

Adsorptive Evaluation of Activated Carbonized Avocado Pear Seed (*Persea Americana*) and Activated Carbonized orange peels (*Citrus Sinensis*) in Taking out Ni^{2+} ion in Contaminated Water

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Abstract

Grave and complicated pollution problems are posed by the existence of heavy metals trendy environment and this has been a focus of study all over the world. The Activated carbonized avocado pear seed (ACAPS) and activated carbonized orange peel (ACOP) were each soaked with potassium hydroxide (KOH) solution at a 1:1 ratio, for 48 hours, the outcome was washed, air-dried and separately oven dried at 105°C for 6 hours and further heated in a muffle furnace (Carbolite AAF1100) at 250°C for 1 hour. The ACAPS and ACOP obtained were physicochemical characterized with scanning electron microscope (SEM) and Fourier-transform infrared (FT-IR) spectrophotometer. Ni^{2+} ions adsorption route on ACAPS as well as ACOP was considered by means of batch adsorption isotherm investigation. Adsorption forms was assessed by Freundlich, Langmuir, DRK, Tempkin as well as Flory-Huggin isotherm models. SEM picture, show that ACAPS possessed an enormous amount of different dimension pores and the ACOP had smaller amount of pores of smaller sizes. The R^2 obtained varies from 0.92 to 1.00 and it points to the fact that all the isotherm simulation was competent to clarify the correlation in the data we got. ACAPS had more adsorption site, however, ACOP took up more Ni^{2+} ion per adsorption site on comparing Flory-Huggins isotherm model n_{FH} values vis-a-vis Langmuir isotherm model q_m values. In conclusion, ACAPS was a superior adsorbent when matched with ACOP, it was more than twice as effective for taking out Ni^{2+} ions from contaminated water owing to a blend of its enormous amount of different size openings and the functional groups on its surface, with a q_m value of 18.18 and 8.70 mg/g in that order.

Keywords: Adsorptive, Avocado, Orange, Physiosorption, chemisorption, Isotherm.

INTRODUCTION

Water polluted with metallic discharge can result to several severe health issues. Serious and complicated pollution problems are posed by heavy metals presence in the environment; this has remained the consideration nerve centre all over the world. The danger of heavy metals has resulted in the need to pressingly and efficiently remove them from contaminated waters. Nickel ions contamination arises from natural and anthropogenic activities and it causes

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serious health problems such as fibrosis of pulmonary, lungs cancer, bone etc. (Lakherwal, 2014; Singh and Gupta, 2016; Abdel-Raouf and Abdul-Raheim, 2017).

Several methods aimed at removing heavy metal ion from aqueous solutions include coagulation, flocculation, ion exchange, sedimentation, solvent extraction, adsorption, (Hossain *et al.*, 2012; Kumar *et al.*, 2014). Latest technologies for removing heavy metals are established on ultra-filtration, nano-filtration, membrane filtration as well as reverse osmosis, these technique are linked with high overbearing as well as running costs (Miretzky *et al.*, 2006; Igwe and Abia, 2007; Lasheen *et al.*, 2017; Vezentsev *et al.*, 2019). The challenges linked with mass-market available activated carbon have energized the search for cheaper options. Now, there is need to explore unconventional and cheap agricultural wastes in generating activated carbon.

Adsorption procedure is deemed superior likened to other techniques because of its comfort, manageable operation as well as ease of design (Amit and Monocha, 2006). This work is designed to evaluate the adsorption effectiveness of activated carbonized avocado pear seed (ACAPS) (*Persea americana*) as well as activated carbonized orange peels (ACOP) (*Citrus sinensis*) in taking out ions of Ni²⁺ in aqueous solution.

Materials and Methods

Collection of Samples

The orange peels and avocado pear seeds were obtained from the oranges and avocado pears acquired from New-Benin Market in Benin City, Edo State, Nigeria. Orange peels as well as avocado pear seeds were thoroughly washed with distilled water to get rid of filths and dried them in an oven at 75°C for 7 days. They were filled in a properly sealed polyethylene sacks. *Activated Carbonized Avocado Pear Seed (ACAPS) and Activated Carbonized Orange Peel (ACOP) Production.*

Avocado pear seeds as well as orange peels were separately dried, cut into smaller sizes, crushed into fine particles form and sieved (using a BSS-53µm mesh sieve). The sieved avocado pear seeds and orange peels were individually carbonized in a muffle furnace (Carbolite AAF1100) at 300°C for 40 minutes. Carbonized avocado pear seeds as well as orange peels were impregnated with Potassium hydroxide (KOH) solution (in order to increase the surface area as well as activated carbon porosity) at a ratio of 1:1 for 48 hours until the mixture turned into slurry in different containers. The activated carbonized avocado pear seed (ACAPS) and orange peels (ACOP) obtained were washed separately with distilled water pending when the pH of their filtrate was 7. The ACAPS and ACOP was oven dried at 105°C for 6 hours and reheated in a muffle furnace at 250°C for one hour.

