Iranian (Iranica) Journal of Energy & Environment
Journal Homepage: www.ijee.net
IJEE an official peer review journal of Babol Noshirvani University of Technology, ISSN:2079-2115

Adsorption Isotherms and Kinetics of Lead and Cadmium Ions: Comparative Studies Using Modified Melon (*Citrullus colocynthis*) Husk

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PAPER INFO

Paper history:
Received 25 May 2020
Accepted in revised form 26 June 2020

Keywords:
Freundlich Isotherm
Kinetics
Langmuir Isotherm
Pseudo

ABSTRACT

Growth in population has caused a corresponding increase in agricultural production which consequently led to an increase in agricultural wastes or byproducts. This is sometimes difficult to manage and may pose a menace to the society if not properly disposed. Hence, this research investigated the efficacy of utilizing agricultural byproduct (melon husk) in removing Pb^{2+} and Cd^{2+} from wastewaters so as to prevent their deleterious impact on ecosystem and public health. This was achieved by treating tannery effluents with melon husks modified with H_3PO_4, NaOH and CH_4N_2O. Results showed that, the Freundlich and Langmuir adsorption capacities on the modified melon husks ranged from 0.1403 – 1.551 mg/g and 0.1499 – 0.6013 mg/g, respectively. Also, the R^2 values for Pseudo 1st and 2nd order rate constants ranged from 0.0272 – 0.2391 and 0.9112 – 0.9958, respectively. Hence, it was concluded that the modified melon husks followed Freundlich isotherm model as well as Pseudo-2nd order kinetics and could be effectively used to remove Pb^{2+} and Cd^{2+} from wastewater.

***doi**: 10.5829/ijee.2020.11.02.10*

INTRODUCTION

The impact of heavy metals in wastewaters or effluents has long been identified by various researchers. Hence, several conventional techniques of removing heavy metals from effluents have been developed in order to reduce or avoid its impacts. However, most of these techniques like chemical precipitation, ion exchange, membrane filtration as well as electrolytic recovery are all associated with both technical and economic constraints in developing countries thus, limiting their usages [1, 2]. Among the various heavy metals, Mercury (Hg), Lead (Pb) and Cadmium (Cd) are known to be highly toxic. This is because the United States Environmental Protection Agency (USEPA) established their maximum permissible limits to be very low compared to others as could be observed in Table 1. Hence, priority should be given to these metals in order to evade their lethal effects. Nevertheless, Lead ion (Pb^{2+}) and Cadmium ion (Cd^{2+}) have been identified of having high concentrations in tannery effluents [3, 4].

Some researchers have worked on the use of agricultural byproducts like coconut shell, maize cob, orange peels, cassava peels, rice husk and banana peels in removing heavy metals from wastewaters [5–10]. However, very little has been done using melon (*Citrullus colocynthis*) husk. Besides, *Citrullus colocynthis* (Figure 1a) is highly demanding in West-African countries (developing countries) especially Nigeria since the seeds (Figure 1b) are used in preparing a special delicacy known as "egusi soup" after threshing. This encourage commercial cultivation of *Citrullus colocynthis* in the mentioned countries. However, the husks or chaffs left after threshing the seeds (Figure 1c) are often considered as waste. Moreover, numerous studies have reported that adsorbent capacity on metal ions could be boosted by treating it with phosphoric acid (H_3PO_4), sodium hydroxide (NaOH) and urea (CH_4N_2O) among others [11–13]. Hence, it is important to investigate the efficacy of using the modified form of this abundant byproduct of *Citrullus colocynthis*, in adsorbing Pb^{2+} and Cd^{2+} in tannery effluents, which have been reported to have high concentrations of the said metals [3].

