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A Statistical Analysis of Experimental Data for the Adsorption Process of Cadmium by Watermelon Rinds in a Continuous Packed Bed Column

Mohammed Nsaif Abbas[a], **Taymoor Husham Nussrat**[b], [a]Environmental Engineering Dept. College of Engineering- Mustansiriayh University -Iraq, [b]Financial & Banking Dept., Baghdad College of Economic Science University-Iraq,

The aim of this research is to study the ability of cadmium removal from simulated synthetic aqueous solution (SSAS) by watermelon rinds residues, at different operating conditions and using the adsorption technique in a packed bed treatment unit of continuous mode. The results show that the maximum adsorption capacity of watermelon rind was increased with increasing the cadmium initial concentration (Co), flowrate (Q), and pH of the SSAS. Furthermore, it is decreased with increasing the adsorbent bed height (H), and feed temperature (T). The complete removal of cadmium was achieved at optimum operating conditions, which were Co=200 ppm, Q=0.5 l/min, pH=6, H=20 cm, and T=40°C, at t=231 minutes. The experimental results were analysed statistically using three mathematical models, namely the Adams-Bohart, Wang, and Thomas models to describe the adsorption breakthrough curve behaviour of the cadmium in the packed bed column, and also to determine the rate-limiting step depending on the statistical measurements (correlation coefficient R2). The results show that the Thomas model provides excellent identical with experimental data, followed by the Wang model, and lastly, the Adams-Bohart model. Thus, it can be considered that the watermelon rinds are an effective adsorbent to remove heavy metals generally, and cadmium especially, from aqueous solutions. It can also be used as an effective, cheap, and economic alternative substance for the expensive activated carbon used recently in wastewater treatment processes.

Keywords: *Statistical analysis, Cadmium, Adsorption, Correlation coefficient R2, Thomas model.*

Nomenclature

A: Area of packed bed column (m2)
$C_{\circ}$: Concentration in the influent (mg. l-1)
C: Concentration of cadmium at any time t (mg. l-1)
H: Bed depth of the fixed-bed column (m)
k_{AB}: Adams-Bohart kinetic rate constant (l.mg-1.min-1)
k_{Th}: Thomas kinetic rate constant (l.mg-1.min-1)
k_{W}: Wang kinetic rate constant (min-1)
m: Mass of watermelon peels used in experiment (g)
$N_{\circ}$: Maximum adsorption capacity (mg. l-1)
pH: Acidity function of aqueous solution (-)
q: Cadmium adsorbate uptake by the adsorbent at any time (mg.g-1)
q_{max}: Maximum Cadmium adsorbate uptake by the adsorbent at exhaustion time t_e (mg.g-1)
Q: Volumetric flowrate of SSAS feed to the adsorption unit (l.min-1)
R^2: Correlation Coefficient (-)
T: Temperature of aqueous solution (°C)
t: Treated time (min)
t_e: Exhaustion time (min)
$t_{(1/2)}$: The time required for 50% adsorbate breakthrough (min)

