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Equilibrium, Kinetics and Thermodynamic Studies of Adsorption of Cadmium(II) Ions unto Activated Carbon from African Elemi Seeds

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Abstract The need to control our environment from industrial waste water pollution has ignited interest in cost effective products to treat industrial effluents. Adsorption of cadmium (II) ions from wastewater using activated carbon from African elemi seeds was studied. Batch adsorption experiments were carried out to investigate the effect of pH, contact time, initial metal ion concentration, temperature and adsorbent dosage on the adsorption efficiency. Results showed that increasing all the parameters, with the exception of temperature, increases the adsorption capacity. The process followed the pseudo-second order kinetics model which showed chemical adsorption. The experimental data was fitted well by the Freundlich isotherm indicating adsorption on heterogeneous surface. Thermodynamic data (negative ΔG, negative ΔH and positive ΔS) showed that the process was feasible, spontaneous, and exothermic with an increase in the randomness at the solid/solution interface. The study revealed that activated carbon from African elemi seeds could be used as a low cost adsorbent for removal of cadmium (II) ions from wastewater.

Keywords Adsorption, Kinetics, Thermodynamics, Cadmium, Activated carbon

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1. Introduction

Industrial activities such as blacksmithing, mining, dyeing, tanning, metal plating, battery manufacture, petroleum refining, paint manufacture, printing etc., generate wastewater that is contaminated with heavy metals such as Cadmium, Zinc, Copper, Nickel, Lead, Mercury and Chromium (Kadirvelu *et al.,* 2001; Adelaja, *et al.,* 2011). These heavy metals are toxic, stable, non-biodegradable and when they contaminate water they become a serious threat to human health (Boudrahem, *et al.*, 2011). Their accumulation in tissues of living organisms can cause diseases and disorders which necessitates their removal from wastewater before discharge.

Cadmium is produced mainly from nickel-cadmium batteries, metal plating and plastic industries (Shotyk *et al.,* 2005). It is extremely toxic even at low concentrations (Wang *et al.,* 2011). The EU sets the maximum acceptable limit of cadmium in drinking water as 5 μg L^{-1} (Boudrahem *et al.*, 2011).

Various techniques such as adsorption, ion exchange, chemical precipitation, electro-dialysis, reverse osmosis, coagulation-flocculation, floatation and membrane processes have been used to treat industrial wastewaters (Mukesh and Lokendra Singh, 2013). Among these techniques, adsorption is superior in simplicity of design, highly efficient and is easier to operate which make it more favourable (Suteu and Bilba, 2005).

The need to control our environment from industrial waste water pollution has ignited interest in cost effective products to treat industrial effluents. Biosorbents prepared from agricultural wastes and by-products have been widely studied because they are inexpensive, abundant in nature and require little processing (Wan Ngah and Hanafiah, 2008). Biosorption using renewable agricultural wastes offers a promising potential alternative to conventional technologies for treatment of industrial effluents. Adsorption studies have been carried out using plant residues such as grape stalk wastes (Villaescusa *et al.,* 2004), moringa oleifera (Aminu *et al.,* 2014), coffee wastes (Kyzas, 2012), degreased coffee beans (Kaikake, 2007), maize leaf (Babarinde *et al.,* 2006), rice husk ash and neem bark (Bhattacharya *et al.,* 2006), etc. However, the use of these plant residues for adsorption has its own problems: it has low adsorption capacity and increases the Biological Oxygen Demand (BOD), Chemical Oxygen Demand (COD) and Total Oxygen Demand (TOD) of the water (Wan and Hanafiah, 2008).

One of the most effective and widely used adsorbents for treatment of water and wastewater is activated carbon, which is an amorphous form of carbon that is specially treated to produce a very large surface area. This structure provides

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activated carbon with the ability to adsorb gases and vapours from gases, and dissolved or dispersed substances from liquids (Othmer, 1979). It is the most widely used adsorbent since most of its chemical (e.g. surface groups) and physical properties (e.g. pore size distribution and surface area) can be tuned according to what is needed.

In developing countries, the use of commercial activated carbon is often too costly; therefore, emphasis is made on the preparation of the AC from locally available materials for use as adsorbents. A number of studies have been carried out on the preparation of AC from agricultural and industrial by-products for use as adsorbent (Ahmedna *et al.,* 2004; Aloko and Adebayo, 2007; Aygun *et al.,* 2003; Bansode *et al.,* 2002). There is a constant need to investigate the potentials of various materials as activated carbon precursors depending on their cost and availability (Misihairabgwi *et al.,* 2014).