Adsorbates Preparation and Characterization

Nickel (II) nitrate salt was used in the preparation of nickel contaminated water and its concentration was established using atomic absorption spectrophotometer (AAS).

ACAPS and ACOP Physicochemical Characterization

The ACAPS and ACOP were physicochemically characterized utilizing scanning electron microscope (phenom pro suite desktop-SEM) to observe their morphological images and their functional groups were investigated using Fourier-transform infrared (FTIR) spectrophotometer (Buck scientific model VGP-210).

Adsorption Isotherm Studies

Ni²⁺ ions adsorption procedure on ACAPS and ACOP was evaluated using the batch adsorption isotherm investigation. The batch adsorption investigation were conducted by varying the ACAPS and ACOP dose beginning with 0.2g to 2.5g, interaction period beginning with 20minutes to 100minutes and pH range from 3 to 7. The concentrations of Ni²⁺ ion in the contaminated water was mixt beginning with 20.0 mg/l to 100.0 mg/l, at observed optimal ACAPS and ACOP dosages, interaction period, pH and 300 rpm agitation speed. The Ni²⁺ ions equilibrium concentration in the contaminated water after treatment was adjudged with AAS (Buck scientific model VGP-210).

Adsorption Mechanisms and Isotherms

Classical adsorption mechanisms involve the dispersion of adsorbate on adsorbent surface via intermolecular forces amongst them; adsorbate movement into openings of adsorbent and monolayer adsorbate accumulation on adsorbent (Iakovleva and Sillanpää, 2013). However, ion exchange may well be the main mechanism for exclusion of heavy metal ions (M²⁺), owing to availability of assorted functional group atop the diverse adsorbents surfaces. The functional groups serve as the active ion exchange sites for arresting ions of heavy metal (He *et al.*, 2019).

Adsorption isotherms interpret the adsorbent mechanism concept taking place at the surface, adsorbate one-tracklayer or multilayer type and adsorbent capability (Iakovleva and Sillanpää, 2013). Adsorbate adsorbed equilibrium capacity (or adsorption capabilities) as well as adsorption proficiency in contaminated water was computed by means of equations 1 and 2 in that order:

$$q_e = \frac{(C_i - C_e)v}{M} \tag{1}$$

$$\%E = \frac{C_i - C_e}{C_i} \times 100 \tag{2}$$

The parameters C_i is the starting amount of adsorbate (mg/L); q_e and C_e are the metal ions amount adsorbed (mg/g) as well as amount (mg/L) at equilibrium in that order; M, represents adsorbent mass (g) and V, represents contaminated water volume in litre.

Freundlich, Langmuir, Dubinin–Radushkevich, Tempkin and Flory-Huggin isotherm models will be applied to assess the data realized in this investigation study. Isotherm model of Langmuir is affirmed based on one-tracklayer adsorption which takes place evenly atop the adsorbent’s energetic locations and there is no interplay in the middle of the adsorbates. It is mainly regularly applied to determine the adsorbate quantity atop an adsorbent as a function of concentration in a specified temperature (Almalike, 2017; He *et al.*, 2019). Langmuir isotherm equation is shown in equation 3:

$$\frac{1}{q_e} = \frac{1}{q_m} + \frac{1}{K_L q_m C_e} \tag{Langmuir isotherm} \tag{3}$$

(Ghasemi *et al.*, 2018, Lim *et al.*, 2019)

q_m is the theoretical utmost one-tracklayer sorption capability (mg/g), and K_L (L/mg) signifies Langmuir isotherm constant for adsorption energy.

