



Optimal Operating Conditions for Adsorption of Heavy Metals from an Aqueous Solution by an Agriculture Waste

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Abstract

The aim of this work is to detect the best operating conditions that effect on the removal of Cu^{2+} , Zn^{2+} , and Ni^{2+} ions from aqueous solution using date pits in the batch adsorption experiments. The results have shown that the Al-zahdi Iraqi date pits demonstrated more efficient at certain values of operating conditions of adsorbent doses of 0.12 g/ml of aqueous solution, adsorption time 72 h, pH solution 5.5 ± 0.2 , shaking speed 300 rpm, and smallest adsorbent particle size needed for removal of metals. At the same time the particle size of date pits has a little effect on the adsorption at low initial concentration of heavy metals. The adsorption of metals increases with increasing the initial of metal concentration while above the 85 ppm, the removal efficiency was decreased. The phenomenon of adsorption of heavy metals on to Al-Zahdi Iraqi Date pits is exothermic process. The maximum adsorption capacity according to the Langmuir equation was 0.21, 0.149, and 0.132 mmol/g for Cu^{2+} , Zn^{2+} , and Ni^{2+} respectively. The adsorption equilibrium was well described by the Freundlich model. The results of Freundlich constants indicated that the adsorption capacity and adsorption intensity of Copper is larger than the Zinc and Nickel. The intraparticle diffusion was involved in this process but it is not the controlling step. The results of this study may inspire to find the optimal operating conditions for adsorption and develop that with large-scale production to clean the polluted water with heavy metals.

Keywords: Adsorption, Heavy metals, Al-Zahdi Iraqi date pits, wastewater, Freundlich.

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1- Introduction

The contamination of water with heavy metals is the biggest problem in the environmental pollution because of their tending to accumulate in the living organisms and cannot be degraded, therefore cause various diseases [1, 2, 3]. The world health organization (WHO) stated that the permissible of Cu^{2+} , Zn^{2+} , and Ni^{2+} for drinking water are 1, 3, and 5 ppm respectively [4].

There are many physical and chemical processes are available for the removal of heavy metals from wastewater, such as electrochemical precipitation, ultra-filtration, solvent extraction, sedimentation, membrane technologic, ion exchange, and reverse osmosis.

For low heavy metal concentrations, these techniques inefficient in certain cases and have high capital and operational cost [5].

The adsorption process can be an attractive alternative method for the removal of heavy metals [6]. Some adsorbents can adsorb a wide range of heavy metals, whereas others are specific for certain types of metals [7].

The application of adsorption in environmental treatment has become a significant research area in recent years [8, 9, and 10].

Many researchers used agricultural wastes for removal of heavy metal ions such as raw orange peel [11], modified sugarcane bagasse [12], modified Oak sawdust [13], modified Lignin [14], wood and bark residues [15], peanut hull [16,17], dehydrated wheat bran [18], maize leaf [19], Bean pee [20], tobacco leaves [21], banana and orange peels [22], coconut husk [23], charcoal [24], modified sugar beet pulp [25], modified cellulosic materials [26], modified corncob [27].

Although, the date pits have been demonstrated as an adsorbent for heavy metal ions, the calibration of the best operating conditions for the adsorption of Cu^{2+} , Ni^{2+} , and Zn^{2+} from aqueous solution using Al-Zahid date pits were not fully investigated. Saad et al [28] stated that the date pits adsorbent offers a unique advantage in rapid pre-concentration and recovery of UO_2^{2+} from dilute aqueous solution at pH 6–7.5 by flow mode of separation.

Also, Al-Ghouti et al [29] demonstrated the potential of date pits for adsorbing methylene blue, Cu^{2+} , and Cd^{2+} to provide fundamental information for studying equilibrium adsorption isotherms and adsorption mechanism. Bouhamed et al [30] indicated that the prepared activated carbon from date stones for removal of Cu^{2+} , Zn^{2+} , and Ni^{2+} ions from an aqueous mixture was suitable for the removal of metal ions present in a multicomponent system in contaminated Wastewater.

Zainab Mahdi, et al. [31] used the date seed biochar for removal of Pb^{2+} , Cu^{2+} , and Ni^{2+} ions from binary and ternary systems.

