



Divalent Nickel (Ni^{2+}) Removal Using Thermally Derived and Regenerated Activated *Cocos nucifera* Shells

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ABSTRACT

Thermo-chemically cracked *cocos nucifera* shells were converted into useful adsorbent using chemical activation method. Regeneration of the spent carbon was carried out via thermal regeneration method. The re-derived filter was used in the adsorption of nickel (Ni) from a simulated Ni^{2+} solution. Qualitative characterization was carried out using the Fourier Transformed Infra Red Spectrophotometer while Atomic Adsorption Spectrophotometer was used to analyze the equilibrium phase Ni^{2+} concentration. Comparative study of the derived and regenerated activated carbons was carried out using kinetic adsorption studies approach. From the experimental runs, it was evident that derived *cocos nucifera* shells activated Carbon provided slightly higher removal efficiency values than the vice for Nickel uptake. Investigations with applicability test supported the pseudo second order kinetics as best model in explaining the sorption rate ($k_2 = 0.744$) as against the proposed linear and first order kinetics. Generally, reuse of spent coconut shell carbon and its investigated film diffusion transport mode in removal of Ni^{2+} is critical a study with a worthwhile adoption in remediation study.

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Introduction

Pollutants refer to substances which increases in quantity due to human activity and adversely affects the environment. Heavy metals such as lead, mercury, cadmium, aluminum, arsenic etc. are the most dangerous among the inorganic pollutants. Heavy metals are elements having atomic weights between 63.546 and 200.590 and a specific gravity greater than 4.0 i.e. at least 5 times that of water ^[1]

Research on regeneration of spent activated carbon was carried out by Rahat and Hao ^[2]. This research work made use of the effect of the different temperature and concentration of HCl and H_3PO_4 on the regeneration of spent activated carbon for iodine and methylene blue solutions.

In another research, Tan *et al.* ^[3] worked on the optimization study for removal of 2,4,6-trichlorophenol using response surface methodology. In this research, coconut husk was used to prepared activated carbon using physicochemical activation method via potassium hydroxide (KOH) treatment and carbon dioxide (CO_2) gasification.

In a similar research conducted by Itodo *et al.* ^[4], batch kinetic studies was carried out on the bioremediation of textile effluent. Their result showed that metal ions (Cd^{2+} , Pd^{2+}) and dyestuff were successfully adsorbed from textile wastewater to an extent by bio sorbents prepared from chemically modified groundnut shell.

Adsorption is also the adhesion of atoms, ions, bimolecular or molecules of gas, liquid, or dissolved solids to a surface. It is a phenomenon whereby molecules adhere to a surface with which they come in contact. This process creates a film of adsorbates (molecules or atom being accumulated) on the surface of the adsorbent. It differs from absorption, in which a fluid permeates or is dissolved by a liquid or solid. The term

sorption encompasses both processes, while desorption is the reverse of adsorption. It is a surface phenomenon. Adsorption is operative in most natural physical, biological, and chemical systems, and is widely used in industrial applications such as activated charcoal, synthetic resins and water purification ^[5]

Activated carbon is an amorphous form of carbon with large internal surfaces and pores. Activated carbons are widely used because of their high adsorption abilities for a large number of organic compounds. However, the price of activated carbons is relatively high, which limits their usage. Thus, many researchers researched for cheaper substitutes, which are relatively inexpensive, and at the same time endowed with reasonable adsorptive capacity. These studies include the use of coal, fly ash, activated clay, palm-fruit bunch and others ^[6]. The most important characteristic of activated carbon is its adsorption capacity or uptake which is highly influenced by the activated carbon preparation conditions.

Coconut shell is the cover protecting the inner content which is thrown away or used as domestic fuel. To make a better use of this cheap and abundant agricultural waste, it was proposed to convert coconut shell into activated carbon. Conversion of coconut shell into activated carbon will serve a double purpose, firstly, waste is converted to useful, value-added adsorbents and also the use of agricultural by-products represents a potential source of adsorbents which will contribute to solving part of the wastewater treatment problem by adsorbing heavy metals and other contaminants in the water.