In the present work, African Elemi (*Canarium schweinfurthii*) seeds were subjected to chemical activation method to produce activated carbon that was used for removal of Cd(II) ions from synthetic wastewater. The aim was to investigate the capacity of activated African Elemi seed for adsorbing Cd(II) ions. The effects of agitation time, adsorbent dosage, pH, temperature, initial ion concentration, and contact time on the adsorption capacity were studied. Kinetics and thermodynamics studies were carried out while adsorption isotherms were used to model the adsorption process.

2. Materials and Methods

2.1. Adsorbent

The African Elemi Seeds were collected randomly within Jos, Plateau State, Nigeria. A sample of the seeds was thoroughly washed with de-ionized water to remove dirt and other particulate matter. The fresh sample was dried in an oven for six hours, at temperature of 100°C and grounded using mortar and pestle. It was then soaked with n-hexane for three days to remove the fats in the seeds after which, it was thoroughly washed again with de-ionized water, then dried and weighed. Impregnation was done with 0.1M HNO_3 for about six hours and then filtered. The sample was carbonized in an electric muffle furnace for six hours at temperature of 300°C in an inert atmosphere and then cooled in the desiccators. The activated carbon was washed with de-ionized water, dried and weighed. The sample was again subjected to a further treatment by grinding and sieving with a 250μm sieve to get a very fine powder then stored in an air tight container.

2.2. Characterization of the Activated Carbon

The activated carbon yield, ash content, bulk density and pH were determined using the method of Ahmedna (1997). The major functional groups in the adsorbent were determined by Fourier transform infrared spectrophotometer (FTIR-8400S).

2.3. Batch Adsorption Experiments

Batch adsorption experiments were performed to determine the effects of temperature, initial metal ion concentration, contact time, pH and adsorbent dosage on the adsorption of Cd(II) ions from solution. For each experiment, 50 mL of synthetic wastewater containing a known concentration of Cd(II) ions was shaken with a calculated amount of the activated carbon in a flask at a known temperature and for a known duration. The pH of working solutions was adjusted to the desired values by the addition of either 0.1M HCl or 0.1M NaOH solution. In each case, after equilibrium, the mixture was filtered using Whatman No.1 filter paper and the concentration of Cd(II) ions remaining in the solution was determined using atomic absorption spectrophotometer (Perkin Elmer; Analyst 200).

The amount of metal ion adsorbed by the adsorbent and the percentage removal of the metal ion were calculated by using the following equation (Al-Anber and Al-Anber, 2008):

$$q_e = \frac{(\mathrm{Co}-\mathrm{Ce})\mathrm{W}}{\mathrm{M}} \text{ and } X\% = \frac{\mathrm{Co}-\mathrm{Ce}}{\mathrm{Co}} \times 100$$

Where q_e is the amount of Cd(II) ions adsorbed on the activated carbon per gram adsorbent (mg/g); C_o is the initial concentration of Cd(II) ions in solution (mg/L); C_e is the concentration of Cd(II) ion at equilibrium with the solid phase (mg/L); W is the volume of metal ion solution (L); M is the mass of the adsorbent (g) and X% is the percentage of metal ions removed.

3. Results and Discussion

3.1. Physicochemical Analysis of the Activated Carbon

The results of the physicochemical analysis of the activated carbon are presented in Table 1. All the values fall within the reported range in literatures (Long and Criscione, 2013; Bassey *et al.,* 2015).