MATERIALS AND METHOD

Preparation and modification of adsorbent

Husks (chaffs) of *Citrullus colocynthis* seeds were obtained from marketers in Sabon Gari Market, Zaria-Nigeria (11°6'24.99''N, 7°43'30.83''E) thereafter, metallic objects in the husks were removed with the aid of a bare magnet. This was done in order to ensure that the husks were free from materials that may create extra metallic ions. Surface

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TABLE 1. Permissible limits for most toxic heavy metals

Heavy metal	Impact	USEPA limit (mg/l)
Arsenic (As)	Skin manifestations, visceral cancers, vascular disease	0.05
Cadmium (Cd)	Kidney damage, renal disorder, human carcinogen	0.01+
Chromium (Cr)	Headache, diarrhea, nausea, vomiting, carcinogen	0.05
Copper (Cu)	Liver damage, Wilson disease, insomnia	0.25
Nickel (Ni)	Dermatitis, nausea, chronic asthma, coughing, human carcinogen	0.20
Zinc (Zn)	Depression, lethargy, neurological signs and nervous system	0.80
Lead (Pb)	Damage the fetal brain, diseases of the kidneys, circulatory system and nervous system	0.006+
Mercury (Hg)	Rheumatoid arthritis, diseases of the kidneys, circulatory system and nervous system	0.00003+

+ = extremely toxic compared to others [14]


(a) (b) (c)

Figure 1. Plate (a). *Citrullus colocythis* fruits, plate (b). *Citrullus colocythis* seeds, plate (c) *Citrullus colocythis* seed husks

impurities were removed by thorough washing the husks with distilled water while size reduction was achieved by milling and sieving the washed husks after draining and oven drying at 105ºC for 12 hours.

Modification with phosphoric acid (H_3PO_4) was accomplished by soaking 2g of the milled, washed and dried husks of melon (*Citrullus colocynthis)* in 2 ml of 0.1M H_3PO_4 for a period of 12 hours in a crucible. Thereafter, the crucible and its content were heated at 500ºC in a muffle furnace for 15 minutes. This was followed by washing it with 0.5M sodium bicarbonate ($NaHCO_3$) until the pH of the liquor was fairly neutral within the range of 6-7. It was further pulverized and sieved through 250µm mesh after rinsed with distilled water and oven dried at 105ºC for 24 hours. The 250µm sieved particles were stored in an airtight container as melon husk modified with H_3PO_4. Similarly, modification with sodium hydroxide (NaOH) was attained by oven heating 500g of the ground melon husk continuously in 0.5M NaOH for a period of 6 hours and then, filtered and washed thoroughly with distilled water until the supernatant became clear. Afterward, it was soaked in 1% formaldehyde (CH_2O) at 50ºC for a period of 4 hours to prevent any further colour interference during activation [15]. Excess CH_2O was removed by rinsing with distilled water, drained and oven dried for 24 hours at 80ºC. The ground treated husk was further pulverized and 250µm sieve particles were collected and stored in an airtight container. However, modification with urea (CH_4N_2O) was accomplished by soaking 100g of the ground melon husks in 10% CH_4N_2O solution for 24 hours. It was filtered and sun-dried for a period of one week for 6 hours on daily basis thereafter, further milling (grinding) was done and 250µm sieve particles were collected and stored in an airtight container for utilization.

Collection of adsorbates

The adsorbate used in this research was tannery effluents obtained in sample bottles at the Nigerian Institute of Leather and Science Technology, Zaria (11°9'26.65"N, 7°39'24.81"E). However, filtration of the tannery effluents was done before collection into the sample bottles in order to remove suspended particles. Samples were labeled and transported in a cooler containing ice to the laboratory thereafter, they were stored in a refrigerator until analysis. This was done in order to prevent bacterial proliferation which could alter the result [16]. Prior to sampling, the sample bottles were washed with 10% nitric acid (HNO_3), thoroughly rinsed with distilled water and allowed to dry. Notwithstanding, the bottles were further rinsed with the sample been sampled [17].