In this work, Al-Zahdi date pits, an agricultural waste generated from food processing and jams production, were used as adsorbent material to remove Cu^{2+} , Ni^{2+} , and Zn^{2+} from an aqueous solution. The influence of operating conditions on the removal of heavy metals was studied to predict the best conditions which help us to improve and develop the efficiency of this process.

2- Experimental work.

2.1. Raw Materials

An agricultural solid waste (Al-Zahdi Iraqi date pits), de-ionized distill water, $CuSO_4 \cdot 5H_2O$, $Ni(NO_3)_2 \cdot 6H_2O$, $ZnSO_4 \cdot 7H_2O$, HCl, and NaOH, with high purity of more than 99 percent, were purchased from Sigma Aldrich and used in this work.

2.2. Prepare the Adsorbent and Aqueous Solutions

The date pits were washed several times with deionized water and left in the open air for several days and then they were transferred to the oven at $80^\circ C$ until dryness.

The adsorbent was crushed, milled, and sieving into a different particle size of, 1500, 1000, 500, and 200 μm . The standard stock of heavy metals aqueous solutions of Cu^{2+} , Zn^{2+} , and Ni^{2+} (1000 ppm) were prepared by dissolve $CuSO_4 \cdot 5H_2O$, $Ni(NO_3)_2 \cdot 6H_2O$, and $ZnSO_4 \cdot 7H_2O$ salt in a certain amount of deionized distilled water. The standard solution of heavy metals was diluted to achieve standard concentrations of 10, 35, 60, 85, and 110 ppm of heavy metals.

3- Experimental Work

3.1. Effect of the Adsorbent Dosage

Date pits of 200 μm mixed heavy metals (Cu^{2+} , Zn^{2+} , and Ni^{2+}) solution with dose of 0.04, 0.08, 0.1, 0.12, 0.16, 0.2, and 0.3 g/ml were put in the bottles of 250ml. The pH (6 ± 0.2) was adjusted by 1M HCl or 1M NaOH. The samples were agitated by a shaker (kottermann, Germen) at 250 rpm at room temperature ($25 \pm 2^\circ C$) until equilibrium. Then the adsorbent was separated by filtration through 0.45 μm of cellulose nitrate membrane. The heavy metal concentrations in the remaining solution were analyzed by using an absorption spectrophotometer (Spectro AA 10). The uptake capacity (q_e) (mg/g) of heavy metals was calculated by using the eq. (1):

$$q_e = (C_{Ai} - C_{Aeq}) V_{sol} / W_d \quad (1)$$

Where C_{Ai} and C_{Aeq} represent the initial heavy metal and equilibrium concentration respectively. V_{sol} is the volume of solution and W_d is the weight of date pits used.

3.2. Effect of Particle Size

The different particle sizes of Date pits (1500, 1000, 500, and 200 μm) were mixed with heavy metals solution (60ppm) with an adsorbent dose of 0.12g/ml and pH 6 ± 0.2 . The mixtures were agitated (250 rpm) at room temperature ($25 \pm 2^\circ C$) until equilibrium, and then the adsorbent was separated and analyzed for the remaining concentration.

3.3. Effect of Shaking Speed

Date pits of a particle size of 200 μm were added to the heavy metal solution (60 ppm) to adsorbent dose of 0.12 g/ml and pH 6 ± 0.2 . The mixtures were agitated for different speeds (150, 200, 250, 300, 350, and 400 rpm) at room temperature ($25 \pm 2^\circ C$) until equilibrium then the adsorbent was separated and analyzed for the remaining concentration.

3.4. Effect of ph of Solution

Heavy metals solution of 60 ppm with different pH ranges from 2 to 12 was prepared. The adjustment of pH solutions to the appropriate value was conducted by adding either 1M HCl or 1M NaOH. The date pits of 200 μm were added to these solutions with an adsorbent dose of 0.12 g/ml. The samples were agitated (300 rpm) at room temperature ($25 \pm 2^\circ C$) until equilibrium. The adsorbent was separated from samples and the remaining solution was analyzed.

3.5. Effect of Temperature.

Heavy metal solutions of different initial concentrations range from 10 to 110 ppm were mixed with date pits of 200 μm with an adsorbent dose of 0.12 g/ml at pH 5.5 ± 0.2 . Then the mixtures were agitated (300 rpm) at different temperatures (25, 35, 45, and $55 \pm 2^\circ C$) until equilibrium. The remainder solutions were analyzed.