The regeneration of activated carbons involves restoring the adsorptive capacity of saturated activated carbon by desorbing adsorbed contaminants on the activated carbon surface.

The most common technique practiced in regeneration is the thermal vitalization in which adsorbed substances are desorbed

by volatilization and oxidation at high temperature [7]. This thermal regeneration technique is characterized by the loss of carbon (10-15%) due to oxidation and attrition, and by the cost of energy in treating the carbon to around 800-900°C [8].

Wet oxidation is a form of hydrothermal treatment. It is the oxidation of dissolved or suspended components in water using oxygen as the oxidizer. It is referred to as "Wet Air Oxidation" (WAO) when air is used. Others include microbial regeneration, and electrochemical regeneration. The regeneration efficiency is judged on the extent that it recovered the adsorption capacity of the used derived activated carbon. The following method of calculation was used to measure the rate of the recovery [7]

The formula for calculating regeneration efficiency is expressed as:

$$RE\% = A_r/A_o \times 100 \quad (1)$$

where A_r is the adsorption capacity of the regenerated adsorbent, A_o is the adsorption capacity of the fresh adsorbent and R.E% is the regeneration efficiency of the regenerated derived activated carbon.

Adsorption dynamics

The study of adsorption dynamics describes the solute uptake rate and evidently this rate controls the residence time of adsorbates uptake at the solid-solution interface. The kinetics of dye adsorption on the activated carbons was investigated using pseudo first, pseudo second-order, Elovich and intraparticle diffusion kinetic models. The conformity between experimental data and the model predicted values was expressed by the correlation coefficients (R^2 , values). A relatively high R^2 value indicates that the model successfully describes the kinetics of adsorption [8,9]. Kinetics of adsorption is one of the important characteristics defining the efficiency of the adsorption and consequently the efficacy of the adsorbent. Kinetics is an adsorption dynamics which describes the solute uptake rate and evidently control the resident time of adsorbates and possibilities of desorption [10]

Investigation of the controlling mechanism of adsorption was done by fitting the generated experimental data into the first and second-order kinetic models, generally given as described in equation

The adsorption process is described in terms of mode of transportation or diffusion as an empirical functional relationship relating uptake of adsorbates to $t^{1/2}$ rather than with the contact time as represented:

$$q_t = k_{id}t^{1/2} + C_i \quad (2)$$

where k_{id} ($\text{mgg}^{-1} \text{min}^{-1/2}$) is the rate parameter of state, i or intraparticle diffusion rate constant and is obtained from the slope of the straight line graph of q_t versus $t^{1/2}$. C_i is the intercept [11, 12]

In this present work, a low cost and available economically viable adsorbent was derived, spent as well as regenerated. Their adsorption abilities were studied as a function time. Batch mode adsorption process was adopted and reports of the applicability of various kinetic models and mechanism of transports was arrayed.

Materials and Methods

Sampling and Sample pretreatment

Coconut shells were obtained from Aliero town in Kebbi State Nigeria. Washing was carried out with plenty of water to remove the surface impurities after which it was sundried and later oven dried at 100°C over night [13].

The dried shells was pounded followed by sieving with a <2mm mesh size sieve, the less than 2mm samples were kept in airtight container.

Carbonization Process

Crucibles were washed and dried in an oven to a constant weight and the weights were taken. 5g of the pulverized coconut shell was taken into each crucible; the furnace was fired to 550°C. The weighed samples were introduced into the furnace and removed after 10 minutes, which was immediately poured into ice bath containing ice for rapid cooling. The sample was first washed with water, rinsed with 0.1M NaOH solution to remove residual ash, this was also rinsed again with distilled water, sun dried and oven dried and the final weight was taken [14]. The process was repeated until much sample was obtained.