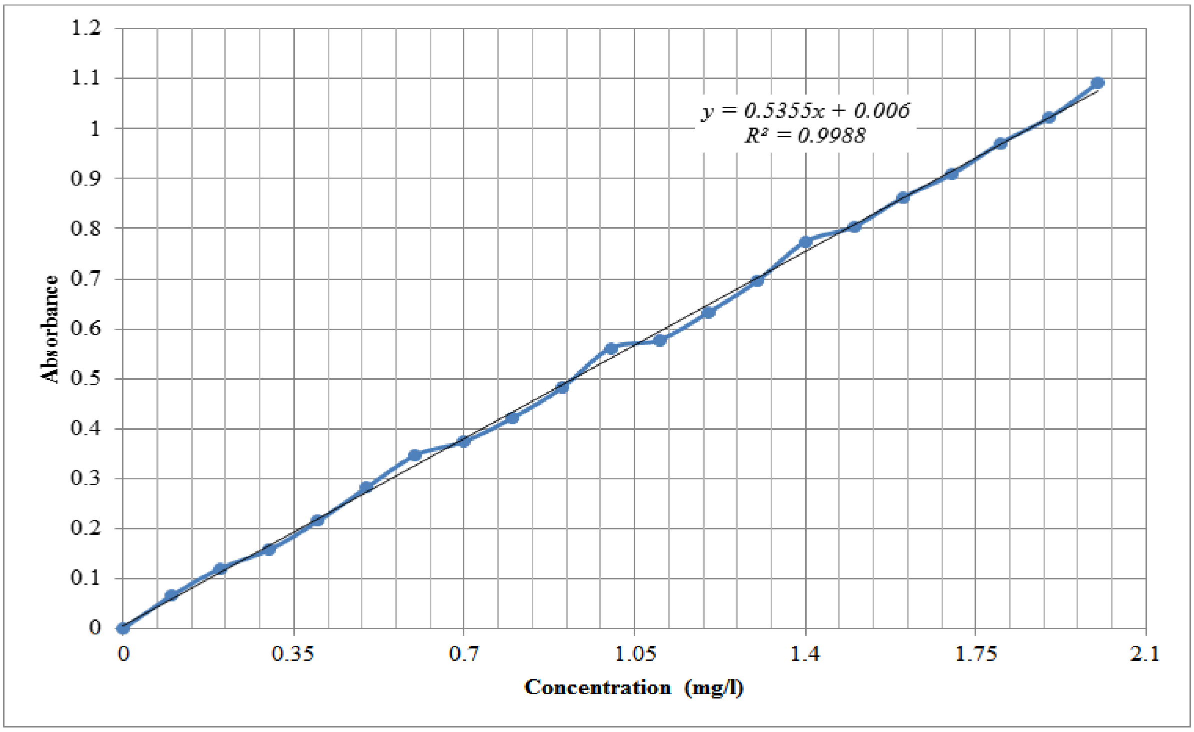
Introduction

Cadmium is a metal of white ranging to silver colour, that is soft meltable and fast-soluble in acids but is not soluble in bases, similar to zinc and tin metals. It is predominantly found wherever zinc is present, so it is often found in zinc alloys (Walker & Tarn, 1990). Scientists reported that cadmium is a modern metal and it was discovered by the two German chemists, Friedrich Stromeyer and Karl Samuel Leberecht Hermann, as an element in 1817 (Cobb, 2012). More than 50 years ago, its use in industry was only secondary or marginal in manner, however, at present, it has become an important metal used in large fields, such as electroplating, where objects are plated due to its properties of corrosion and rust resistance (Sigel, 2013). Cadmium is used in the manufacture of alloys, welding materials and coating metals, and as dyes, especially in certain colour names used in artistic drawing. Furthermore, it can be used as a fixation material in plastic materials, and the main use of cadmium is in long life batteries (Hasanuzzaman & Fujita, 2013). These activities are considered the main sources of cadmium consumption, while at the same time, it is also considered as the main source of cadmium effluent to the water resources (Abbas & Abbas F., 2013). Biologically, cadmium is classified as a non-necessary metal for life, i.e. non-necessary for all body functions. On the other hand, the cadmium, when entering to the human body, even in trace concentrations, is accumulated in the kidneys and liver. This leads to renal failure, hypertension, anaemia, and itai-itai disease, not to mention, its side effects on male fertility, backbone, and gastrointestinal tract (Lane et al., 2015).The maximum accepted concentration of cadmium in drinking water, according to the World Health Organization, is 0.003 ppm (Khitous & Kherat, 2015). Thus, it is essential to reduce and remove this toxic heavy metal from water as soon as possible because of its half-life reach of more than ten years in some isotopes (Richter & Pappas, 2017). Many methods and techniques are suggested and used to treat polluted water with heavy metals in general, and with cadmium especially, such as precipitation, coagulation-flocculation, ultra-filtration, advance-oxidation, ion-exchange, etc. However, the main method which exhibits efficiency, and feasibility, and is economical, is adsorption (Abbas et al., 2019). At present, this method uses non-valuable adsorbent material, such as industrial wastes like aluminium foil (Ghulam & Sachit, 2019), lemon peel (Alalwan & Alminshid, 2019), and agricultural residues, such as rice husk (Abbas, 2013), banana peel (Abbas & Ibraheem, 2014), eggshell (Abbas & Alalwan, 2019), watermelon rind (Khitous & Kherat, 2015), and so on. The adsorption technique can be performed by the aforementioned wastes using a two-part procedure, batch and continuous mode. Many models are suggested to describe the behaviour of polluted molecule transfer through the adsorbent media to obtain the optimum conditions needed to remove the toxic material with a simple, efficient, and economic route (Abbas & Husein, 2019). The main models used to explain the adsorption process by the adsorbents are Langmuir, Freundlich, Temkin, Dubinin-Radushkevich, and BET. All these models show the relationship between the adsorbed amounts with the concentration of the adsorbed substance (Chen & Shammas, 2016). The same is needed in the adsorption process which is achieved by a continuous unit. However, the model, in this case, is more developed and depends, in some cases, on the above models, especially the Langmuir, and Freundlich models (Xu & Pan, 2013). The famous models used in the continuous mode are the Adams-Bohart, Thomas, Wang, Clark, and Yoon-Nelson Models. In this paper, the main goal is to represent the best continuous models that describe the time-concentration profile for the cadmium adsorption process from a simulated synthetic aqueous solution (SSAS) that is carried out by non-treated watermelon rinds. Furthermore, to find the kinetic parameters of these models to determine the best operating conditions for the adsorption process.