3.6. Kinetic Experiments

A certain amount of adsorbent of 200 μm was added to the heavy metal solution of different concentrations (10, 35, 60, 85, and 110 ppm) and pH 5.5 ± 0.2 with an adsorbent dose of 0.12 g/ml. The mixtures were agitated by shaker at 300 rpm at room temperature ($25 \pm 2^\circ C$). Then the samples were taken periodically for analysis to study the dynamic of adsorption of heavy metals by date pits process.

4- Result and Discussion

4.1. Effect of the Adsorbent Concentration

Fig. 1 presents the effect of date pits dose on the heavy metals uptake capacity at constant other conditions until equilibrium.

The heavy metals uptake increases with increasing the adsorbent dose to 0.12g/ml of heavy metals solution value and then decreases with increasing the adsorbent dose. This is due to increase the contact surface area and the active adsorption sites with increasing the adsorbent dose [13]. Further increase in the adsorbent dose may cause agglomeration of particles which affects reducing the adsorption efficiency of heavy metals [18].

4.2. Effect of the Particle Size

The effect of particle size of date pits on the uptake capacity of heavy metals from aqueous solution is shown in Fig. 2.

While Fig. 3 shows the copper uptake capacity against the initial concentration of heavy metal in the solution for different particle sizes of date pits at constant operating conditions(adsorbent dose 0.12g/ml, pH=6,250 rpm, and 25°C) until equilibrium.

As shown in Figs 2 and 3, the heavy metals uptake capacity increases with decreasing the particle size of the adsorbent.

Fig. 4 presents the SEM micrographs for the raw date pits before (4a) and after (4b) adsorption of heavy metals respectively. The raw date pit has numerous pores and channels on the surface. This is a positive for the ability of date pits to be a good surface for adsorption of heavy metals from aqueous solution as shown in Fig4b. The raw date pit has porosity on the surface and many channels between the particles of date pit material.

When decreasing the particle size during the crushing and grinding process, the sealed channels were opened in the adsorbent, and that increase in the surface area and the active sits of the adsorbent which then becomes available for adsorption, therefore the increase of heavy metals uptake capacity.