Isotherm model of Freundlich is suitable for non-ideal adsorption plus a multilayer adsorption mechanism on heterogeneous surfaces by means of an uneven adsorption heat distribution on top of surfaces (Iakovleva and Sillanpää, 2013; Lasheen *et al.*, 2017). The Freundlich isotherm equation is shown in equation 4:

$$\log q_e = \log k_f + (1/n_f) \log C_e \tag{4}$$

(Bankole *et al.*, 2019, Lim *et al.*, 2019)

The K_F and n_F are the Freundlich isotherm constants depicting adsorption capability as well as intensity, in that order. The value of $1/n$ indicates whether the adsorption process is spontaneous and it is a dimensionless heterogeneity parameter. Spontaneity or a favourable normal adsorption conditions apply when n value is more than 1 and the lesser the worth of $1/n$, the more the heterogeneousness (Saini *et al.*, 2021; Yildiz, 2017).

Dubinin–Radushkevich (DRK) isotherm model is not established on ideal notions for instance, steric interruption absence in the middle of sorbed and arriving particles, uniform potential of the adsorption locations as well as surface homogeneousness atop microscopic height (Sarı and Tuzen, 2009; Amin *et al.*, 2015; Moftakhar *et al.*, 2016). It also interprets adsorption’s physical as well as chemical qualities (Essomba *et al.*, 2014). DRK isotherm based on the theory of capacity stuffing of micro pores and a semi-empirical equation (TVFM) (Worch, 2012), its adsorption route keep an eye on a pore stuffing means (Sarı and Tuzen, 2009), as a result, offering a perceptive into the adsorbent porousness as well as seeming adsorption free energy which make available facts on character of sorption route or means, specifically, whether chemical or physical sorption is taking place on uniform and non-uniform surfaces put together (Inyinbor *et al.*, 2015; Onwu and Ngele, 2015). DRK equation (equation 5) remain established to describe a solid’s energetic heterogeneity at low coverage as one-tracklayer expands in micropores and the adsorption isotherms of sole solute systems (Essomba *et al.*, 2014; Hossain *et al.*, 2016; Inyinbor *et al.*, 2016).

$$\ln q_e = \ln q_D - \beta \cdot \varepsilon^2 \quad 5$$

(Adekola *et al.*, 2016, Ghasemi *et al.*, 2018)

$$[\varepsilon = RT \ln(1 + 1/C_e)] \quad 5a$$

$$E = 1/(-2\beta)^{1/2} \quad 5b$$

q_D (mol/g) signifies all-out adsorption capability, β , the DRK isotherm constant (mol²/kJ²) and the adsorbate’s free energy for each mole as it move to surface of adsorbent from inestimable distance. E is adsorption mean free energy (kJ/mol), if E is less than 8, the adsorption process is influenced by physisorption mechanism; if E is greater than or equal to 8 or E is less than or equal to 16, the adsorption mechanism is influenced via means of chemisorption (ion-exchange mechanism) route as well as if E greater than 16, the adsorption means is influenced by means of particle diffusion. R , is gas constant (Jmol⁻¹ K⁻¹), T , is absolute temperature (Kelvin) and ε is Polanyi (adsorption) potential energy.

The Temkin’s model was advanced on chemisorption notion and presumes that adsorbate molecule’s adsorption heat reduces linearly with adsorbent layer coverage owing to adsorbent-adsorbate interplay; likewise, it adopts a binding energy uniform allocation (Tie *et al.*, 2017). The Tempkin isotherm equation is shown in equation 6:

$$q_e = B \ln K_T + B \ln C_e \quad 6$$

(Tie *et al.*, 2017)

$$B = (RT/b) \quad 6a$$

B is heat of adsoption ($B = RT/b_T$); b_T (J/mol), is constant of Temkin isotherm; K_T (L/g), is equilibrium binding constant as well as associated to the all-out binding energy (Tie *et al.*, 2017, Ghasemi *et al.*, 2018, Bankole *et al.*, 2019). When heat of adsorption is less than 40kJ/mol, it indicates physical adsorption or physisorption (Saini *et al.*, 2021).

The Flory-Huggins isotherm is valuable in explaining surface coverage degree appearances of adsorbate on adsorbent; this isotherm model can express the practicability and spontaneity of an adsorption technique (Almalike, 2017, Ayawei *et al.*, 2017). The Flory-Huggins isotherm equation is shown in equation 7:

$$\text{Log} (\theta / C_i) = \text{Log} K_{FH} + n_{FH} \text{Log} (1-\theta) \text{ (Flory-Huggins isotherm)} \quad 7$$

(Almalike, 2017, Ayawei *et al.*, 2017)

$$[\theta = (1 - [C_e / C_i])] \quad 7a$$

$$\Delta G^\circ = -RT \ln k_{FH} \quad 7b$$

θ is surface coverage of adsorbent by the adsorbate, C_i is initial concentrations, K_{FH} and n_{FH} are Flory-Huggins model equilibrium constant and n_{FH} is number of adsorbate occupying adsorption site respectively. ΔG° is standard free energy change. If ΔG° calculated is negative, it implies adsorption technique was spontaneous in nature and supports an exothermic nature and vice versa if positive (Almalike, 2017).