Activation Process

3g of carbonized sample was weighed into a crucible, and impregnated with 3cm³ of 1M H₃PO₄ and was allowed to stand for some time, then the furnace was fired up to 800°C. Sample was introduced for 5minutes. The sample was firstly washed with water, then 0.1M NaOH solution [15] to remove surface ash, followed by warm and cold water rinsing to remove residual acid [16]. The sample was sun dried and then oven dried after which the weight was taken. The process was repeated in order to obtain more activated carbon.

Thermal regeneration process

Thermal regeneration was carried out on the spent activated carbon. The used activated carbon was first washed with water, then with 0.1M NaOH and rinsed with distilled water and left to dry in the oven, after which the weight was taken. The activation process was repeated and was washed with water and 0.1M NaOH [15] to remove surface ash, followed by warm and cold water rinsing to remove residual acid [16]. The sample was then dried in an oven at 105°C overnight.

Characterization of the activated carbon

Bulk density

A 100ml measuring cylinder was filled to a given volume with the produced cocos nucifera shells carbon. The cylinder was tapped for least 2 minutes to compact the carbon and then weighed. The bulk density was calculated as [17].

$$\text{Bulk density} = \frac{\text{carbon mass(g)}}{\text{Vol. of packed carbon(cm}^3\text{)}} \quad (3)$$

Moisture and dry matter content

An amount of 3.0 g each of the carbonized coconut shell samples was placed in a clean silica crucible that was previously dried in desiccators and weighed. It was then dried in an oven at 105°C for 3 h after which the sample was cooled in desiccators and then weighed. Results were expressed as percentage of the initial weight carbonized samples (W_o).

Ash content

Copper crucibles were heated in a furnace at 550°C, then cooled in a desiccator and weighed. The oven dried samples from the previous section were placed in the crucible and transferred into the Muffle furnace at 800°C for 10min. The sample were removed and poured into ice water bath. Excess water was drained. The crucibles/content was allowed to cool in a desiccator and then weighed to obtain the weight of the ash. The ash content was expresses as percentage of the oven dry weight (W_f).

Batch equilibrium studies

Batch experiment of adsorption was carried out in 250cm³ Erlenmeyer flasks; 0.1g of activated carbon was mixed with 10ml of 10ppm concentration of the Ni solution in 6 different flasks. These solutions were allowed to stand for 60,120,180,240,300 and 360 minutes contact time. Mixtures were filtered using Wattman filter paper [18,19]. Heavy metal

concentrations (mg/L) remaining after adsorption was estimated using the Atomic Adsorption Spectrophotometer.

Results And Discussion

The effect of contact time on Removal efficiency

The removal efficiency %RE was calculated using the equation 4

$$\%RE = \frac{C_0 - C_e}{C_0} \times 100 \tag{4}$$

For the derived powdered activated carbon (DPAC), percentage Ni²⁺ removal was evidently high time dependent though of a non linear pattern. Adsorption increases from 25.73%RE (60 seconds) to 35.82% (120 seconds). A desorption phenomenon was evidenced before the 180th second (%RE= 28.99). This trend continued towards the 300th second (%RE= 21.81). A similar adsorption-desorption phenomenon was also observed for Ni²⁺ uptake onto the regenerated activated carbon (RPAC). The highest contact time (360s) was linked to the least removal efficiency (47.84%) with sorption being favourable at the 240th second (%RE= 97.50%)

Effect of contact time on adsorbed concentration

The effect of contact time on the concentration of Ni²⁺, was studied (Table 1). It was observed that the initially adsorbed concentration increases with increase in contact time but the concentration gradually drops after reaching a maximum (9.759) for adsorption of Ni²⁺ at contact time 240mins. The adsorbed concentration Ca was calculated using the formula below

$$C_a = C_0 - C_e \tag{5}$$

Effect of contact time on Amount adsorbed per unit weight of activated carbon

The decrease in adsorption with increased contact time revealed that the activated carbon efficiency in removing Ni²⁺ from aqueous solution suggests that a large adsorption took place at short time intervals, close to 240s for the metals. The adsorption capacity was calculated from the equation 6

$$q_e = \frac{C_a}{C_e} \times \frac{V}{W} \tag{6}$$

FTIR spectra for commercial (CAC), derived (DAC) and regenerated carbon (RAC)