Adsorbate (effluent) treatment

The adsorbates (tannery effluents) were taken out from the refrigerator and allowed to attain room temperature (20-25ºC) thereafter, it was thoroughly agitated and 250ml was poured into a conical flask. A weighing balance (FA/JA series, made by HANNA LTD, England) was used in measuring 3g of the adsorbent into the conical flask and it was again agitated for proper mixing of both adsorbate and adsorbent. A set of 11 conical flasks of capacity 100ml (labelled as 0, 10, 20, 30, …, 100) were arranged and 20ml of the mixture were measured into each of the conical flasks arranged. The first 20ml sample labelled ‘0’ was quickly filtered at zero (0) minute interval using filter paper (whatman). This was done in order to serve as control for the sake of determining initial concentration of adsorbate (i.e. at time t = 0). Adsorption of metals in adsorbate by adsorbent have been reported to be affected by duration of stirring the mixture [15]. Hence, the remaining 20ml samples were stirred simultaneously in different magnetic stirrers at different duration as 10, 20, 30, …, 100 min, respectively.

Adsorbent dosages were varied at 0.2, 0.4, 0.6, 0.8 and 1.0g at constant adsorbate concentration and stirring duration (contact time). Likewise, concentrations of the adsorbate (effluent) were varied (using distilled water) at constant dose of adsorbent and stirring duration. The concentrations of Pb^{2+} and Cd^{2+} in all the experimental conditions for the treated adsorbate (effluents) were determined through atomic adsorption spectrometer (model number: AAS 500, made by Fredonia Ltd, USA).

Adsorption isotherm studies

Freundlich and Langmuir models are known to be the most commonly used isotherms models on activated carbon in water and wastewater treatment [18, 19]. Hence, this research considered the said models on the adsorption isotherm studies. The amount of metallic ions adsorbed on the various modified adsorbents (q_t) was determined through Equation (1) as stated below:

$$q_t = \frac{V}{M}(C_0 - C_t) \qquad (1)$$

In equation (1), V is the volume of adsorbate in litres, M is the mass of adsorbent in gram, C_o is the initial concentration of metallic ions in adsorbate in mg/l and C_t is the concentration of metallic ions remaining in adsorbate at a given time t in mg/l. In addition, the concentrations of adsorbate observed not to have changes with time in the various adsorption processes were recorded as equilibrium concentrations (c_e). The adsorption isotherm governing the removal of both metallic ions (Pb^{2+} and Cd^{2+}) from the adsorbate by the various modified adsorbents was known by plotting graphs of $\log q_t$ against $\log c_e$ (for Freundlich model) and $\frac{1}{q_t}$ against $\frac{1}{C_e}$ (for Langmuir model). Thereafter, the best-fitted equations of the plots were compared with the linearized forms of Freundlich and Langmuir isotherm models, which lead to the determination of adsorption capacities. The model that gave higher values of coefficient of determination (R^2) was recorded as the adsorption isotherm governing the adsorption process. The linearized form of Freundlich isotherm model is given in Equation (2) as follows:

$$\log q_t = \frac{1}{n}\log c_e + \log k_f \qquad (2)$$

where: q_t is the amount of ions adsorbed on adsorbent in (mg/g), c_e is the equilibrium concentration (mg/l), k_f is the Freundlich constant, which indicate adsorption capacity of adsorbent (mg/g), n is the Freundlich constant which indicate adsorption intensity (g/L). On the other hand, the linearized form of Langmuir isotherm model is given in Equation (3) below:

$$\frac{1}{q_t} = \frac{1}{q_m k}\left(\frac{1}{C_e}\right) + \frac{1}{q_m} \qquad (3)$$

In Equation (3), q_t is the amount of ions adsorbed on adsorbent in (mg/g), c_e is the equilibrium concentration (mg/l), q_m is the monolayer adsorption capacity at equilibrium (mg/g) and k is the Langmuir equilibrium constant (l/mg).