The isotherm factors are obtained with the aid of slope and intercept of the linear plot. Isotherm models fitting were evaluated using R^2 values.

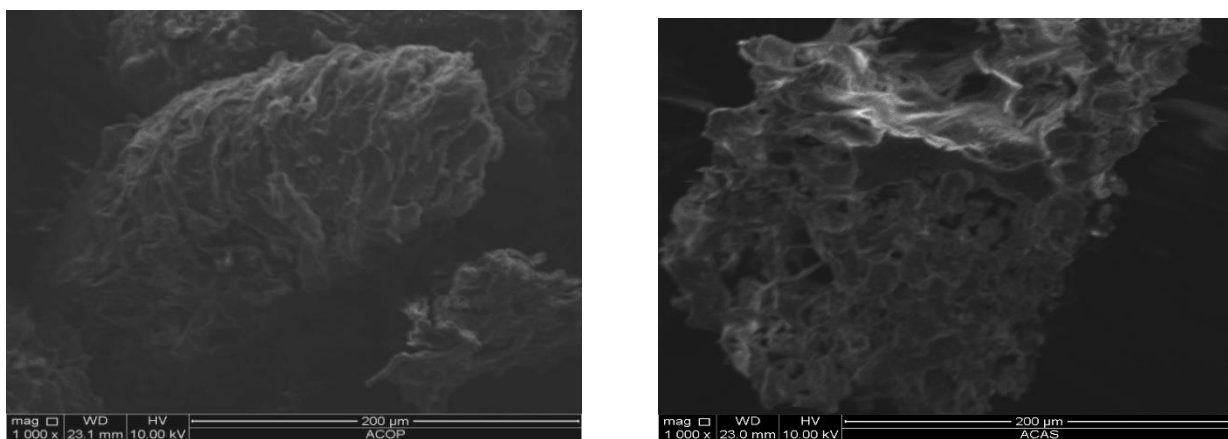
Results and Discussion

Activated carbonized avocado pear seed (ACAPS) and activated carbonized orange peel (ACOP) characterisation

The ACAPS and ACOP was physicochemically characterized with the aid of scanning electron microscopy (SEM) as well as Fourier transformed infrared spectroscopy (FTIR). The ensuing results were achieved:

Analysis of scanning electron microscopy (SEM)

Scanning electron microscope imageries obtained is displayed in Fig. 1; it revealed the superficial framework of ACAPS as well as ACOP.



(a) (b)
Figure 1: ACOP (a) and ACAPS (b) SEM pictures

The SEM photograph, revealed that activated carbonized avocado pear seed possesses higher pores of various sizes with fibrous structure while the activated carbonized orange peel had smaller number of smaller size pores. The SEM images of both adsorbent revealed that the particles comprise of openings with uneven contours. The variance in pore proportions

illustrates the variances in physical characteristics in the middle of the activated carbonized orange peels and activated carbonized avocado pear seeds.

Fourier transformed-infrared spectroscopic (FT-IR) study

The spectrum of FT-IR study in fig. 2 presented functional groups existing atop ACAPS and ACOP.

The FT-IR spectrum for ACAPS revealed very strong band characteristics at 3393.15cm⁻¹ which signifies medium appearance of NH-stretching vibrations of primary-RNH₂ as well as secondary- R₂NH amine groups. Likewise, a peak at 2923.25cm⁻¹ agreed in the direction of OH-stretch of carboxylic acid (-COOH) group.