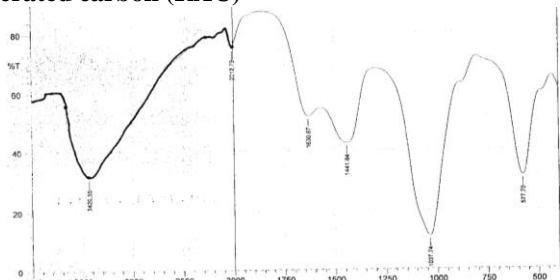


Figure 1: FTIR spectra for commercial activated carbon

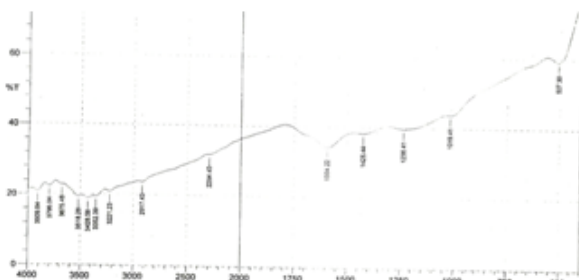


Figure 2 : FTIR Spectra for regenerated cocos nucifera shell derived activated carbon

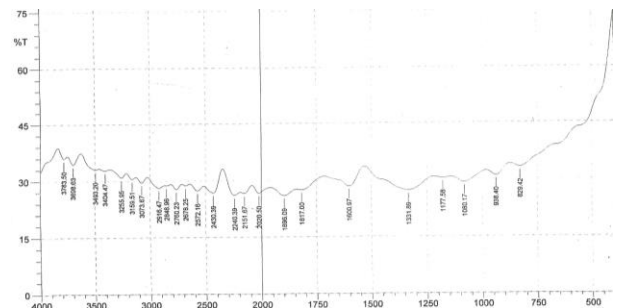


Figure 3: FTIR Spectra for cocos nucifera shell derived activated carbon

FTIR Spectra comparison between commercial and regenerated activated Carbon

The FTIR Spectra for the regenerated activated carbon showed different peaks along the functional group region (figure 2). These several peaks were seen in the FTIR Spectra of the commercial activated carbon. The 3429.55 peak of the commercial activated carbon spectra represents N-H amine functional group or N-H amide functional group; this may result from either the nature of raw material, experimental condition and environment of production of commercial activated carbon. Also the several peaks showing within 2294.40, 2917.43 and 3221.23 to 3909.84 ranges may also be as a result of un-expelled residual chemical during experimental process.

FTIR Spectra comparison between derived and regenerated activated carbon

The derived activated carbon spectra (figure 3) showed several peaks at the functional group region. Lesser peaks were observed from the FTIR spectra of the regenerated activated carbon. This observation may be as a result of repeated experimental procedures and washing stages to produce the regenerated activated carbon.

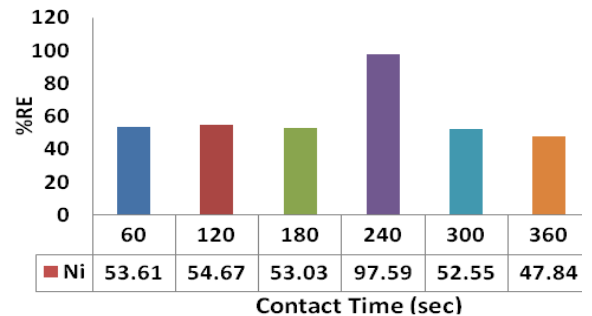


Fig. 4: Effect of Contact time on removal efficiency for adsorption of Ni onto Regenerated powdered Activated Coconut Shells Carbon

Figure 4 is a chart showing the effect of contact time on the removal efficiency for adsorption of Ni²⁺ onto regenerated powdered activated coconut shells carbon. From the result, it was observed that Ni²⁺ has the highest adsorption at contact time, 240s with %RE of 97.59.