Adsorption kinetics studies

The amount of ions adsorbed at equilibrium (mg/g) at different contact time t were determined and recorded as q_e through Equation (4) stated below:

$$q_e = \frac{V}{M}(C_0 - C_e) \qquad (4)$$

All symbols in Equation (4) remain the same as previously explained. Graphs were plotted based on the linearized forms of Pseudo 1st and 2nd order kinetics. The rate constants of adsorption were determined by comparing the equation of the best-fitted plots with the linearized forms of Pseudo 1st and 2nd order kinetics given in Equations (5) and (6), respectively.

$$\log(q_e - q_t) = -k_1 t + \log q_e \qquad (5)$$

$$\frac{t}{q_t} = \frac{1}{q_e}(t) + \frac{1}{k_2 {q_e}^2} \qquad (6)$$

In Equations (5) and (6), k_1 and k_2 are the rate constants of Pseudo 1st and 2nd order adsorption in min^{-1} and $g.mg^{-1}.min^{-1}$, respectively; while, other symbols remain the same as previously explained. The order of kinetics that governs the adsorption process was identified through the coefficient of determination (R^2) values associated with the equation of best-fitted plots.

RESULTS AND DISCUSSION

Freundlich adsorption isotherm

The adsorption capacity (k_f) and intensity (n) of the various adsorbent used were determined by plotting $\log q_t$ (amount of ions adsorbed on adsorbent in mg/g) against $\log c_e$ (equilibrium concentration in mg/l) for the various adsorbent applied. This is demonstrated in Figure 2 by considering Pb^{2+} adsorbed by melon husk modified with phosphoric acid (H_3P_4O).

By comparing the equation displayed in Figure 2 and the standard Freundlich isotherm model given in Equation (2), implies that the gradient $\left(\frac{1}{n}\right)$ of Equation (2) is equivalent to 0.5681 in Figure 2. That is; $\frac{1}{n} = 0.5681$ and $n = 1.76$

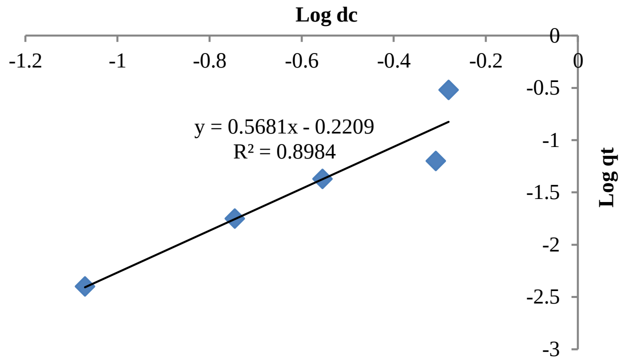

Figure 2. Freundlich isotherm model for adsorption of Pb^{2+} by melon husk modified with H_3PO_4

Similarly, the intercept $\left(\log k_f\right)$ of Equation (2) is equivalent to the intercept of the equation displayed in Figure 2. That is; $\log k_f = -0.2209 => k_f = 10^{-0.2209} = 0.6$

In other words, the adsorption capacity (k_f) of melon husk modified with phosphoric acid (H_3PO_4) on Pb^{2+} is 0.6 mg/g while the intensity (n) is 1.76 g/l. The Freundlich constants k_f and n for the other adsorbents were calculated in the same way and their values are summarized in Table 2.

Langmuir adsorption isotherm

The Langmuir monolayer adsorption capacity (q_m) and the equilibrium constant (k) of the adsorbents were obtained by plotting the reciprocals of the amount of ions adsorbed $\left(\frac{1}{q_t}\right)$, against the reciprocals of equilibrium concentration $\left(\frac{1}{c_e}\right)$. This is illustrated in Figure 3 by considering the case of Pb^{2+} adsorbed by melon husk modified with phosphoric acid (H_3PO_4).