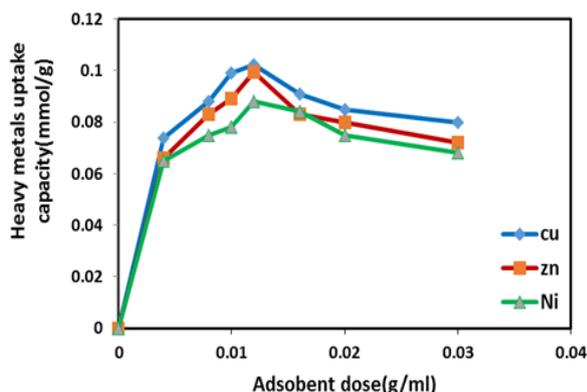


Fig. 1. Heavy metals adsorption capacity vs. adsorbent dose

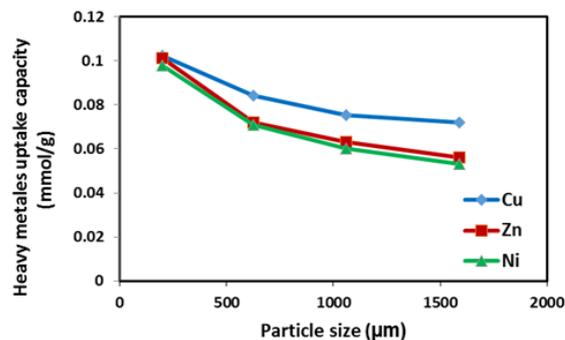


Fig. 2. Heavy metals adsorption capacity vs. particle size of date pits

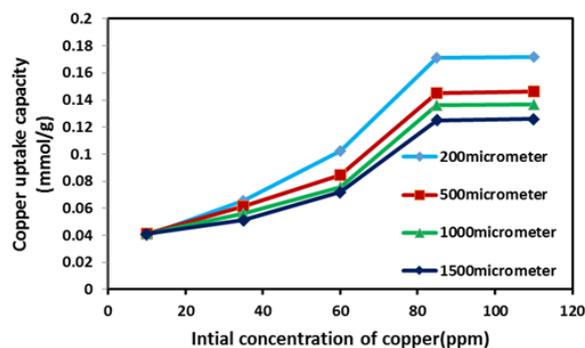
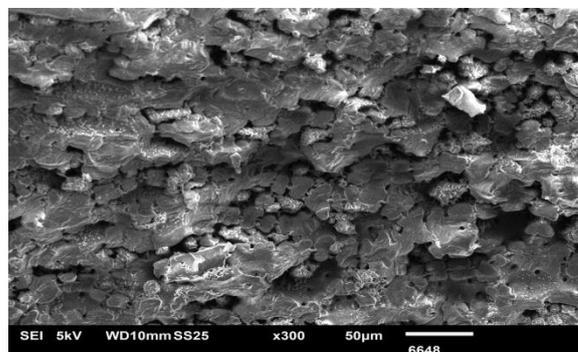
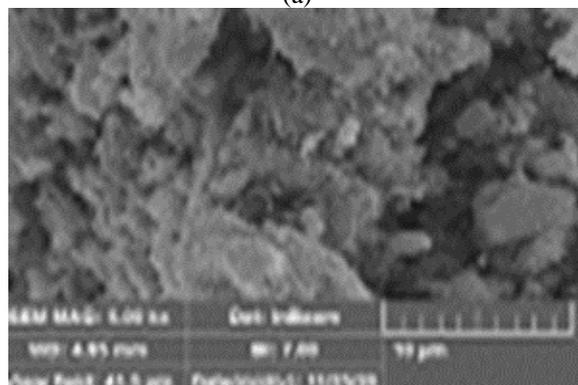


Fig. 3. Heavy metals adsorption capacity vs. initial concentration of copper in the solution for different particle size of date pits



(a)



(b)

Fig. 4. SEM micrographs for the raw date pits of date pits (a) and after adsorption of heavy metals (b)

4.3. Effect of Shaking Speed

Fig. 5 shows the effect of shaking speed on the removal of heavy metals aqueous solution of 60 ppm with constant operating conditions (adsorbent dose 0.12 g/ml, pH 6 at room temperature, and adsorbent particle size of 200 μ m) until equilibrium. The results show that the heavy metals uptake capacity increased with increasing the speed of shaking up to 300 rpm and then decreased when the shaking speed increased above 300 rpm. At high shaking speed, the mass transfer resistance was decreased. Further increase in shaking rate may desorb the heavy metal ions from the adsorbent. Therefore it could conclude that a shaking rate of 300 rpm of agitated speed was sufficient to obtain better adsorption.

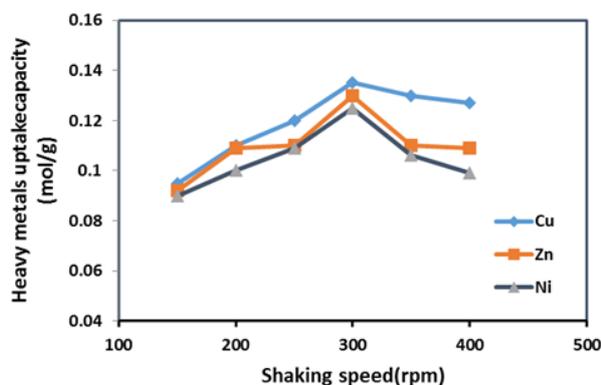


Fig. 5. Heavy metals adsorption capacity vs. shaking speed

4.4. Effect of Initial pH of Solution

Fig. 6 shows the uptake capacity of the heavy metals onto date pits with constant operating conditions (adsorbent dose 0.12g/ml, initial heavy metal solution of 60 ppm, adsorbent particle size of 200 μ m at room temperature and shaking speed of 300 rpm) for different pH solution. The results show that adsorption capacity of heavy metals increased with increasing the pH of solution up to 5.5 and then decreased with increasing the pH above this value.

This is attributed to that the high concentration of H^+ ions in acidic solution and they make a competition with heavy metals on the active sites onto the surface of the adsorbent. Özer et al [18] stated that at low pH values, the surface of adsorbent would be surrounded by hydronium ions (H_3O^+), which decrease the copper interaction with binding sites of the adsorbent.