Table 1. Physicochemical properties of the AC

Parameter	Values
Activated carbon yield (%)	55.00
Bulk density (g/m^3)	0.56
Ash content (%)	5.00
pH	6.80
Hardness (%)	5.22
Surface area (cm^2/g)	54.1

3.2. Functional Groups

The FTIR spectra are shown in Fig. 1 while a summary of the peaks and their possible assignments are given in Table 2. Result of peaks displayed by FTIR due to the presence of some functional groups on the surface of activated carbon powder is translated as follows:

Table 2. Functional groups present in the activated carbon

Band position	2925 and 2854cm^{-1}	1210cm^{-1}	1640cm^{-1}	1745cm^{-1}	1156cm^{-1}
Possible assignments	C-H	C-C	C=C	C=O	C-O

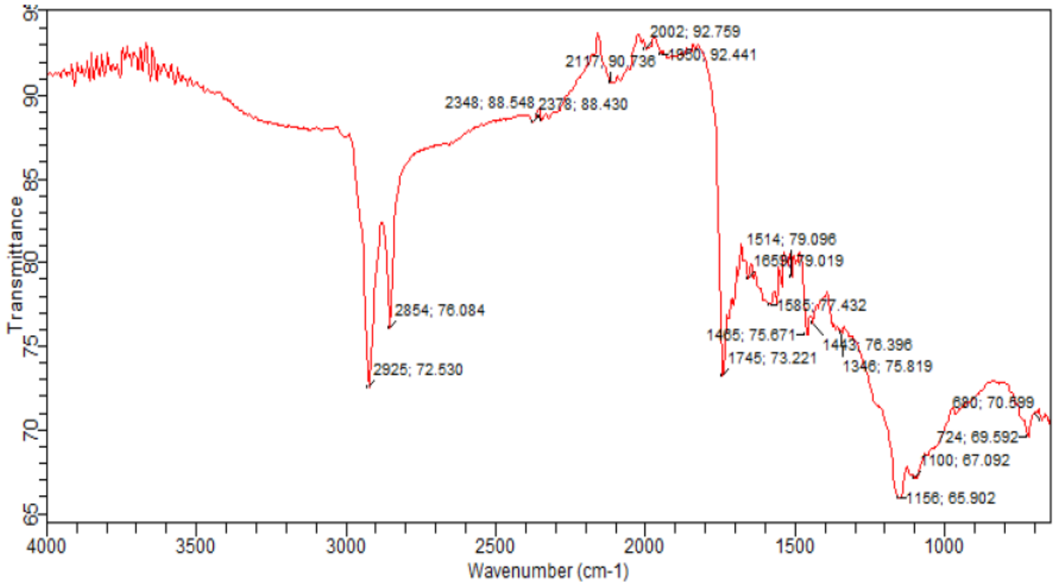

Figure 1. FTIR spectra of the activated carbon

3.3. Effect of pH

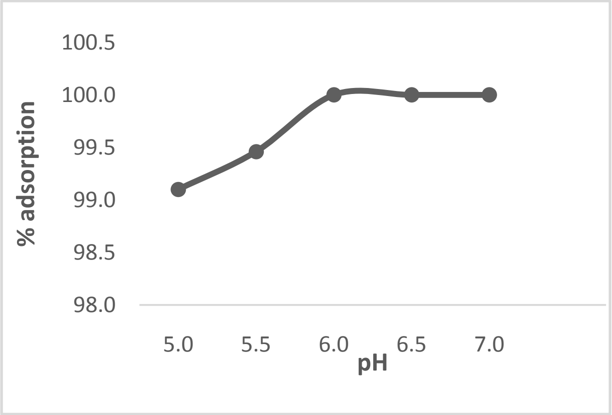

Figure 2. Effect of pH on the adsorption of Cd(II) ions from solution

Adsorption is strongly affected by pH since the properties of the adsorbent and the degree of ionization of the solution are influenced by pH. Adsorption of Cd(II) ions on the activated carbon was investigated within a pH range of 5.0 to 7.0 and the result shown in Fig. 2. It can be seen that adsorption of Cd(II) ions increased rapidly from pH 5.0 – 5.5 and reaches a maximum capacity (100%) at pH 6.0. This could be described in terms of the interaction between the charge on the adsorbent and the H^{+} ions present in the solution (Malkoc *et al,* 2005). At low pH values, the concentration of protons was high which competed with the positively charged Cd(II) ions for binding sites on the adsorbent surface. The concentration of protons decreased with increasing pH which resulted in more adsorption of the Cd(II) ions as shown in the figure. In addition, most of the functional groups on the adsorbent are protonated at low pH, this reduced the number of sites available for the adsorption of metal ions (Putra, *et al.,* 2014). Thus, increasing the pH in an acidic media increases the amount of metal adsorbed. This result agrees with that reported by Amarasinghe and Williams (2007); Putra *et al.,* (2014); Chen *et al.,* (2012).