Experimental Procedure

The adsorption experimental data was obtained via achieving multiple sets of cadmium adsorption experiments using watermelon peel as the adsorbent material. Different operating conditions were used, which included the initial concentrations of cadmium in SSAS (C_o), pH of SSAS (*pH*), the height of adsorbent material (*H*), volumetric flowrate of SSAS entering to the adsorption unit (*Q*), and temperature of SSAS (*T*), which varied between 25–200 mg/l, 2–8, 0.5–0.8 l/min, and 25–40 °C, respectively. All experiments were carried out in an adsorption unit of down-flow continuous mode, and the adsorbent material (watermelon peels of density 0.35 g/cm^3) was used without any further treatment. The concentration of cadmium in the solutions, before and after the adsorption process, were measured using atomic adsorption spectroscopy (AAS). Firstly, 45 different tests were accomplished at random values of operating conditions selected between the above ranges to determine the optimum operating conditions that provide the best cadmium adsorption by the watermelon peel. Furthermore, the adsorption experiments were completed by the following procedure. A stock solution of 1000 mg/l of cadmium was prepared by dissolving the required amount of cadmium powder (100 mesh, 99.5 per cent trace metal basis, BDOSMKKIYDKNTQ-UHFFFAOYSA-N Sigma Aldrich) in double distilled water. To obtain the precise concentration of cadmium necessary to perfume the experiment, five litres of SSAS was prepared from a stock solution, according to dilution calculations. Its pH was adjusted by 0.1N NaOH and 0.1N HCl solutions. It was then heated to the required temperature and charged to an isolated storage tank. A clean packed bed column of 8cm in diameter and 20cm in height was filled with carefully washed watermelon peel of approximately 0.5×0.5 cm in size, to a desired height. The peel layer was separated from the top and bottom by two layers; one of fibre glass, and the other of glass beads, to obtain uniform distribution of the SSAS. The column was then connected with an isolated storage tank of five litres in capacity via a booster pump. The flowrate was adjusted to a suitable value by rotameter measurement. The experiment began by allowing the SSAS to penetrate the packed bed column at the desired parameters and have contact with the watermelon peel chips for a determined period. The samples were withdrawn from the bottom of the column each minute, and the concentration of cadmium was measured by AAS (PerkinElmer PinAAcle™ 900H atomic absorption) at a wavelength of 228.8nm, and according to the calibration curve illustrative in Figure 1.