This is hindering the access of metal ions by greater repulsive forces. While above pH of 5.5, maybe occurring the precipitated components as a result of reacted the OH^- ions with the heavy metal ions [12, 32]. Özer A. et al [18] and Quek et al [33] demonstrated that the critical pH value was 5 for removal of copper onto dehydrated wheat bran and sago waste respectively.

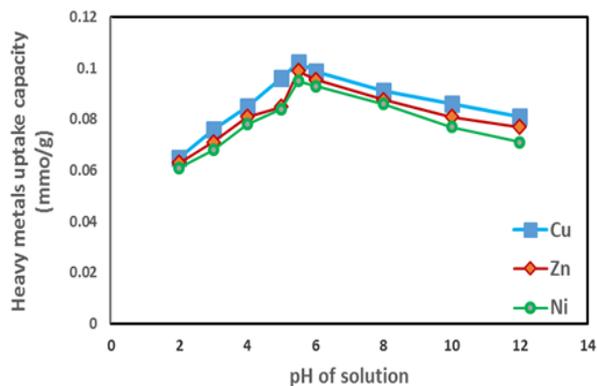


Fig. 6. Heavy metals adsorption capacity vs. PH of the solution

4.5. Effect of Initial Concentration and Adsorption Time

The effect of initial concentration of heavy metals and time of adsorption on the uptake capacity of Cu^{2+} , Zn^{2+} , and Ni^{2+} is shown in Fig. 7, Fig. 8, and 9 respectively, at constant operating conditions (adsorbent dose 0.12g, adsorbent particle size of 200 μ m, pH 5.5, 300 rpm of shaking speed).

These figures show that the adsorption capacity of heavy metals increased with increasing the initial metal concentration of heavy metals as a result of increase the driving force of mass transfer. Above initial concentration of 85 ppm of heavy metals, increasing the removal efficiency of metals was insignificant as a result of saturated active sites on the date pits. It could be seen that the larger uptake capacity of Cu^{2+} , Zn^{2+} , and Ni^{2+} occurred at 72 h of adsorption time, and then the efficiency of metal adsorption becomes constant with increasing the adsorption time.

This is attributed to that the adsorbent reached to saturate with heavy metals. Increasing the shaking speed to the optimum value of 300 rpm is sufficient to assure the reduction in the boundary layer around the surface of the particles, then the effect of external film diffusion assumed not significant as mentioned previously in sec. (4.3). Eq. (2) was used to state the role of intraparticle diffusion.

$$q_t = k_d t^{1/2} + C \quad (2) \quad [34]$$

Where q_t is the adsorption capacity of metal (mg/g), k_d is the initial rate of diffusion ($mg L^{-1} min^{-1/2}$), and C is constant of intercept. Figs 10, 11, and 12 show the Cu^{2+} , Zn^{2+} and Ni^{2+} uptake capacity versus the square root of the adsorption time respectively for different initial concentrations of heavy metals in the solution.

Most of data represented by straight lines but these data did not pass through the origin point. Therefore according to Weber and Marries [35], Intraparticle diffusion was involved in the adsorption process but is not the controlling step.

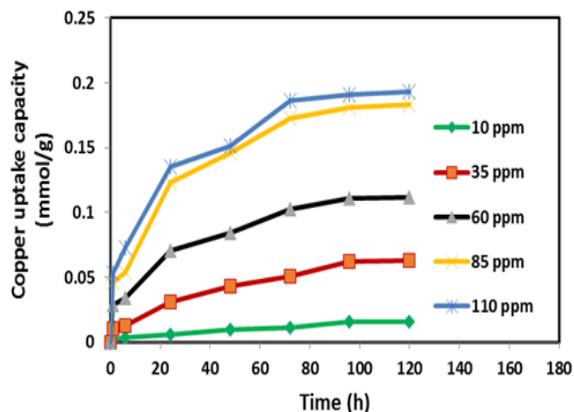


Fig. 7. Copper adsorption capacity vs. time of adsorption with several of Copper concentration

