

Original Research Article

REMOVAL OF HEAVY METALS FROM PRODUCED WATER USING ACTIVATED CARBON FROM COCONUT HUSK ENHANCED WITH GRAPHENE OXIDE

Abstract

Produced water, which contains a high amount of hydrocarbons, heavy metals, and other pollutants, is one of the major waste streams in the Niger Delta's oilfields and refineries. The treatment of produced water can be done in a variety of ways, including physical (desalination, membrane separation and adsorption), chemical (precipitation, absorption and oxidation), and biological. The heavy metals and contaminants in the wastewater pose a threat to living creatures and the environment. This research evaluates the effectiveness of Activated carbon modified with Graphene oxide, a new thriving material for researchers derived from graphite, to improve the adsorption capacity of coconut husk using the "wet impregnation technique" for the removal of heavy metals from produced water. Batch adsorption of the pollutant was studied for the Activated Carbon, and the modified activated carbon (AC-GO) at a time intervals, for each of the adsorbents. The removal efficiency of the adsorbent was found to increase proportionately from 6.71% to 92.96%, 3.01% to 52.23% and 51.28% to 67.77% for Cd, Ni and Pb respectively with increasing contact time. Atomic absorption spectroscopy (AAS) analysed the treated produced water samples. The adsorption isotherms and kinetics studies of the adsorbed metals were evaluated and correlated with empirical models.

Keywords: Coconut Husk, Graphene Oxide, Activated Carbon, Adsorption

Introduction

Produced water is one of the oil and gas industries' major streams of wastewater production that is accompanied by oil during the extraction process. There are events in the oil and energy sector that produce such wastewater. First, is the mixture of water and oil produced during extraction, with the source being the seawater that surrounds the oil well. Second, the water that was injected into the oilfield to bring the deep oil to the surface, produces this effluent wastewater. The fluid stream and produced water are typically separated using process equipment, such as separators. This phase separator could be two-phase or three-phase, but these devices do not guarantee 100% separation (Ajani & Isehunwa, 2022). In the majority of oil-bearing formations, it is thought that rocks were entirely saturated with water before petroleum invaded and trapped them (Al-Ghouti et al., 2019). Produced water is categorised as the largest waste stream from field operations and must be disposed of without violating environmental regulations.

Because of the volume, expense, and environmental effects of entrapped chemicals, produced water management has become crucial. As a result, several nations that produce oil have established laws and regulations regarding the quality of produced water that may be released. (Udeagbara et al., 2020)

Activated carbons, also known as activated charcoal, are carbonaceous materials that have been prepared to have a small and low pore volume to enhance the surface area, allowing them to effectively capture and hold materials. It is an exceptional and flexible adsorbent because of its microporous structure. Activated carbon (AC) is the most commonly used adsorbent for treating wastewater among other adsorbents used for both inorganic and organic contaminants (Yahya, Abubakar, et al., 2020).

Due to their agro-renewable, biodegradable waste, environmentally friendly, and affordable qualities, a variety of ways have recently been studied to find inexpensive and effective adsorbents made from a variety of waste raw materials (Man et al., 2015). To remove heavy metals from wastewater, adsorbents, including activated carbon, bio-waste, and clay have been applied

because of their surface properties, porous structures, low density, and chemical inertness, although most of them have limited adsorption capacities and selectivity. These bio-masses include corn husk (Ismail et al., 2022), almond shell (Yahya, Abubakar, et al., 2020), coconut shell (Onyeji&Aboje, 2011), orange peel (Udeagbara et al., 2020), bamboo fibre(Kuti et al., 2018), sawdust (Mdoe, 2004), and kola nut pod (Yahya, Obayomi, et al., 2020) Mangifera indica peels (Ajani &Isehunwa, 2022).

Coconut husk is one of the materials used to create the most cost-effective adsorbent material from agricultural wasteand is readily available especially within the research area.Utilizing the husk for this research reduces cost of importation of industrially produced adsorbent and reduces waste of the husks in the environment as the husks are converted to useful materials. 33–35% of the husk is made up of the mesocarp of farmed coconuts (Man *et al.*, 2015). These substances are utilized in the production of significant and useful biosorbents for wastewater treatment. If properly disposed, the detrimental impact on the environment will be reduced.