Figure 1. Atomic Absorption Spectroscopy Calibration Curve of Cadmium

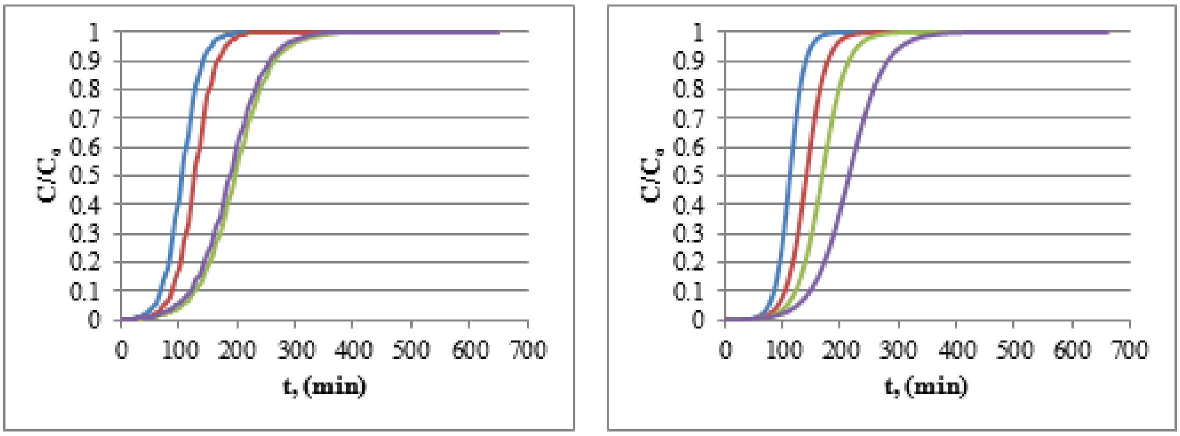


The experiment was continued until the effluent cadmium concentration (C_o) approach to the inlet concentration (C_i). Then, each breakthrough model set was performed using 20 experiments with 3–5 replicates by varying one of the operating parameters and keeping the other parameters constant and at the optimum conditions. The details of the performed experiments are explained in Table 1 and Figure 2. The deviations of the experiments were around three per cent, and the mean values were used in the statistical analysis.

Table 1: Experimental Operation Conditions used for Cadmium Adsorption in Continuous Packed Bed Column

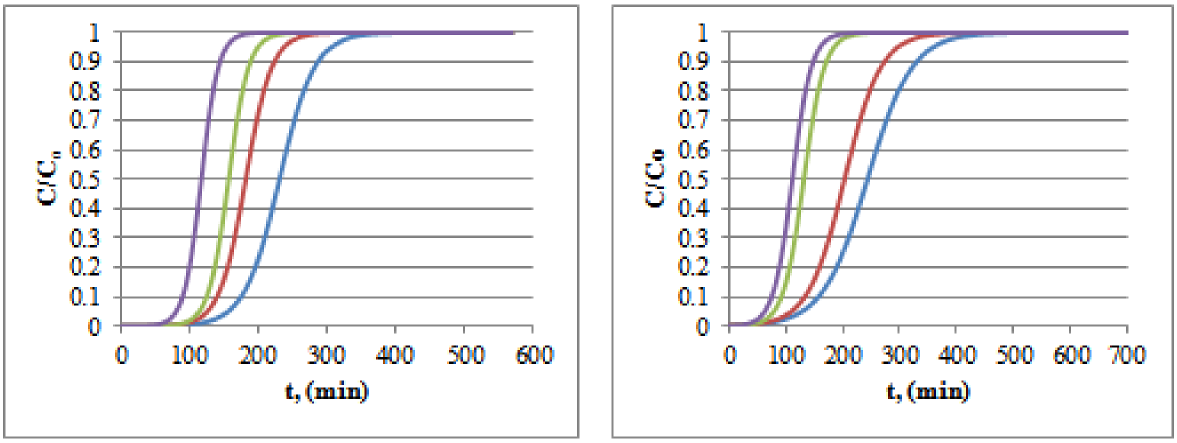
H (cm)	**Q (l/min)**	**C_o (mg/l)**	**pH**	**T (°C)**	**C_b (mg/l)**	**t_b (min)**	**q_b (mg/g)**	**t_e (min)**	**q_e (mg/g)**	**q_{exp} (mg/g)**
5	0.5	25	6	40	2.5	55	7.868976	247	37.19880	37.25
10	0.5	25	6	40	2.5	78	5.480769	349	25.81361	25.85
15	0.5	25	6	40	2.5	113	5.282972	491	24.16339	24.20
20	0.5	25	6	40	2.5	135	4.646739	586	21.23188	21.25
20	***0.5***	25	6	40	2.5	157	5.403986	605	21.92029	22.00
20	***0.6***	25	6	40	2.5	133	5.493478	496	21.56522	21.50
20	***0.7***	25	6	40	2.5	92	4.433333	297	15.06522	15.00
20	***0.8***	25	6	40	2.5	75	4.130435	264	15.30435	15.30
20	0.5	***25***	6	40	2.5	175	6.023551	466	16.88406	17.00
20	0.5	***50***	6	40	5	140	9.637681	357	25.86957	26.00
20	0.5	***100***	6	40	10	125	17.21014	291	42.17391	42.00
20	0.5	***200***	6	40	20	90	24.78261	231	66.95652	67.00
20	0.5	25	***2***	40	2.5	69	2.375	258	9.347826	9.500
20	0.5	25	***4***	40	2.5	88	3.028986	293	10.61594	10.65
20	0.5	25	***6***	40	2.5	128	4.405797	484	17.53623	17.50
20	0.5	25	***8***	40	2.5	119	4.096014	475	17.21014	17.25
20	0.5	25	6	***25***	2.5	85	2.925725	228	8.260870	8.250
20	0.5	25	6	***30***	2.5	104	3.57971	295	10.68841	10.75
20	0.5	25	6	***35***	2.5	122	4.199275	361	13.07971	13.00
20	0.5	25	6	***40***	2.5	147	5.059783	496	17.97101	18.00