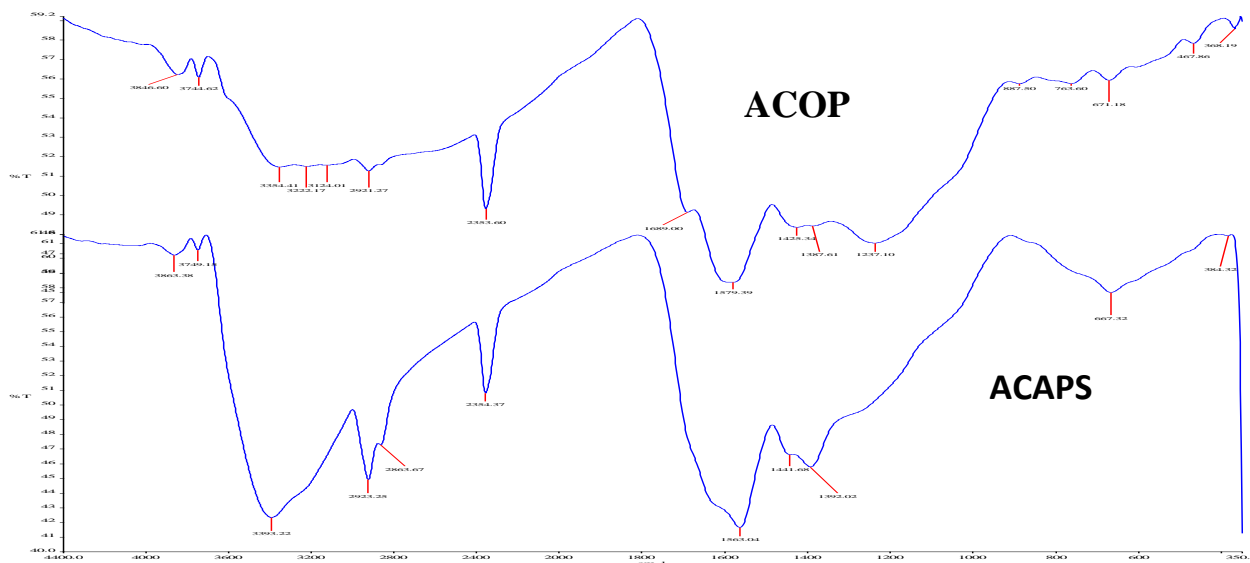


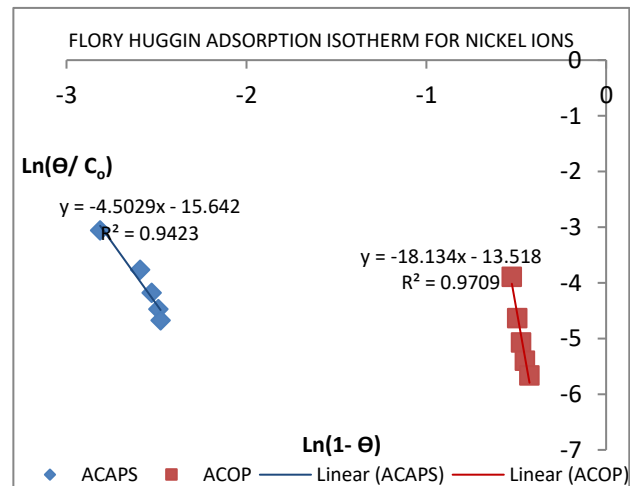
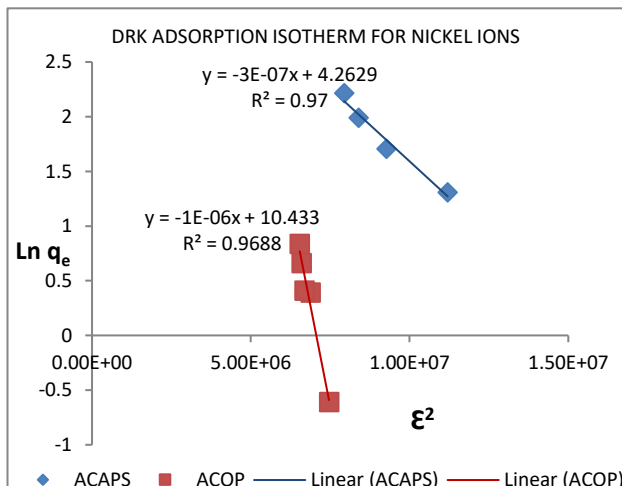
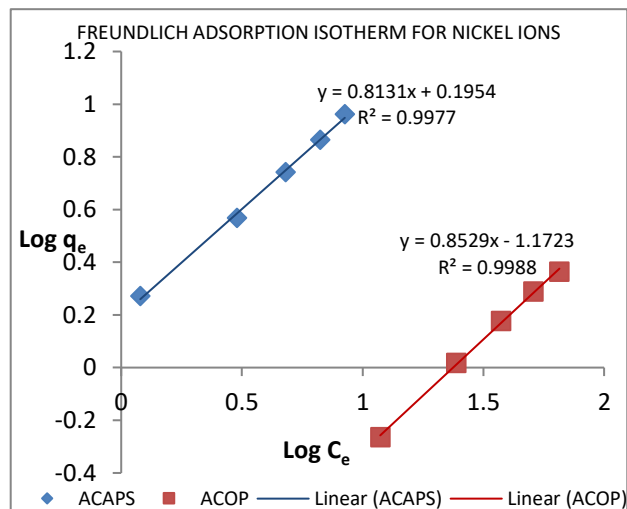
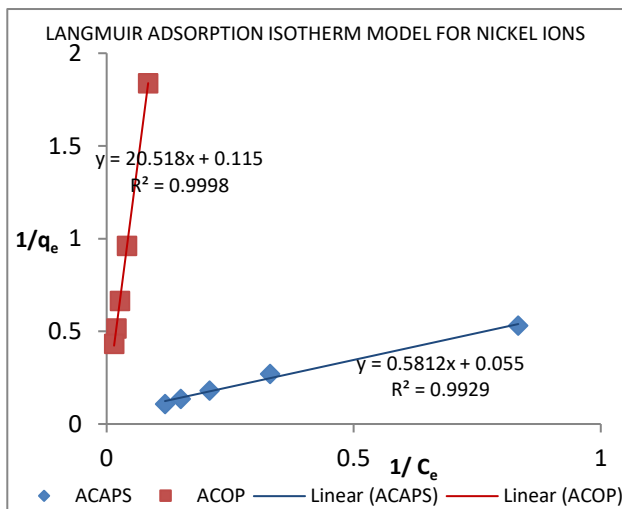
Figure 2: FT-IR Spectrum for ACOP and ACAPS