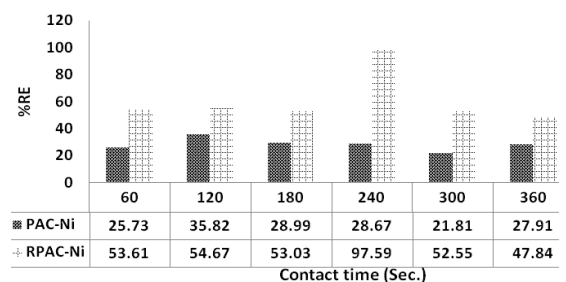


Fig. 5: Estimation of Removal efficiency for Ni adsorption onto Derived and Regenerated Powdered Activated Coconut Shells Carbon

Figure 5 compares the removal efficiency for Ni^{2+} adsorption using derived powdered activated coconut shells carbon and regenerated powdered activated coconut shells carbon. From the chart, it was obvious that Ni^{2+} has highest adsorption at time 240s with (%RE= 97.59) using the regenerated activated coconut shells carbon and also, it was clear that Ni uptake onto regenerated powdered activated coconut shells carbon is high at most of the contact time. Therefore regenerated powdered activated coconut shells carbon is more efficient in this study for the adsorption of Ni^{2+} than does the corresponding derived granular activated carbon. This evidenced the role played by surface area via a re- modification.

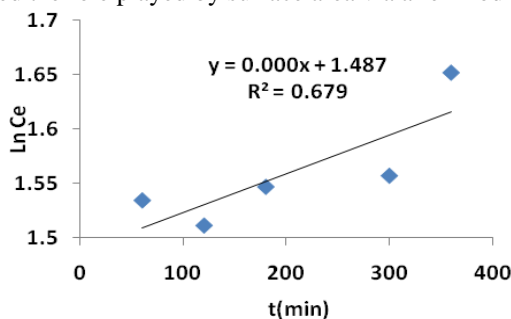


Fig. 6: Linear First Order Adsorption Plot for Ni^{2+} adsorption onto Regenerated powdered *Cocos nucifera* Activated Carbon

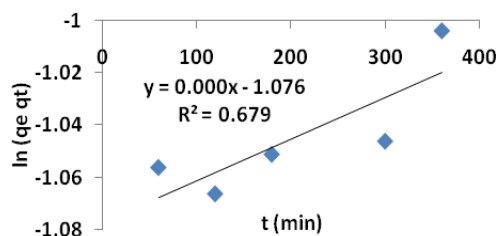


Fig. 7: Pseudo First Order Adsorption Plot for Ni^{2+} adsorption onto Regenerated powdered *Cocos nucifera* Activated Carbon

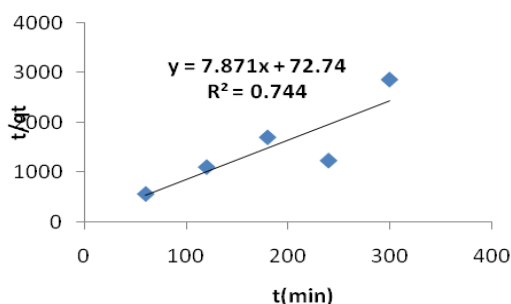


Fig. 8: Pseudo Second Order Adsorption Plot for Ni^{2+} adsorption onto Regenerated powdered *Cocos nucifera* Activated Carbon