Figure 2. Effect of Operation Conditions on the Breakthrough Curve of Cadmium Adsorption by Watermelon Peel (experimental results)



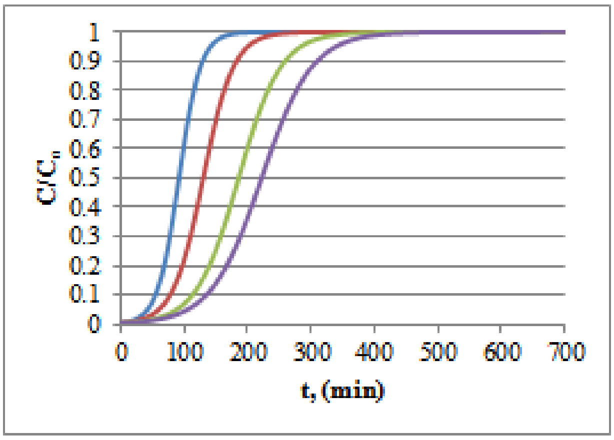
Temperature

pH



Concentration

Flowrate



Height of Adsorbent Media

Dynamic Study of Packed Bed Column

The dynamic behaviour of the packed bed column can be determined meticulously due to the concentration-time distribution (i.e. breakthrough curve) of the experimental data, and also of different mathematical models **(**Alalwan & Alminshid, 2018). The breakthrough curve is defined as the shape that illustrates the path of the following adsorbate concentration in the packed bed column (Larsson & Tsang, 2012). This shape of the breakthrough curve plays a vital role to clarify the attitude of the solid-liquid transfer process in the adsorption unit. In general, any adsorption process (adsorbate–adsorbent system) can be described by four diffusion steps: the ***molecular diffusion*** of adsorbate mass in the fluid phase (liquid or gas); ***film or interface diffusion*** between adsorbate and the outer surface of solid phase (adsorbent); ***surface diffusion*** of adsorbate at adsorbent material surface; and ***pore diffusion*** according to mass transfer inside the solid phase pores (adsorbent) **(**Abbas, 2015). In this study, three mathematical models were applied for describing the behaviour of the cadmium removal process by the adsorption technique in the treatment unit of continuous mode and at the same time, predict the kinetic parameters of the packed bed column **(**Abbas, 2009 & 2012). By conducting a statistical analysis of the mathematical description and kinetic parameters for those models, it can guess the step of rate limiting for the adsorption process and determine which of these models are comparative with the experimental data. The mathematical models used to describe the concentration-time profile via a correlation between ($C/C_{\circ}$) and t, were the Thomas, Adam–Bohart, and Wang models. The linear and non-linear equations that explain these models are listed in Table 2.

