable weight of the prepared peanut shells was carbonized at 550 °C in a closed furnace equipped with argon tank to supply 2 ml/min of argon gas to obtain a carbonized peanut shell (biochar). The collected biochar were chemically activated using 7M KOH solution, immersed in 500 ml of KOH at 60 °C for 4 h, filtered by using 45 µm filter paper, and then dried at 110 °C for 24 h. The obtained KOH activated biochar then activated at the same above mentioned furnace for 2 h at 750 °C (Wang et al., 2020). After cooling the samples to room temperature, the resulting material was washed with deionized water and dried in oven at 110 °C for 24 h.

**Metal ions solution preparation**

The stock solutions for the studied metal ions were prepared by dissolving an appropriate amount of Zn(NO₃)₂, Pb (NO₃)₂ and Cd(NO₃)₂ in distilled water to achieve 1000 mg/l of Zn²⁺, Pb²⁺ and Cd²⁺ respectively. The prepared stock solutions were used to prepare different initial metal ion concentrations. To adjust the pH of the prepared solutions, 0.1M of both HCl and NaOH was prepared and used to achieve the desired pH values.

**Physico-chemical characterization study**

The activated peanut shell was characterized by SEM (scanning electron microscope INPECT S50, Germany) to investigate its external surface morphology. The BET (Brunauer–Emmett–Teller; Micro active for Tri-Star II Plus 2.03, U.S.A) method that is based on adsorption–desorption isotherm of nitrogen was performed to observe the specific surface area of the activated peanut shell. The XRD (X-ray diffraction analysis; Shimadzu
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trations (5 – 700 mg/l) at optimum pH value and by adding a desired amount of adsorbent to 100 ml metal ions per unit mass of activated peanut shell, the relationship between the content of adsorbed Isotherm study was conducted at 25 – 60 °C and optimum metal ion concentration, optimum pH and nut shell dose was conducted by adding different pH and contact time. The effect of activated pea- sorbent to individual metal ions (10 mg/l) at 25 °C. The effect of solution pH was conducted by adding 0.5 g of adsorbent to different pH values (3 – 8) at 25 °C. The effect of contact time was performed by adding 0.5 g of adsorbent to individual metal ions (10 mg/l) at 25 °C and optimum pH with different time intervals (5 – 240 min). The effect of initial metal ions was performed by adding 0.5 g of adsorbent to different individual metal ion concentrations (5 – 100 mg/l) by adding 0.5 g from adsorbent at optimum pH and contact time. The effect of activated peanut shell dose was conducted by adding different amounts of adsorbent (0.5 – 4 g) to 100 ml at opti- mum metal ion concentration, optimum pH and optimum contact time. The effect of temperature was conducted at 25 – 60 °C.

Batch adsorption analysis

The batch adsorption experiments were performed to evaluate the effect of variable parameters on the adsorption efficiency of the activated peanut shell as a metal ions adsorbent. These parameters included: value of the solution pH, peanut shell dose, initial metal ion concentrations, contact adsorption time and temperature. The batch experiments were performed by shaking a known weight of activated peanut shell with 100 ml of individual metal ions in 250 ml conical flasks at 200 rpm using a rotary shaker. The adsorbed solutions were filtered using 45 µm filter paper, and the metal ion concentrations in the filtrates were determined by ICP-MS spectrophotometry.

The effect of solution pH was conducted by adding 0.5 g of adsorbent to 10 mg/l of individual metal ion at time of 120 min with different pH values (3 – 8) at 25 °C. The effect of contact time was performed by adding 0.5 g of adsorbent to individual metal ions (10 mg/l) at 25 °C and optimum pH at 200 rpm using a rotary shaker. The adsorbed solutions were filtered using 45 µm filter paper, and the metal ion concentrations in the filtrates were determined by ICP-MS spectrophotometry.

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Kinetic study

The following equation that represents the pseudo-first-order model proposed by Lagergren, describes that the rate of adsorption onto the occupied sites is proportional to a number of unoccupied sites (Tounsadi et al., 2015), in addition, this kinetic model works only when the adsorption occurs rapidly:

\[ q_t = q_e (1 - \exp(-k_1 t)) \]  

(5)

where: 
- \( q_t \) (mg/g) – the adsorption capacity at time \( t \);
- \( q_e \) (mg/g) – the adsorption capacity at equilibrium;
- \( k_1 \) – the constant of the 1st order kinetic (min\(^{-1}\)).