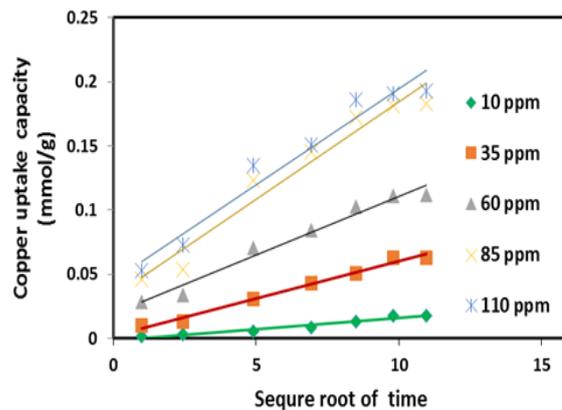


Fig. 10. Copper adsorption capacity vs. time square of adsorption

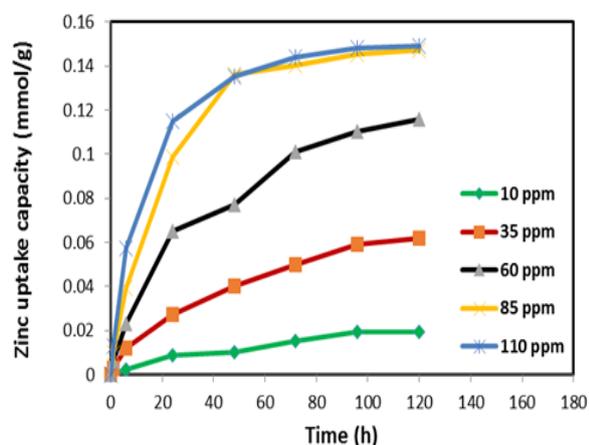


Fig. 8. Zinc adsorption capacity vs. time of adsorption with several of Zinc concentration

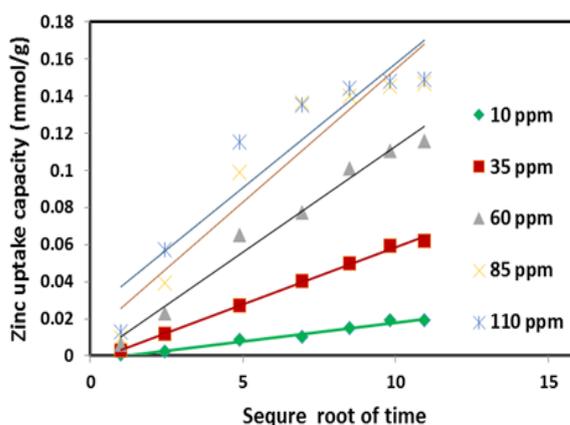


Fig. 11. Zinc adsorption capacity vs. time square of adsorption

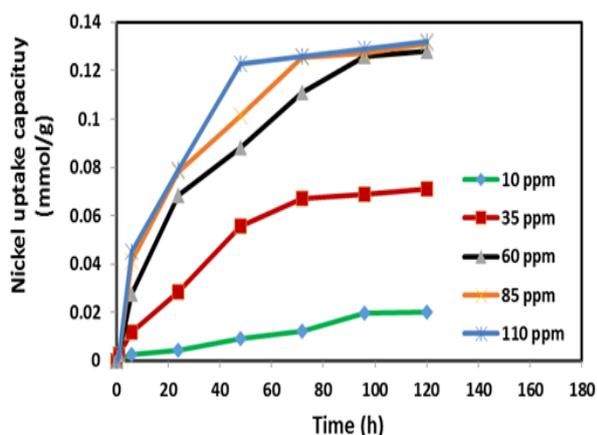


Fig. 9. Nickel adsorption capacity vs. time of adsorption with several of Nickel concentration

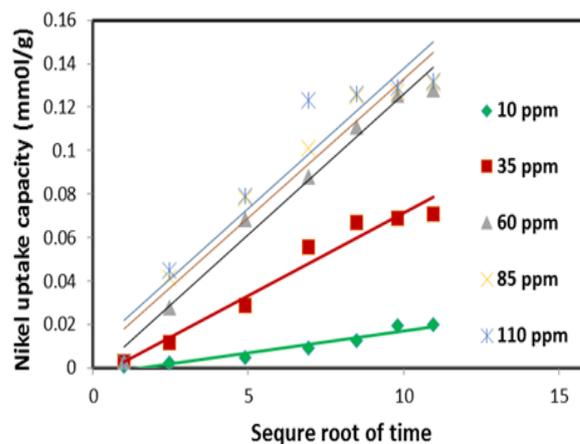


Fig. 12. Nickel adsorption capacity vs. time square of adsorption

4.6. Adsorption Isotherm

Equilibrium studies gave the capacity of the adsorbent for adsorption [36]. Many researchers have shown that the concentration of the adsorbent is usually proportional exponentially to the concentration that remains unadsorbed. The Langmuir equation 3 [37] and Freundlich equation 4 [38] are given by.