3.4. Effect of Contact Time

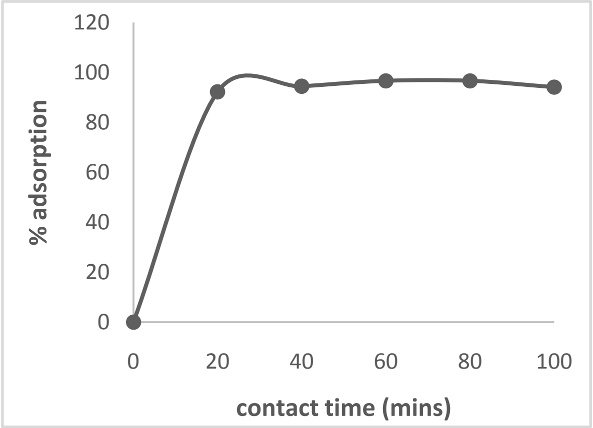

Figure 3. Effect of contact time on the adsorption of Cd(II) ions from solution

Contact time is an important factor in the adsorption process. It affects the treatment efficiency and also gives an insight into the kinetics of the adsorption. The rate of Cd(II) ion uptake is shown in Fig. 3. The rate of adsorption increased rapidly in the first 20 minutes, and then it slowed down and reached equilibrium at 60 minutes with a maximum adsorption capacity of 96.68%. The initial faster rate of removal of Cd(II) ion may be due to the availability of the uncovered surface area of the adsorbents, since, the adsorption kinetics depends upon the surface area of the adsorbent (Qadeer *et al.,* 2005). As the number of available sites decreased, the rate of adsorption also decreased as seen in the figure. Another reason for the slower rate could be as a result of repulsive forces between the adsorbed Cd(II) ions on the surface and those still remaining in the solution.

3.5. Effect of Initial Metal Ion Concentration

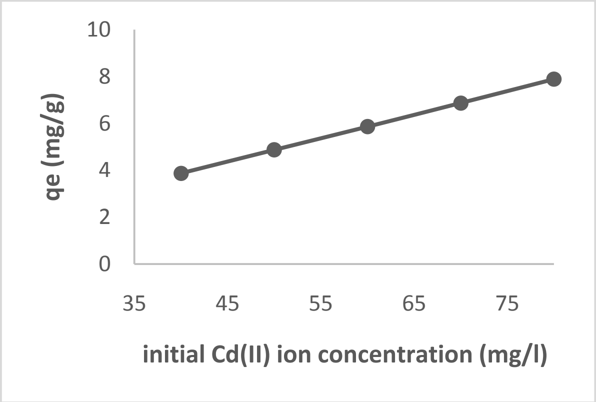

Figure 4. Effect of initial metal ion concentration on adsorption of Cd(II) ions from solution

In adsorption processes, the concentration gradient between the metal on the adsorbent and the metal ions in aqueous phase is the driving force. Fig. 4 shows the effect of initial metal ions concentration on the adsorption of Cd(II) ions onto the activated carbon. The amount of Cd(II) ions uptake increased as the initial metal ions concentration increased. This is because with increase in initial metal ion concentration, the mass transfer resistance of metal between aqueous and solid phases is reduced by the driving force of the concentration gradient (Akpomie and Dawodu, 2014). Another reason is that an increase in concentration of ions means more ions become available for adsorption.