The pseudo-second order kinetic which occurred in the solid phase and referred to that the adsorption capacity is based on the available adsorbent active surface site and can be expressed as follows (Vijayaraghavan et al., 2017):

\[ q_t = \frac{q_e^2 k_2 t}{1 + q_e k_2 t} \]  

(6)

where: 
- \( k_2 \) – the constant of the 2nd order kinetic (g.mg\(^{-1}\).min\(^{-1}\)).

RESULT AND DISCUSSION

Characterization of activated peanut shell

The images of the SEM for KOH-activated peanut shell are shown in Figure 1. It can be seen that a rougher, irregular and rigid surface with porous structure was observed in comparison to the inactivated peanut shell. The activation of peanut shell by KOH enhanced the formation of peanut shell porous structure surface which made it capable of adsorbing the metal ions from their solutions onto its accessible surface active sites and also promoting the metal ions diffusion onto adsorbent surface (Dotto et al., 2014). In addition, increasing the temperature of carbonization also played an important role in the formation of cleaner and smoother pore size via completely removing the residues that were generated from the carbonization process (Nazir et al., 2021).

The FTIR spectrum for the activated peanut shell is presented in Figure 2. The broad peak at 3385 cm\(^{-1}\) was assigned to stretching vibrations of the O–H groups with intra-molecular hydrogen bonds of the cellulose stretching (Huo et al., 2013). The band at 2970 cm\(^{-1}\) was assigned to C–H stretching vibration of alkyl groups (Iqbal et al., 2009). The band at 1637 cm\(^{-1}\) represents the C=O stretching of carboxylic acid in hemicelluloses, lignin or and pectin (Adiana & Mazura, 2011). The peak of 1404 cm\(^{-1}\) corresponds to the C–C stretching vibration (Georgin et al., 2016). The peak at 1001 cm\(^{-1}\) was assigned to stretching C–O bond in alcohols or ethers. The peaks at 701 cm\(^{-1}\) and 833 cm\(^{-1}\) were attributed to C–H stretching vibration.

The X-ray diffraction (XRD) test showed that the activated peanut shell contains an amorphous phase for hemicelluloses, lignin and also pectin (Fig. 3). The cellulose is the only substance that represents a crystalline phase which is represented by its peaks at: 22.4°, 24.4°, 30.6° and 31.8° 2\(\theta\), meaning that the peanut shell is mainly composed from cellulose.
The textural properties of the activated peanut shell are shown in Table 1. It can be seen that the KOH activated peanut shell had a highly BET value and a highest micro pores volume and total pore volume, compared to unactivated peanut shell. This result actually confirmed mesoporous nature and the high peanut shell surface density as a result of its highly surface area. The porosity of the peanut shell surface promotes the binding of the metal ions onto the active sites and therefore makes it as an efficient adsorbent.

### Factors affecting the efficiency of metal ions adsorption

The adsorption of the studied metal ions onto the surface of KOH-activated peanut shell was affected by various factors such as:

**pH value**

The effect of pH values (3 – 8) on the efficiency of the adsorption process are presented.
in Figure 4. It can be seen that the percentage of metal ions removal was increased by increasing the value of solution pH from 4 to 6. The maximum values for the adsorption capacities were achieved at pH 6. Afterwards, the adsorption efficiency for metal ions removal was decreased with the increasing pH value. These results can be explained based on the fact of that the protonation of the active functional groups such as carboxyl as well as amino groups is altered by the solution pH. It is well known that at lower pH, the concentration of protons increased and take place on the peanut shell surface which subsequently increased the competition between metal ions and protons onto the available active sites of peanut shell surface causing an increase in the remaining free metal ions in the solution (Witek-Krowiak et al., 2011). On the other hand, when pH increased, the metal ions will precipitate as a result of hydroxyl anion, increasing in the solution which reduces the removal percentage of the studied ions (Aryee et al., 2021). Therefore, the pH value was adjusted at 5 to 6 not higher. These results were consistent with many researchers (Zanin et al., 2017), reported that when pH solution increased up to 5, the reduction of Cu concentration was also increased. In the same trend (Nageeb Rashed et al., 2016), found that the adsorption of the methylene blue by activated carbon increased along with the pH.

**Contact time**

The data that obtained from the removal of the studied metal ions by activated peanut shell at various contact times (5 – 240 min) at optimum pH is presented in Figure 5.

It can be seen that the removal percent increased along with contact time from 5 to 60 min, and then it remained constant or decreased gradually with the increasing of contact time. At the initial stages of contact time, the removal of the studied metal ions happened rapidly, which might be attributed to the availability of a large amount of active sites that enhance the removal percentage of the metal ions onto the adsorbent surface (Abo-El-Enein et al., 2017). In contrast, when the contact time increased, the remaining active sites as well as the uncovered surface area decreased. Therefore, according to obtained data, 60min of contact time was the best time to reach the maximum adsorption of metal ions onto the activated peanut shell surface where the removal percent was up to 95%.