Comparing the equation displayed in Figure 3 and the standard Langmuir isotherm model given in Equation (3), it could be inferred that the intercept; $\frac{1}{q_m} = 4.1929 => q_m = 0.2385$

Also, the gradient; $\frac{1}{q_m k} = 34.9992 \quad => k = \frac{1}{34.9992 q_m} = \frac{1}{34.9992(0.2385)} = 0.1198$

In other word, the Langmuir monolayer adsorption capacity (q_m) and equilibrium constant (k) for Pb^{2+} adsorbed by melon husk modified with urea (H_3PO_4) are 0.2385 mg/g and 0.1198 l/mg, respectively. The Langmuir constants (q_m and k) of all the adsorbents considered were determined in the same way and their values are shown in Table 3.

By comparing Figures 2 and 3, it is conspicuous that the data points fitted more into the Freundlich model than Langmuir model. This observation was also noted for the other experimental conditions. thus, buttressing the reason why higher values of coefficient of determination (R^2) were recorded in Table 2 compared to Table 3. In other words, the reason why the data points in Figure 3 (Langmuir model) did not fit into the regression line is because the adsorption process followed Freundlich isotherm model not Langmuir isotherm model. This is in line with previous research [20]. Hence, the adsorption of Pb^{2+} and Cd^{2+} occurred on heterogeneous surfaces not homogenous monolayer. Furthermore, the adsorption capacities for both Freundlich (k_f) and Langmuir (q_m) models were higher in Pb^{2+} than Cd^{2+} under the same condition thus, suggesting that Pb^{2+} were more easily adsorbed by the different modified melon husks. This affirmed the assertions of some earlier related studies [21, 22].

Pseudo-1st order adsorption kinetics

The rate constant (k_1) of Equation (5) was determined by plotting $\log(q_e - q_t)$ against contact time t and the gradient obtained was equated to $-k_1$. This is illustrated in Figure 4 by considering Pb^{2+} adsorbed by melon husk modified with phosphoric acid (H_3PO_4). The comparison between the standard linearized model for Pseudo-1st order kinetics given in Equation (5) and the equation displayed in Figure 4 revealed that; $-k_1 = -0.0128$ i.e. $k_1 = 0.0128$

Hence, the rate constant (k_1) of Pseudo-1st order kinetics for Pb^{2+} adsorbed by melon husk modified with phosphoric acid (H_3PO_4) is 0.0128 min^{-1}. The same method was employed in all the adsorbents for both metal ions (Pb^{2+} and Cd^{2+}) and their rate constants (k_1) as well as coefficients of determination (R^2) are shown in Table 4. However, the data

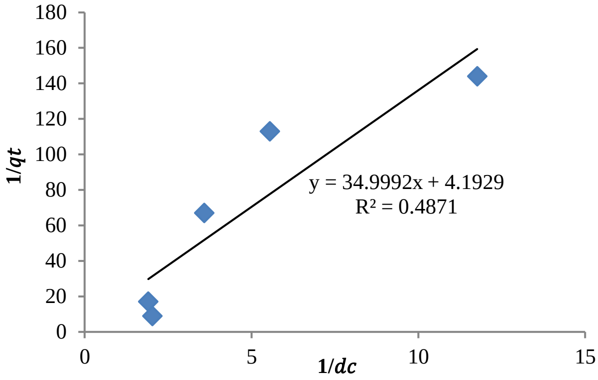

Figure 3. Langmuir isotherm model for adsorption of Pb^{2+} by melon husk modified with H_3PO_4

TABLE 2. Freundlich adsorption constants for melon husks modified with H_3PO_4, NaOH and CH_4N_2O

	Lead ions (Pb^{2+})			Cadmium ions (Cd^{2+})		
	k_f (mg/g)	n (g/l)	R^2	k_f (mg/g)	n (g/l)	R^2
H_3PO_4	0.6013	1.7603	0.8984	0.4417	1.6111	0.7601
NaOH	0.4644	1.4914	0.8433	1.2650	1.1527	0.4709
CH_4N_2O	0.3809	1.3305	0.9713	0.3169	1.0204	0.7028

TABLE 3. Langmuir adsorption constants for melon husks modified with H_3PO_4, NaOH and CH_4N_2O