Table 2: General and Linear Forms of Mathematical Models Used

No.	**Model's Name**	**General (Non-linear) form**	**Linear Form**
1.	Adams-Bohart	$\frac{C}{C_{\circ}} = \exp\left(k_{AB}C_{\circ}t - k_{AB}N_{\circ}\frac{HA}{Q}\right)$	$\ln\left[\left(\frac{C_{\circ}}{C}\right)\right] = k_{AB}N_{\circ}\frac{HA}{Q} - k_{AB}C_{\circ}t$
2.	Wang	$\frac{C}{C_{\circ}} = 1 - \exp\left[k_W\left(t - t_{\frac{1}{2}}\right)\right]$	$\ln\left[\frac{1}{1-\left(\frac{C}{C_{\circ}}\right)}\right] = k_W t_{\frac{1}{2}} - k_W t$
3.	Thomas	$\frac{C}{C_{\circ}} = \left[\frac{1}{1+\exp\left(\frac{k_{Th}q_{max}m}{Q} - k_{Th}C_{\circ}t\right)}\right]$	$\ln\left[\left(\frac{C_{\circ}}{C}\right) - 1\right] = \frac{k_{Th}q_{max}m}{Q} - k_{Th}C_{\circ}t$

The amount of cadmium eliminated by the packed bed column can be determined by calculating the area under the breakthrough curve from equation 1:

$$q = \frac{Q}{m}\int_{0}^{t_e} C dt \qquad (1)$$

Results and Discussions

1. The Adams–Bohart model is one of the most famous models used for the determination of the packed bed column breakthrough curves and kinetic parameters. Due to this model, the saturation state is cumbersome, and the rate of absorption is proportional to the adsorbent material capacity. The assumption of the Adams–Bohart model, as is evident from the mathematical expression, is that the adsorption rate is related to the maximum adsorption capacity and the initial concentration of adsorbate in the solution. Further, this model is dependent on the height of adsorbent material and the velocity of solution in the packed bed column. In other words, it depends on the dimensions of the adsorption bed. The parameters of this model, $\boldsymbol{k_{AB}}$ and $\boldsymbol{N_\circ}$, can be calculated from the slop and intercept of the linear form of the Adams-Bohart model by plotting $\mathbf{ln}[(\boldsymbol{C_\circ}/\boldsymbol{C})]$ against $\boldsymbol{t}$. The values of the calculated $\boldsymbol{k_{AB}}$ and $\boldsymbol{N_\circ}$, are listed in Table 3. From this, it can show that the values of $\boldsymbol{k_{AB}}$ calculated according to this model increased with the increasing bed height, pH, and temperatures of the SSAS, meanwhile decreasing with the increasing initial concentrations and flowrate. However, the values of $\boldsymbol{N_\circ}$ were completely counter to the results of $\boldsymbol{k_{AB}}$, and it is very higher than the calculated values. It is obvious from the statistical measurement (R^2), which ranges between 0.3347–0.7914 for this model, that the Adam-Bohart model isn't well fitted with the experimental data obtained, so this model is not applicable to represent the cadmium removal using the adsorption process.