**Initial metal ion concentrations**

The data of the effects of the different metal concentrations (5–100 mg/l) on the adsorption efficiency of the activated peanut shell is shown in Figure 6. The obtained results showed that the removal percentage for the studied metal ions increased along the initial metal concentrations from 5 to 40 mg/l. These results can be explained depending on the fact of that the increasing of the metal concentration caused an increase in the adsorption efficiency. This can be explained by the fact that at higher concentration, the metal ion molecules occupied more active binding sites (Georgin et al., 2016). The removal rate for Zn, Pb and Cd ranged from 95–97%, which
reached the maximum capacity when the initial metal ion concentrations was 20 mg/l for Zn and Pb and 40 mg/l for Cd. Afterwards, the removal percent of the metal ions decreases considerably. Therefore, the optimum initial concentration of 20 and 40 mg/l was chosen to be the optimum concentrations.

**Adsorbent dose**

The amount of adsorbent is responsible for the number of the available active sites on adsorbent surface. For this reason, the effect of the activated peanut shell dose was studied at optimum pH, contact time and initial concentration of metal ions value by varying the dosage of activated peanut shell from 0.5 to 4 g. The removal efficiency of the different adsorbent doses was presented in Figure 7. The results showed that the best removal percentage was observed with using 2 g of activated peanut shell. This result is attributed to the increasing of the potential active binding sites with the increase of the adsorbent dose (Garg et al., 2019). The decrease of the removal efficiency with adsorbent dose higher than 2.5 g can be attributed to the increasing of the covered active sites by metal ions which subsequently cause a loading in adsorbent capacity. Therefore, the optimum adsorbent was chosen to be 2 g for the next adsorption experiment.

**Temperature**

The effect of temperature on removal percentage of the metal ions by activated peanut shell at different temperatures (25–60 °C) with optimum experimental conditions is presented in Figure 8. The results showed that the varying of temperature was affected the adsorption efficiency. With the increase of temperature from 25–45 °C, the removal percent was also increased, and then it decreased gradually. The decrease of the metal ions removal percent with increasing of temperature can be attributed to the weakness of the adsorptive forces between metal ions and the active sites onto peanut shell surface. On the other hand (Isawi, 2020; Mohammed & Kareem, 2019), reported that the increasing of temperature will increase the viscosity of the solution resulting in low metal ions movements which finally reduce the adsorption efficiency. From the obtained results, 45 °C was chosen as a best temperature for the adsorption of metal ions.

**Adsorption isotherm and kinetic studies**

The study of the adsorption isotherm provides important information about the relationship between the mass of the studied metal ions per unit
mass of the adsorbent as well as the metal ion concentrations in the solution when reached equilibrium.

In this work, two well-known adsorption isotherm models named Langmuir and Freundlich were chosen to fit the obtained experimental data that related to the sorption of the metal ions onto peanut shell using Origin Pro 8 software. The higher correlation coefficient ($R^2$) as well as the lower error values can be used to determine the best fitted isotherm model.

The values of the isotherm data for the Langmuir and Freundlich models were calculated by nonlinear regression as shown in Figure 9, whereas the other related constants were presented in Table 2.

The data that were obtained from the Langmuir model showed a higher correlation coefficient with a value of $R^2$ which ranged from 0.989 to 0.994 for the studied metal ions as compared to that obtained from the Freundlich model which ranged from 0.873 to 0.917.

The separation factor $R_L$ that represents the adsorption modes can be expressed as: when $R_L$ = 0, the adsorption is irreversible, when $R_L$ = 1, the adsorption is linear, the adsorption is favorable when 0 < $R_L$ < 1, whereas adsorption is unfavorable when $R_L$ > 1. The obtained $R_L$ values for the studied metal ions were ranged from 0.016 to 0.76 which indicates that the adsorption of Zn, Pb and Cd onto the surface of the activated peanut shell were favorable.

The $n$ value that represents the intensity of the metals adsorption in Freundlich equation can be expressed as follows: when $n$ > 1, the adsorption process is physical, $n$ = 1 is linear, and when $n$ < 1 the adsorption is chemical. The presented results in Table 2, showed that the $n$ value was > 1, indicating that the adsorption process of the metal ions onto peanut shell surface was physical adsorption.

The value of $q_{max}$ obtained from Langmuir was close to experimental data $q_{exp}$. In addition, the value of SSE (sum of the squared errors) that obtained from Langmuir model was lower than that obtained by the Freundlich model.