	Lead ions (Pb^{2+})			Cadmium ions (Cd^{2+})		
	q_m (mg/g)	k (l/mg)	R^2	q_m (mg/g)	k (l/mg)	R^2
H_3PO_4	0.2385	0.1198	0.4871	0.1962	0.1131	0.5733
NaOH	0.1874	0.1129	0.3426	0.1551	0.1086	0.6892
CH_4N_2O	0.1499	0.1106	0.6612	0.1403	0.1002	0.5545

TABLE 4. Pseudo-1st order rate constants for Pb^{2+} and Cd^{2+} sorption by modified melon husks

	Lead ions (Pb^{2+})		**Cadmium ions (Cd^{2+})**	
	k_1(min^{-1})	**R^2**	**k_1(min^{-1})**	**R^2**
H_3PO_4	0.0128	0.2391	0.0042	0.0164
NaOH	0.0062	0.0324	0.0016	0.0022
CH_4N_2O	0.0055	0.0272	0.005	0.0136

points in all the cases involving Pseudo-1st order kinetics, did not fit into their various regression lines as could be seen in Figure 4. This made the associated R^2 values shown in Table 4 to be very small. Since the data points did not fit into the regression line, it suggests that the adsorption process did not follow Pseudo-1st order kinetics.

Pseudo-2nd order adsorption kinetics

The gradient and intercept of Equation (6) were determined through the plot shown in Figure 5. The comparison between the standard linearized model for Pseudo-2nd order kinetics given in Equation (6) and the equation displayed in Figure 5, inform that the gradient; $\frac{1}{q_e} = 13.698$ and $q_e = 0.073$

Similarly, the intercept; $\frac{1}{k_2 {q_e}^2} = 50.087 \;=>\; \frac{1}{k_2(0.0730^2)} = 50.087$; $k_2 = \frac{1}{(0.0730^2)50.087} = 3.7465$

In other words, the Pseudo-2nd order rate constant (k_2) for the adsorption of Pb^{2+} by melon husk modified with phosphoric acid (H_3PO_4) is 3.7465 g.mg^{-1}.min^{-1}. The Pseudo-2nd order rate constants for the remaining adsorbents were determined in likewise manner and their values are summarized in Table 5. Unlike Figure 4 (Pseudo-1st order kinetics), the data points in Figure 5 (Pseudo-2nd order kinetics) fitted well in the regression line. Consequently, it explains the reason why the associated R^2 value in Figure 5 is high. This observation was also noted in the remaining adsorbent as could be seen in Table 5. In other words, the fitness of the data points in the regression line for cases involving Pseudo-2nd order kinetics simply implies that the adsorption process in the various adsorbent obeyed Pseudo-2nd order rate expression, which is in consonance with previous observations [20–23].

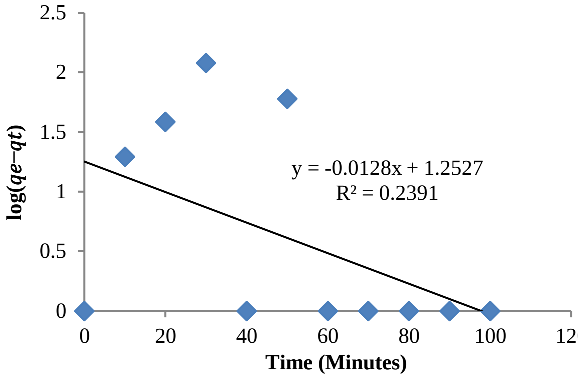

Figure 4. Pseudo-1st order kinetics for Pb^{2+} adsorbed by melon husk modified with H_3PO_4