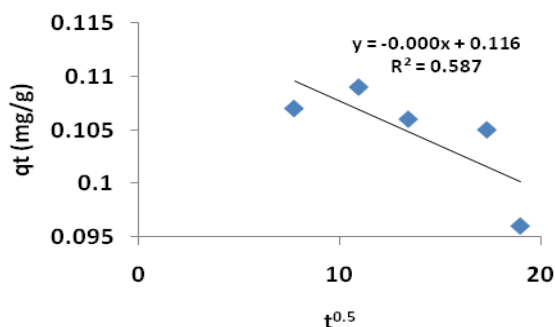


Fig. 9: Intraparticle Diffusion Plot for Ni^{2+} adsorption onto Regenerated powdered *Cocos nucifera* Activated Carbon

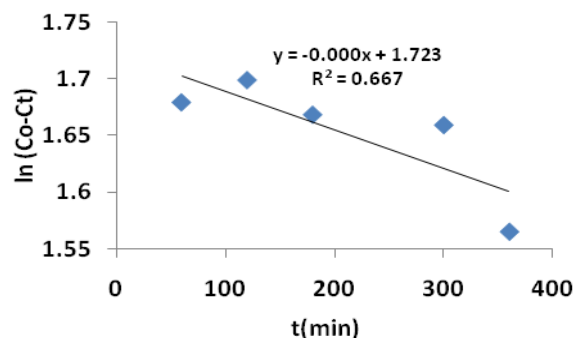


Fig. 10: Mass transfer Equation Plot for Ni^{2+} adsorption onto Regenerated powdered *Cocos nucifera* Activated Carbon

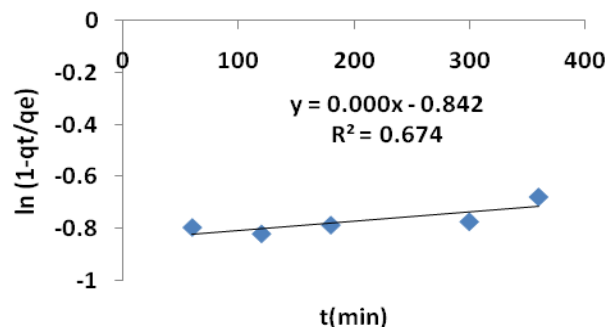


Fig. 11: Film Diffusion Equation Plot for Ni^{2+} adsorption onto Regenerated powdered *Cocos nucifera* Activated Carbon

Table 4 summarizes the data extracted from plots in Figures 6 to 11. Deductions from the table presented the values of coefficient of determination (R^2) for three kinetic models. R^2 value for the pseudo second order kinetics (0.744) is higher compared to those of linear kinetics (0.679) and Pseudo First order kinetics (0.679). It was thus believed that the second order kinetics is most applicable in the interpretation of the sorption phenomenon and rate of Ni^{2+} uptake onto regenerated powdered *Cocos nucifera* activated carbon. It should be noteworthy that the rate, $K_2 = 0.852$ is in relationship to the adsorption capacity and not necessarily defined in term of concentration. The mode of transport was also investigated. Film diffusion equation evidently applied best ($R^2 = 0.674$) to Ni^{2+} uptake onto regenerated *Cocos nucifera* shells activated carbon.

Conclusion

The activated carbon derived from *cocos nucifera* shells was found to be very efficient in the adsorption of heavy metal (Ni) as predicted from this research. It was evident from the findings of this work that Ni^{2+} was successfully adsorbed to an extent using the regenerated *cocos nucifera* shells activated carbon. From the result; it was observed that the increase in adsorption with increased contact time was non uniform. Serial adsorption-desorption routine was observed for both adsorbents at the selected contact time range. Pseudo second order kinetics best described the adsorption process while Ni^{2+} uptake onto regenerated powdered *Cocos nucifera* activated carbon is by film diffusion mode of transport. This research work has shown that agricultural waste can be converted into useful products that can help improve and protect the environment.