![Figure 9. Adsorption isotherm models of Langmuir and Freundlich for (a) Pb, (b) Zn, (c) Cd](image-url)
According to the above results, the higher $R^2$ value, the small difference between $q_{\text{exp}}$ and $q_{\text{max}}$, and the lower SSE values, makes the Langmuir model as the best fitted isotherm model for metal ions adsorption onto the activated peanut shell surface in comparison to Freundlich model.

To assess the rate of adsorption, the adsorption constants as well as the mechanism of the metal ions sorption onto peanut shell, the obtained data was fitted to two different kinetic models: pseudo-first order and pseudo-second order (Fig. 10). The correlation coefficient ($R^2$) and the kinetic

<table>
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<th>KOH-activated peanut shells</th>
<th>Langmuir</th>
<th>Freundlich</th>
</tr>
</thead>
<tbody>
<tr>
<td>Metals</td>
<td>$R^2$</td>
<td>$K_L$</td>
</tr>
<tr>
<td>Pb</td>
<td>0.990</td>
<td>0.085</td>
</tr>
<tr>
<td>Zn</td>
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<td>0.062</td>
</tr>
<tr>
<td>Cd</td>
<td>0.994</td>
<td>0.106</td>
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Table 2. Adsorption isotherm values for metal ions

<table>
<thead>
<tr>
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<th>Pseudo first order</th>
<th>Pseudo second order</th>
</tr>
</thead>
<tbody>
<tr>
<td>Metals</td>
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<td>$K_1$</td>
</tr>
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<tr>
<td>Zn</td>
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</tr>
<tr>
<td>Cd</td>
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</table>

Table 3. Adsorption kinetic values for metal ion

![Figure 10. Adsorption kinetic for (a) Pb, (b) Zn, (c) Cd](image)
constants that determined by using nonlinear regression were presented in Table 3.

From the fitting results, a good agreement has been observed between the proposed model and the experimental data for the studied metal ions. It can be seen that the obtained data was fitted better with the pseudo-first order kinetic than the pseudo-second order kinetic with a correlation coefficient (R²) > 0.99 which revealed a good applicability of the first-order kinetic for the adsorption of the metal ions using activated peanut shells.

**Effect of ionic strength**

The presence of co-ions in water or in soil solutions may influence the metal ions adsorption efficiency. The effect of the ionic strength of different competitive ions such as PO₄³⁻, SO₄²⁻ and NO₃⁻ on the sorption ability of the Zn²⁺, Pb²⁺ and Cd²⁺ onto activated peanut shell surface is presented in Table 4. The results showed that the removal percentage of the metal ions decreased with the increasing of ionic strength from 5 to 20 mg/l and following the order PO₄³⁻ > SO₄²⁻ > NO₃⁻. No significant differences were observed with ionic strength of 5 mg/l, whereas, a significant decrease was observed with 20mg/l. (Yang et al., 2021), also reported that the increasing of the ionic strength reduced the adsorption capacity of Cd²⁺ on biochar. The ionic strength is an important factor that affects the affinity between the aqueous phase and solutes which subsequently affect the equilibrium of the aqueous phase (Ahmaruzzaman, 2011). The ionic strength can affect the interface potential, as well as the double layer thickness between the metal ions solution and peanut shell surface and therefore limit the binding of metal ions with adsorbents (Li et al., 2012). The presence of some inorganic ions such as phosphate, sulfate and chloride in the aqueous solution will compete with the adsorbent to form complexes with metal ions and therefore reduce the removal percentage of adsorption by the current adsorbent (Gan, 2000; Weng & Huang, 2004).

**CONCLUSIONS**

This work examined the potential and the efficiency of the available, natural and low cost peanut shells for the removal of Zn, Pb and Cd from wastewater.

The carbonization of peanut shell at 550 °C as well as the KOH-activation increased the S BET (349.2 m²/g) and also enhanced the availability of the active functional groups that contribute to adsorption of metal ions onto the adsorbent surface. The metal ions bio sorption was significantly affected by different parameters such as pH, adsorbent dose, metal concentrations, contact time and temperature. The adsorption isotherm models that were used in this study to describe the adsorption equilibrium showed that the maximum adsorption capacities were observed with the Langmuir model compared to the Freundlich model. The kinetic experiment for the metal ions adsorption followed the pseudo-first order model. The comparison of the obtained results from this work with the other reported results in the literature indicated that the carbonization as well as KOH-activation of peanut shells exhibits a good adsorption for the studied metal ions.

**REFERENCES**


3. Adiana, M.A., Mazura, M.P. 2011. Study on Senna alata and its different extracts by Fourier