The peak at 1580.61 agreed in the direction of a medium appearance of strong -C-H stretch of alkyl group of CH₃ an electron donating moiety (with positive inductive effect) that can boost the adsorption of Ni²⁺. Also, a peak at 1441.68cm⁻¹ agreed in the direction of a strong C-H bend allotted to alkyl group. ACOP spectrum showed characteristic peak at 3354.41 and 3124.01 agreeing in the direction of medium N-H stretch of primary as well as secondary amine groups as well as O-H stretch (H-bonded) of carboxylic acid (-COOH) in that order. The peak at 2921.27 and 1425.34cm⁻³ corresponded to a medium appearance of strong -C-H stretch and -C-H bend respectively of alkyl group of CH₃ an electron donating moiety (with positive inductive effect) that can boost the adsorption of Ni²⁺. The peak at 1689.00 gave a very strong C=O stretch for amides (RCONH₂). A peak at 1237.10cm⁻³ revealed a strong -C-O stretch agreeing in the direction of acid as well as anhydrides and esters (-COOH, R(COOH)₂ and RCOOR). Both adsorbent holds a variety of functional groups on their surfaces, which could allow for ion exchange mechanism for the elimination of nickel ions (Ni²⁺).

Adsorption Isotherm Models and Parameters

The R² value ranging from 0.92 to 1.00 (Figure 3a-e and Table 1), indicated the whole isotherm models are capable of expressing the correlation in the obtained data. The Langmuir and Freundlich isotherm constants showed that ACAPS had a stronger adsorption energy, intensity and capacity for Ni²⁺ ions than ACOP based on their K_L, k_F and n_F respectively (Table 1), an indication that ACAPS would have a stronger hold and adsorb Ni²⁺ ions more than