3.6. Effect of Temperature

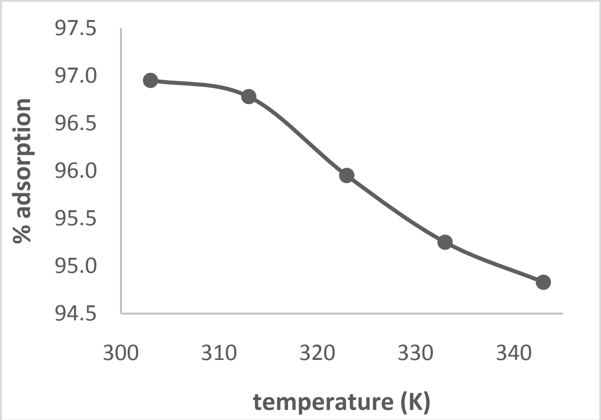

Figure 5. Effect of temperature on the adsorption of Cd(II) ions from solution

Researchers have carried out studies to investigate the effect of increasing temperature on the adsorption process and their results presented different behaviours such as increase in adsorption capacity (Malkoc and Nuhoglu, 2005), decrease in adsorption capacity (Akpomie *et al.,* 2013) and irregular behavior (Adekola *et al.,* 2014). In this study the effect of temperature on the adsorption process was investigated in the range of 303 – 343K and the results shown in Fig. 5. It was observed that there was gradual decrease in the percentage removal of Cd(II) ions as the temperature increases from 303-343K implying that the process is exothermic. The decrease in the amount of metal ions adsorbed as temperature increased might be attributed to the fact that high temperature causes rupture in the active binding sites of the adsorbent. This reduces the adsorptive forces between the metal ions and the binding sites resulting in a lower adsorption capacity.

3.7. Effect of Adsorbent Dosage

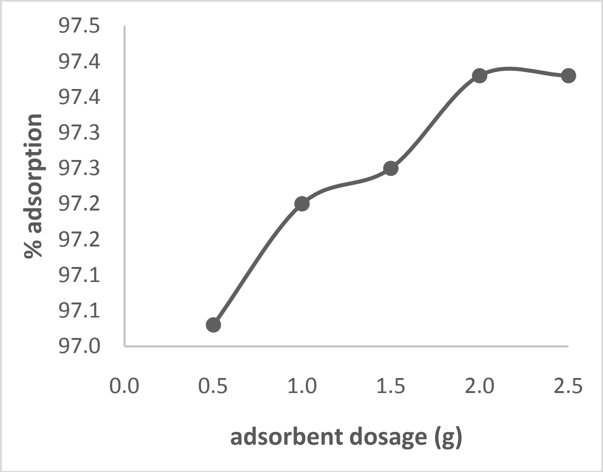

Figure 6. Effect of adsorbent dosage on the adsorption of Cd(II) ions from solution

As shown in Fig. 6, an increase in adsorbent dosage led to increase in removal of Cd(II) ion, this is not surprising as increasing adsorbent dosage enhances availability of adsorption sites and creates more surface area. With an adsorbent dosage of 2g, 100% removal of metal ion was obtained. The results obtained corroborated the findings of Bassey *et al.,* (2015), that as the concentration of the adsorbate increases; the metal ions removed also increase.

3.8. Adsorption Kinetics

Kinetics studies are important in the design and operation of adsorption systems (Akpomie *et al.,* 2013). In analyzing the controlling mechanism for the adsorption of Cd(II) ions onto the activated carbon, pseudo-first order and pseudo-second order kinetic models were used. The kinetic parameters for pseudo-first order and pseudo-second order models are shown in Tables 3 and 4 respectively. Low correlation coefficient of the pseudo-first order model shows that the reaction is not elementary and the adsorption is not exclusively for one site per ion. The adsorption of Cd(II) ions onto the activated carbon can best be described by the pseudo-second order model due to larger correlation coefficient. This shows that the adsorption of Cd(II) ions by the activated carbon follows the second order mechanism where rate of occupation of adsorption sites is proportional to the square of the number of unoccupied sites (Adekola *et al.,* 2014). This implies that the rate limiting step could be chemisorption promoted by either valency forces or covalent forces (Boudrahem *et al.,* 2011).

Table 3. Pseudo-first order constants for the adsorption of Cd(II) ions by the AC

$k_1(min^{-1})$	$q_e(mg/g)$	R^2
0.00015	2.1677	0.452

Table 4. Pseudo-second order constants for the adsorption of Cd(II) ions by the AC

$k_1(g/mgmin)$	$q_e(mg/g)$	R^2
3.163	3.984	0.989

3.9. Adsorption Isotherms

Adsorption isotherms show the relationship between the amount of adsorbate on the adsorbent and its equilibrium concentration in solution (Akpomie *et al.,* 2013). Important information on the mechanism of the adsorption and the surface properties of the adsorbents are provided by the adsorption isotherms. The adsorption experiments in this work were described using the Langmuir and Freundlich isotherm models.