Table 3: Kinetic Parameters of Mathematical Models used According to Operating Conditions

Operating Parameters					**Kinetic Parameters**								
					Adams-Bohart Model			**Wang Model**			**Thomas Model**		
H (cm)	**Q (*l*/min)**	**C₀ (mg/l)**	**pH**	**T (°C)**	k_{AB}	$N_\circ$	R^2	k_W	$t_{1/2}$	R^2	k_{Th}	q_{max}	R^2
5	0.5	25	6	40	0.000144	1701.44	0.3347	0.0556	66.6547	0.9916	0.00237	13.8343	0.9954
10	0.5	25	6	40	0.000192	649.320	0.4597	0.0372	84.1317	0.9808	0.00168	9.62329	0.9981

15	0.5	25	6	40	0.000252	348.077	0.6220	0.0238	101.861	0.9593	0.00120	9.16106	0.9943
20	0.5	25	6	40	0.000280	234.934	0.7089	0.0182	109.907	0.9430	0.00101	8.06016	0.9975
20	***0.5***	25	6	40	0.000324	245.365	0.7432	0.0174	114.874	0.9306	0.00102	8.80364	0.9993
20	***0.6***	25	6	40	0.000308	275.226	0.6575	0.0239	107.226	0.9503	0.00126	8.82318	0.9955
20	***0.7***	25	6	40	0.000252	426.308	0.4392	0.0493	86.3083	0.9796	0.00222	6.66965	0.9982
20	***0.8***	25	6	40	0.000204	445.667	0.3822	0.0552	77.1685	0.9864	0.00241	6.45968	0.9968
20	0.5	***25***	6	40	0.000620	247.568	0.7651	0.0237	107.578	0.8837	0.00157	8.37049	0.9979
20	0.5	***50***	6	40	0.000288	586.403	0.6359	0.0382	98.2042	0.9243	0.00105	13.1699	0.9963
20	0.5	***100***	6	40	0.000148	1434.26	0.5563	0.0540	91.963	0.9433	0.00069	22.7235	0.9950
20	0.5	***200***	6	40	0.000053	3021.15	0.4364	0.0701	76.9971	0.9713	0.00040	33.9691	0.9994
20	0.5	25	***2***	40	0.000216	364.072	0.3865	0.0550	72.7164	0.9849	0.00242	3.81683	0.9998
20	0.5	25	***4***	40	0.000276	352.908	0.4507	0.0487	82.768	0.9761	0.00222	4.61911	0.9980
20	0.5	25	***6***	40	0.000332	239.150	0.6647	0.0238	102.513	0.9422	0.00128	7.11048	0.9973
20	0.5	25	***8***	40	0.000308	234.987	0.6458	0.0243	100.082	0.9477	0.00128	6.79937	0.9957
20	0.5	25	6	***25***	0.000516	311.956	0.4706	0.0670	72.0433	0.9600	0.00320	4.07688	0.9983
20	0.5	25	6	***30***	0.000560	253.658	0.5704	0.0456	81.9912	0.9335	0.00238	5.10256	0.9967

20	0.5	25	6	***35***	0.000596	219.627	0.6583	0.0329	89.5836	0.9045	0.00191	6.08483	0.9971
20	0.5	25	6	***40***	0.000580	172.321	0.7914	0.0182	97.3352	0.8578	0.00131	7.75573	0.9978

2. The Wang Model is a development model depending on a mass transfer phenomenon for describing the breakthrough curves in the adsorption process and through the packed bed column in a continuous system. The main assumption of this model is based on the isothermal adsorption process with uniform breakthrough. Further, this model is considered the mass transfer due to axial dispersion being neglected and assuming x is the fraction of the adsorption by the adsorbate, while y is the fraction of the adsorbate inside the packed bed. The assuming relation between y and x in this model is as follows: $x + y = 1$. The linear and non-linear forms of Wang's model are listed in Table 3. According to the linear form, it can be predicted by the two parameters of this model, k_W and $t_{1/2}$, by plotting $\mathbf{ln[1-(\boldsymbol{C}/\boldsymbol{C}_\circ)]}$ against $\boldsymbol{t}$. The values of the Wang model's kinetic parameters, which were $\boldsymbol{k_W}$ and $\boldsymbol{t_{1/2}}$ calculated, are listed in Table 3. From revision of the data in the Table 3, it is noticed that the $\boldsymbol{k_W}$ increased with increasing the height of the packing and the pH of the SSAS, while it is decreased with increasing the initial concentrations of cadmium, flowrates, and temperatures of the SSAS entering the solutions. In contrast with these results, $\boldsymbol{t_{1/2}}$ decreases when $\boldsymbol{k_W}$ increases, and vice versa, i.e the relation between them is inversed completely. It can be observed that there is a good agreement between the experimental data and the data calculated by the Wang model. The values of $\boldsymbol{t_{1/2}}$ are roughly equivalent to the experimental saturation time, and on the other hand, the statistical measurement is high and ranges between 0.8578–99.16. Therefore, these results mean the behaviour of the adsorption bed is an ideal plug flow style and the shape of the breakthrough curve is symmetrical. Despite these closely fitted and similar results, the Wang model cannot be applied to describe the performance of the packed bed column because it gives incomplete information about the adsorption process, unlike other models.