$$q_e = q_m k C_e / (1 + k C_e) \quad (3)$$

$$q_e = K_f C_e^{1/n} \quad (4)$$

Where q_m is the maximum adsorption capacity (mg/g), k is the fitting parameter, C_e is the concentration of the heavy metal in solution at equilibrium (mg/l), q_e is the uptake capacity (mg/g), while K_f and n are the Freundlich constants which are empirical constants depending on several environmental factors and. K_f and n indicate how favorable the adsorption process. The maximum adsorption capacity according to the Langmuir equation was 0.21, 0.149, 0.132 mmol/g for Cu^{2+} , Zn^{2+} , and Ni^{2+} respectively.

These results are in agreement with the previous study for the removal of heavy metals onto raw date pits and prepared activated carbon from date pits. Bouhamed et al.[30] stated that the maximum adsorption capacity was 0.29, 0.18, and 0.27 mmol/g for Cu^{2+} , Zn^{2+} , and Ni^{2+} using prepared activated carbon from date pits in ternary mixture and them was 0.49, 0.33, and 0.27 mmol/g [39,40] in single metal system respectively. Banat et al. [41] found that the adsorption capacity for non-activated date pits towards Cu and Zn was 0.15 and 0.09 mmol/g respectively. Therefore, the date pits without any pretreatment could be used as an effective adsorbent for adsorption of heavy metals from an aqueous solution. The isotherm experimental data follow the Freundlich model very well with the values of R^2 (0.866-0.921) as shown in table 1. The Freundlich constants for the removal of metals in comparison with other results using different adsorbent for the same metals are given in table 1. From table 1 it could be seen that the K_f and $1/n$ values of Cu^{2+} are higher than that of Zn^{2+} and that of Ni^{2+} . This is indicated to that the adsorption capacity and adsorption intensity for copper removal was the highest as shown in figures 7, 8, and 9. The adsorption capacity of Cu^{2+} is higher than for Zn^{2+} and Ni^{2+} . This result is in agreement with the results of Al-Ghuoti et al [29] and Banat et al [42]. Banat et al explained that this behavior is based on ionic radii.

4.6. Effect of Temperature

Fig. 13 shows the heavy metal adsorption capacity versus different adsorption temperatures for various initial concentrations of heavy metal with pH 5.5 by using 0.12g of date pits (200 μ m) /ml of solution for 72 h of mixing time.

The adsorption capacity of heavy metal ions decreased with increasing temperatures, thereby demonstrating the exothermic nature of the bioadsorption process. This is in agreement with the finding of AL-Asheh and Duvnjak [43].

Table (1) Freundlich constants for adsorption of Cu^{2+} , Zn^{2+} and Ni^{2+} for different adsorbents.

Reference	Sorbent	R^2	$1/n$	K_f	Metal
Viraraghavan and Dronamraju[44]	Peat	0.898	0.895	0.0002	Cu^{2+}
Mullen et al [45]	A. Niger	0.956	0.894	1.035	
De Rome and Gadd [46]	c. resinae	----	0.6	3.467	
Banat et al.[41]	Data pits	0.9939	1.2979	2.5653	
	AC date pits*	0.999	1.377	1.769	
Bouhamed al[30]	AC date pits*	0.96	0.69	0.6	
This work	Date pits	0.866	0.606	0.529	
Viraraghavan and Dronamraju[44]	Peat	0.885	0.771	0.0001	Zn^{2+}
Banat et al.[41]	Data pits	0.9973	0.3268	0.0781	
	AC date pits*	0.9921	0.412	0.069	
Bouhamed al[30]	AC date pits*	0.95	0.74	0.32	
This work	Date pits	0.912	0.711	0.2374	
Viraraghavan and Dronamraju[44]	Peat	0.907	0.565	0.0001	Ni^{2+}
Bouhamed al[30]	AC date pits*	0.98	0.7	0.46	
This work	Date pits	0.846	0.608	0.1739	

*(AC) prepared activated Carbone.