The Langmuir isotherm is based on the assumption that each adsorption site can accumulate only one molecule with no interactions between the sites, the adsorption surface is homogeneous and only a monolayer is formed on the adsorbent. It is expressed mathematically as:

$$^{C_e}/_{q_e} = {}^{1}/_{Q_o K} + {}^{C_e}/_{Q_o}$$

where Ce is the equilibrium concentration (mg L^{-1}), qe the amount adsorbed at equilibrium (mg g^{-1}), Q_0 is the monolayer adsorption capacity of the adsorbent and K is the Langmuir constant related to adsorption capacity and energy of adsorption. The linear plot of Ce/qe versus Ce gives a straight line and Qo and K are determined from the slope and intercept of the plot.

The Freundlich isotherm model is based on adsorption on heterogeneous surface with independent sites where there is an exponential decrease in the sorption site energy distribution (Boudrahem *et al.,* 2011). The linearized form of the equation is given as:

$$\text{Log } q_e = \log K_F + \frac{1}{n} \log C_e$$

where K_F $(mg/g)(mg/L)^{1/n}$ and n are the Freundlich constants related to adsorption capacity and adsorption intensity, respectively. A plot of log q_e against log C_e gives a straight line with an intercept and slope K_F and 1/n respectively.

Table 5 presents the isotherm parameters for both Freundlich and Langmuir models. It was observed that the Freundlich model describes the experimental data better than the Langmuir model because of high correlation coefficient. This shows that the surface of the activated carbon is heterogeneous with independent binding sites. More realistic description for adsorption on organic matter is given by the Freundlich model because it accounts for different binding sites (Soltani *et al.,* 2009).

Table 5. Equilibrium isotherm parameters

Freundlich model			
K_F (mg/g)	**n**	**1/n(L/mg)**	**R^2**
0.989	0.596	1.678	0.963
Langmuir model			
$1/Q_o$	Q_o	K_L	R^2
0.798	1.252	0.968	0.914

3.10. Thermodynamic Studies

Thermodynamic studies show the feasibility and spontaneity of the adsorption process and the structural changes due to metal ion binding. Thermodynamic parameters were investigated for the adsorption of Cd(II) ions onto the activated carbon within the range 303 – 343K while keeping other parameters constant.

The free energy change is given by the following expressions (Liu and Liu, 2008)

$$\Delta G^o = - RT \ln K$$

in which T is absolute temperature and R is the gas constant.

$$\ln K = - (\Delta H^o/RT) + (\Delta S^o/R)$$

The values of standard enthalpy change (ΔH^o) and standard entropy change (ΔS^o) were obtained from the slope and intercept respectively, of the graph of ln K against 1/T (K^{-1}). The thermodynamic parameters are shown in Table 6. The value of the correlation coefficient (0.968) shows good agreement between the free energy change and the temperature.

For the range of temperature investigated, negative values of free energy (ΔG^o) were observed which shows that the adsorption process is thermodynamically feasible and spontaneous. It was also observed that standard enthalpy change (ΔH^o) was negative while standard entropy change (ΔS^o) was positive. Negative value of ΔH^o confirmed exothermic nature of the process while positive value of standard entropy change (ΔS^o) was an indication of an increase in the degree of the randomness at the adsorbent-adsorbate interface during the adsorption (Liu and Liu, 2008). Similar results were found by Liu and Xu (2007).

Table 6. Thermodynamic parameters for Cd(II) ions adsorption from solution

ΔG^o(J/mol)	**ΔH^o(J/mol)**	**ΔS^o(J/mol/K)**	**R^2**
-122.909	-1503	3.775	0.968

4. Conclusions

African elemi seeds have been successfully converted to activated carbon and used as a low cost adsorbent for Cd(II) ions removal from wastewater. Based on the experimental results obtained, the following conclusions were drawn:

- ❖ FTIR spectra showed the presence of some functional groups responsible for binding of the metal onto the activated carbon.
- ❖ The contact time, pH, initial metal ions concentration, adsorbent dosage and temperature had a remarkable effect on the metal adsorption.
- ❖ The adsorption process of the metal ions was best described by a pseudo-second order.
- ❖ The Freundlich isotherm model fitted well the experimental data indicating a heterogeneous surface with independent binding sites.
- ❖ The thermodynamics parameters showed that the adsorption of Cd(II) ions by the activated carbon was spontaneous and exothermic, with an increase in the randomness at the solid/solution interface during the adsorption process.
- ❖ Activated carbon from African elemi seeds could be used as a low cost adsorbent for removal of cadmium (II) ions from wastewater.

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