3. The Thomas model is the most renowned and simple model that describes the dynamic behaviour of the packed bed column breakthrough curves of the adsorption process. The linear and non-linear forms of the Thomas model are listed in Table 3. This model assumes that there is no axial, internal and external diffusions in the system, the flow of solution is the plug flow, and the adsorption process obeys the Langmuir theory, with second-order reversible kinetics. According to this model, the concentration-time distribution depends on the properties of the adsorbate material and the initial concentration of the adsorbent. The parameters of this model, which are

$\boldsymbol{k_{Th}}$ and $\boldsymbol{q_{max}}$, can be determined from the linear form equation by plotting $\mathbf{ln}[(\boldsymbol{C_{\circ}}/\boldsymbol{C}) - \mathbf{1}]$ against $\boldsymbol{t}$. The values of the calculated adsorbate capacity, according to this model, are very close to the real capacity and all the rest results are identical with the logical concepts of adsorption. Besides that, it is clear from the results listed in Table 3, that this model exhibits an excellent agreement with the experimental results and the values of the statistical measurement (R^2), which is higher than 0.9943 and indicates to this. This model provides a match with the experimental results better than the above two models. Therefore, this model is applicable to represent the obtained data.

Conclusions

The watermelon peels, without any pre-treatment, were prepared for use as adsorbent material to study the removal of cadmium from SSAS in a continuous packed adsorption unit and at different operating conditions of the initial concentration of cadmium, height bed of watermelon peels flowrate, pH, and temperature of SSAS. According to the experimental results obtained, this study provides the following conclusions:

1. In the continuous adsorption system, the removal efficiency depended on the pH, temperature, and flowrate of the SSAS, as well as on the bed height of the adsorbent, and in addition to the feed concentrations of the pollutant metal. It was noticed that the maximum adsorbent capacity was directly proportional with the initial concentration pH and temperature of the SSAS, and inversely proportional with both the bed height and the volumetric flowrate.
2. The watermelon peel, without any further treatment, exhibited a good behaviour in different operating conditions to remove cadmium from SSAS. The adsorption capacity reached a maximum value at 231 minutes. It was approached to 67 mg/g at Co=200 mg/l, Q=0.5 l/min, H=20 cm, pH=6, and T=40°C. Meanwhile, the lowest value was 8.26 mg/g, occurring in 228 minutes at Co=25 mg/l, Q=0.5 l/min, H=20 cm, pH=6, and T=25°C. Thus, the main parameters that have a direct effect on the adsorption process were the initial concentration and feed temperatures, besides the effect of the other variables.
3. The statistical measurement (R^2) was used in the affect method to determine the better model for describing the breakthrough curve of the removal process in the packed bed by the adsorption technique via a continuous unit. For this purpose, three models were used to achieve this mission, namely the Adams-Bohart, Wang, and Thomas models. The Thomas model described that the breakthrough curve occurred in the packed bed better than the Adams-Bohart, and Wang models. The Thomas model provides a perfect symmetry with the experimental data, according to the statistical measurement (R^2). Meanwhile, although the Wang model has a high (R^2) and detected the flow pattern in the bed precisely, it cannot describe the adsorption process in the column with sufficient

information. Finally, the Adams-Bohart model cannot be applied to show the behaviour of the adsorption of cadmium in the bed of watermelon peels packing because the statistical measurements (R^2) obtained were lower than the other models. Thus, it can say that the best model used to describe the performance of the packed bed column is the Thomas model.

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