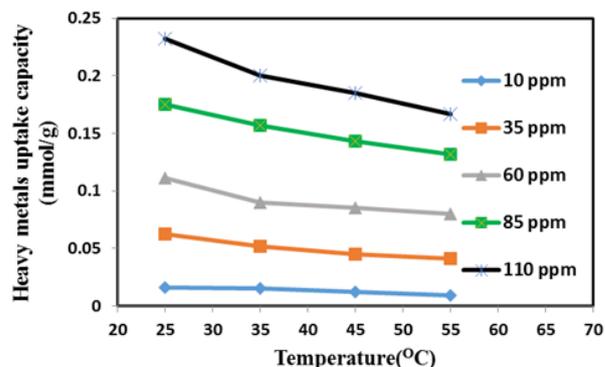


Fig. 13. Heavy metals adsorption capacity vs. temperature of adsorption

5- Conclusion

The results clearly showed that the study of the operating parameters is essential for finding the best conditions and all of these parameters affected the removal efficiency of heavy metals from an aqueous solution. From this work, it could be concluded that the best adsorbent dose was 0.12g of date pits/ml of solution. The adsorption capacity of heavy metals increases with decreasing the adsorbent particle size while the reduction of particle size has a little effect on the removal of heavy metals at a low initial concentration of heavy metals in the solution.

While at a higher concentration of heavy metals an effect was observed. The experimental work indicated that the increases in the shaking speed results increase in the efficiency of heavy metals removal until up to 300 rpm of shaking speed for 72 h and then decreases with an increase above that.

The critical pH of the solution was 5 ± 0.2 and below or above this value of pH, the adsorption capacity of heavy metal ions is decreased.

The efficiency of removal of heavy metals decreased when the concentration of the initial metals became greater than 85 ppm.

The maximum adsorption capacity according to the Langmuir equation was 0.21, 0.149, 0.132 mmol/g for Cu^{2+} , Zn^{2+} , and Ni^{2+} respectively. The adsorption of heavy metals onto Al-Zahdi Iraqi Date pits was an exothermic process and the adsorption equilibrium was well described by the Freundlich model.

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الظروف التشغيلية المثلى لأمتزاز المعادن الثقيلة من المحلول المائي باستخدام النفايات الزراعية

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الخلاصة

يهدف البحث الى دراسة الظروف التشغيلية المثلى والتي تؤثر على ازالة ايونات النحاس والزنك والنيكل من المحلول المائي باستخدام نوى التمر في تجارب الامتزاز بالدفعات . اشارت النتائج الى ان نوى التمر الزهدي فعال في امتزاز المعادن الثقيلة عند ظروف محددة من الجرعة 0.12g/ml في المحلول ,وزمن امتزاز 72h وحمضية 5.5 وسرعة اهتزاز 300rpm وصغر الحجم الحبيبي وفي نفس الوقت لوحظ ان الحجم الحبيبي له تاثير قليل على الامتزاز عند التراكيزالواطئة للمعادن الثقيلة . وان الامتزاز يزداد بزيادة التركيز الابتدائي للمعادن بينما تقل كفاءة الامتزاز عند تركيز اعلى من 85 ppm . واكدت النتائج ان امتزاز المعادن القيلة باستخدام نوى التمر الزهدي هي عملية باعثة للحرارة .وان اعلى سعة امتزاز طبقا لمعادلة لانكميور كانت 0.21 و 0.149 و 0.132 mmol/g للنحاس والزنك والنيكل على التوالي.ان معادلة فريدلخ كانت افضل من يمثل امتزاز التوازن وان نتائج ثوابت فريدلخ اشارت الى ان سعة الامتزاز وشدة امتزاز النحاس كانت اكبر من الزنك والنيكل . وان الانتشار داخل الحبيبية كان تأثيره موجود ولكن ليس المسطر على العملية . وان نتائج هذه الدراسة ربما تساعد في ايجاد الظروف المثلى للامتزاز وتطويرها للعمليات الانتاجية الصناعية لتنظيف المياه الملوثة بالمعادن الثقيلة.

الكلمات الدالة: الامتزاز, المعادن الثقيلة, نوى التمر الزهدي, المياه الملوثة, فريدلخ, لانكميور