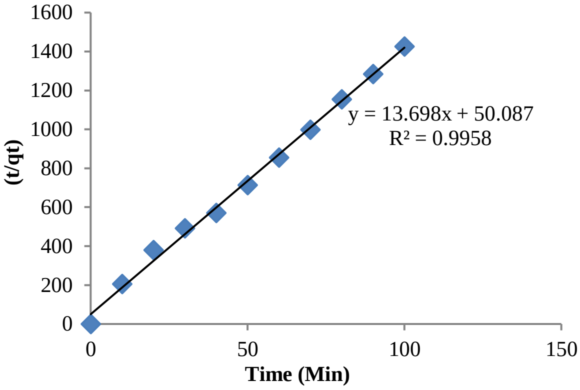

Figure 5. Pseudo-2nd order kinetics for Pb^{2+} adsorbed by melon husk modified with phosphoric acid (H_3PO_4)

TABLE 5. Pseudo-2nd order rate constant for Pb^{2+} and Cd^{2+} sorption by modified melon husks

	Lead ions (Pb^{2+})		**Cadmium ions (Cd^{2+})**	
	k_2(g.mg^{-1}.min^{-1})	**R^2**	**k_2(g.mg^{-1}.min^{-1})**	**R^2**
H_3PO_4	3.7465	0.9319	0.09431	0.6224
NaOH	0.8784	0.9958	11.1857	0.7657
CH_4N_2O	1.0136	0.9112	11.6174	0.7043

CONCLUSION

On the basis of the analyzed data, it could be concluded that melon husk modified with either H_3PO_4, NaOH or CH_4N_2O could be effectively used in the removal of Pb^{2+} and Cd^{2+} from wastewater as their adsorption capacities were quite high. However, modification with H_3PO_4 performed best. The adsorption of Pb^{2+} and Cd^{2+} by melon husk modified with either H_3PO_4, NaOH or CH_4N_2O followed Freundlich isotherm model as well as Pseudo-2nd order adsorption kinetics. In addition, melon husks modified with either H_3PO_4, NaOH or CH_4N_2O adsorbed more lead ions (Pb^{2+}) than cadmium ions (Cd^{2+}) under the same experimental condition. Hence, it is recommended that whenever the need of using modified melon husk to adsorb metal ions from effluent arises, modification with H_3PO_4 should be considered first before NaOH and CH_4N_2O.

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Persian Abstract

DOI: 10.5829/ijee.2020.11.02.10

چکیده

رشد جمعیت موجب افزایش متناظر تولید محصولات کشاورزی شده که در نتیجه منجر به افزایش ضایعات یا فرآورده‌های کشاورزی شده است. مدیریت این مسئله گاهی اوقات دشوار است و اگر به درستی انجام نشود می‌تواند تهدیدی برای جامعه ایجاد کند. از این رو، این پژوهش به بررسی اثربخشی استفاده از فرآورده‌های جانبی کشاورزی (پوسته خربزه) در حذف Pb^{2+} و Cd^{2+} از فاضلاب‌ها پرداخته تا از تأثیرات مضر آن‌ها بر اکوسیستم و بهداشت عمومی جلوگیری شود. این کار با استفاده از تصفیه جریان‌های شامل پوسته‌های خربزه، اصلاح شده با H_3PO_4، NaOH و CH_4N_2O حاصل شد. نتایج نشان داد که ظرفیت جذب ایزوترم Freundlich و Langmuir در پوسته‌های خربزه اصلاح شده به ترتیب از ۰/۱۴۰۳-۱/۵۵۱ mg/g و ۰/۱۴۹۹-۰/۶۰۱۳ mg/g می‌باشد. همچنین مقادیر R^2 برای ثوابت مرتبه شبه اول و دوم به ترتیب در محدوده ۰/۰-۲۳۹۱/۰۲۷۲ و ۰/۹۹۵۸-۰/۹۱۱۲ بود. از این رو، می‌توان نتیجه گرفت که پوسته‌های خربزه اصلاح شده از مدل ایزوترم فروندلیچ و همچنین معادله سینتیک مرتبه شبه دوم تبعیت می‌کنند و می‌توانند به طور موثری برای حذف Pb^{2+} و Cd^{2+} از فاضلاب استفاده شوند.