ACOP. These observations are attributable to the various functional groups on adsorption locations of the adsorbents (Fig. 2). ACAPS adsorb more than twice of Ni²⁺ ions likened to ACOP based on the worth of their all-out adsorption capacity (q_m) from Langmuir isotherm model (Table 1), this observation is attributed in the direction of blend of functional groups as well as larger number of pores with variety of size, ACAPS possessed (Fig. 1). The Freundlich isotherm model revealed that ACOP and ACAPS adsorbed the Ni²⁺ ions spontaneously judging from their n_F value which was greater than one. The adsorption process was spontaneous as well as exothermic as confirmed by Flory-Huggins isotherm model with negative ΔG° values for ACOP and ACAPS (Table 1). The mean free energy of adsorption (E , kJ/mol) from Dubinin-Radushkeviek isotherm model revealed that adsorption mechanism was influenced by physisorption process ($E < 8$ kJ/mol, Table 1), this was corroborated by Tempkin model ($B < 40$ kJ/mol, Table 1). The energy per mole (β) required by Ni²⁺ ions to migrate to ACOP surface is higher than for ACAPS surface (Table 1), this is in agreement with the adsorption intensity n_F of Ni²⁺ ions onto ACOP. Tempkin adsorption isotherm showed that ACAPS had a greater adsorption heat (B) and all-out binding energy (k_T) (Table 1) confirming that ACAPS had firmer hold on the Ni²⁺ ion. The n_{FH} values for ACAPS and ACOP of 4.59 and 18.13 revealed that the ACAP had more adsorption site, however, ACOP took up more Ni²⁺ ion per adsorption site on Comparing Flory-huggins isotherm model n_{FH} values vis-a-vis Langmuir isotherm model q_m values.



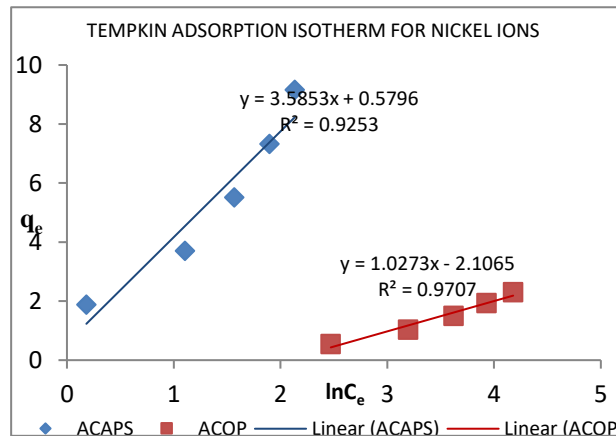


Figure 3: Isothermal models graphical representation for nickel ions adsorption

Table 1: Parameter values of adsorption isotherm model

Models of adsorption isotherm	Parameters	Units	ACOP	ACAPS
Langmuir $1/q_e = 1/q_m + 1/K_L q_m C_e$	R ²	-	1.00	0.99
	k _L	l/mg	0.0056	0.095
	q _m	mg/g	8.70	18.18
Freundlich $\log q_e = \log k_f + (1/n_f) \log C_e$	R ²	-	1.00	1.00
	K _F	-	0.067	1.57
	n _F	-	1.17	1.23
Dubinin-Radushkevick (DRK) $\ln q_e = \ln q_m - \beta \cdot \varepsilon^2$ [$\varepsilon = RT \ln(1 + 1/C_e)$]	R ²	-	0.97	0.97
	q _D	mg/g	71.02	33962.08
	B	J/molecule	1 × 10 ⁻⁶	3 × 10 ⁻⁷
	E	kJ/mol	0.71	1.29
Tempkin $q_e = B \ln K_T + B \ln C_e$ B = (RT/b _T)	R ²	-	0.97	0.92
	K _T	L g ⁻¹	0.13	1.18
	B	J/mol	1.03	3.58
	b _T	J/mol	2452.26	702.65
Flory-Huggin $\text{Log}(\theta/C_0) = \text{Log} K_{FH} + n_{FH} \text{Log}(1-\theta)$ [$\theta = (1 - [C_e/C_0])$] $\Delta G^\circ = -RT \ln k_{FH}$	R ²	-	0.97	0.94
	n _{FH}	-	18.13	4.50
	K _{FH}	-	1.35 × 10 ⁻⁶	1.61 × 10 ⁻⁷
	ΔG ^o	kJ/mol	-78.41	-90.73

CONCLUSION

The adsorption of Ni²⁺ ions onto ACOP and ACAPS occurred through a physisorption mechanism, it was spontaneous and exothermic in nature. Ni²⁺ ions were more firmly held by ACAPS likened to ACOP owing to functional group location on its surface. The ACAPS had more adsorption site, however, ACOP took up more Ni²⁺ ion per adsorption site on Comparing Flory-Huggins isotherm model n_{FH} values vis-a-vis Langmuir isotherm model q_m values. Generally, ACAPS was a superior adsorbent for removing Ni²⁺ ions, it was more than twice effective for elimination of Ni²⁺ ions from aqueous solution compared with ACOP owing to a blend of its significant amount of various size pores as well as surface functional groups.

Conflict of Interest

The authors declare that they do not have any conflict of interest.

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