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Table 1: Experimental data of Ni adsorption onto Derived Powdered Activated Carbon, Co =10mg/L

Sample	Ce(mg/L)	Ca(mg/L)	q _e (mg/g)	%RE	Kc	ΔG
DPAC/Ni/60	7.427	2.53	0.051	25.730	0.346	2643.975
DPAC/Ni/120	6.418	3.582	0.072	35.820	0.558	1454.581
DPAC/Ni/180	7.101	2.899	0.0579	28.990	0.408	2234.479
DPAC/Ni/240	7.133	2.867	0.0570	28.670	0.402	2273.378
DPAC/Ni/300	7.819	2.181	0.044	21.810	0.279	3184.528
DPAC/Ni/360	7.209	2.791	0.056	27.910	0.387	2366.822

Table 2: Experimental data of Ni adsorption onto Regenerated Powdered Activated Carbon, Co =10mg/L

Experiment	Ce(mg/L)	Ca(mg/L)	q _e (mg/g)	%RE	Kc	ΔG
RPAC/Ni/60s	4.639	5.361	0.107	53.610	1.156	-360.790
RPAC/Ni/120s	4.533	5.467	0.109	54.670	1.206	-467.279
RPAC/Ni/180s	4.697	5.303	0.106	53.030	1.129	-302.668
RPAC/Ni/240s	0.241	9.759	0.195	97.590	40.494	-9231.404
RPAC/Ni/300s	4.745	5.255	0.105	52.550	1.107	-254.629
RPAC/Ni/360s	5.216	4.784	0.096	47.840	0.917	215.633

Table 3 : Kinetic Studies Experimental data for Ni²⁺ uptake onto Regenerated Powdered Activated Cocos nucifera shells (t=60 to 360s)

Experiments	q _e	q _t	Log(q _e -q _t)	t/q _t	t ^{1/2}	Ln t	Ct
RPAC/Ni/60s	0.195	0.107	-1.056	560.748	7.746	4.094	4.639
RPAC/Ni/120s	0.195	0.109	-1.066	1100.917	10.954	4.787	4.533
RPAC/Ni/180s	0.195	0.106	-1.051	1698.113	13.416	5.193	4.697
RPAC/Ni/240s	0.195	0.195	0.000	1230.769	15.492	5.481	0.241
RPAC/Ni/300s	0.195	0.105	-1.046	2857.143	17.321	5.704	4.745
RPAC/Ni/360s	0.195	0.096	-1.004	375.000	18.974	5.886	5.216

Table 4: Rate and transport mode for Ni²⁺ adsorption onto Regenerated powdered Cocos nucifera Activated Carbon

Models	Types	Experimental Data/Constants		Rate
		Linearity(y =)	R ²	
Kinetics	Linear	0.000x+1.487	0.679	NA
	Pseudo First order	0.000x-1.076	0.679	NA
	Pseudo second order	7.871x+72.74	0.744	K ₂ =0.852 q _e = 0.127
Transport	Intraparticle Diffusion	0.000x+0.116	0.587	NA
	Mass transfer	0.000x+1.723	0.667	NA
	Film Diffusion	0.000x-0.842	0.674	NA

Keys: NA –Not Applicable, q_e unit; mg/g, R²– Coefficient of determination/Applicability.

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Other Equations and Nomenclatures

$\log c_t = \frac{k_1}{2.303}t + \log c_o$ Linear form of the simple first order kinetics

$\log(q_e - q_t) = \log q_e - \frac{k_1}{2.303}t$ Linear form of the pseudo

first-order equation

$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{1}{q_e}t$ Linear form of the pseudo

second-order rate expression

$\ln(C_o - C_t) = \ln D + K_o t$ Linear form of the mass

transfer equation

C_e – adsorbates (SFA) concentration at equilibrium time (mol/L).

C_o – initial adsorbate (SFA) concentration in solution (mol/L)

q_e – amount of adsorbate in the adsorbent at equilibrium (mg/g)

q_{e.cal} –calculated amount of adsorbate at equilibrium

q_{e.exp} – measured or experimental amount of adsorbate at equilibrium.

K – specific rate constant

K₁ – rate constant in pseudo first order kinetic equation (min⁻¹)

K₂ – rate constant in pseudo second order kinetic equation (gmg⁻¹min⁻¹)

t– time (s)