The produced water is a complex mixture with a composition of dissolved organic and inorganic chemicals that varies greatly depending on the geochemistry of the hydrocarbon-bearing formation, the field's geographic location, the extraction technique, the type of produced hydrocarbon, the age and depth of the underlying geological formation, and the reservoir's chemical makeup (Al-Ghouti et al., 2019). Produced water contains metals, which include Fe, Cr, Ba, Ni, and Zn, among others. The geological age and characteristics, injected water volume, and chemical composition all have an impact on the type, chemical content and concentrationof the metals (Udeagbara et al., 2020).

Many treatment procedures have been suggested to treat produced water since it contains a variety of contaminants in varied concentrations. Some of these contaminants might not be eliminated with a single procedure, the treatment system often requires a number of separate unit processes. Selecting a treatment system that would effectively remove the majority of impurities from produced water is difficult. To accomplish the various treatment objectives, a combination of chemical, biological, and physical treatment techniques should be applied. For several effluent water constituents, adsorption, chemical precipitation, and membrane filtration have higher removal efficiencies of above 90% (Yahya et al., 2013). As a result, the choice of methods may be influenced by the treatment cost as well as the intended reuse of treated water and the desired standards associated with effluent water discharge(Alhassan et al., 2016).

The investigation of the capacity of contaminants to adsorb to activated carbon (AC) modified with graphene oxide (GO), which is considered to be the most identified graphene composite material. Applying Hummers' method to strongly oxide graphite, a chemical process that exfoliates graphite, results in the generation of graphene oxide (GO), a precursor for the production of graphene (Yusuf et al., 2015). Graphene is a two-dimensional carbon nanomaterial that has drawn the attention of scientists in recent years. (Novoselov et al., 2012), had mentioned graphene as a "miracle fabric" due to its tremendous network, which combines an excessive three-dimensional aspect ratio and huge specific surface area, higher flexibility, especially excessive thermal and electronic conductivities, gas impermeability, and other advanced properties. Graphene oxide consists, on its surface, numerous and different types of large amounts of oxygen atoms in the form of hydroxyl, epoxy, phenol, and carboxyl groups. Due to these functional groups on graphene oxide, it is extremely hydrophilic and can be utilized in both biological and aquatic situations (Balarak et al., 2019; Kyzas et al., 2018).

Materials and Methodology

This research used coconut husk as the raw material, which was outsourced from Kure Market in Minna, of Niger State while the graphene oxide was purchased. The produced water was outsourced from an undisclosed oilfield in Warri, Delta State.

Adsorbent Preparation

Water was used to properly wash the coconut husk to remove surface contaminants after a size reduction from its initially obtained size. This process was followed by a liberal soaking and then

rinsing with distilled water (Yahya, Abubakar, et al., 2020), which was further sun-dried in 1atm and then in an oven at 80 °C until constant in weight for 30 hours. The dried husk was crushed to a fine powder and sieved through a 180-200µm mesh sized sieve to achieve a fine, uniform size. The fine powder was preserved in a tightly sealed container for further use.



Figure 1 – photos of raw and crush coconut husk before activation and produced water sample

Activation of Coconut Husk

A chemical activation methodology was adopted for the preparation of the carbon (Iyayosa Andrea et al., 2023). The fine powdered coconut was obtained after its preparation. A solution of phosphoric acid of 3M concentration was prepared and mixed with the fine powdered coconut husk in a ratio of 4:3 (cm³:g) and stirred for proper mixture. This was then left for adequate **soaking**, for 10 hours. After 10 hours of impregnation, it was sent to the muffle furnace for carbonization at 450 °C for 1 hour 30 minutes (Bello et al., 2021). This was allowed to cool inside the furnace to prevent it from becoming ash. It was collected after cooling, and washed with distilled water over and over again to a neutral pH concentration. Then the carbon was collected and dried in the oven at 70 °C for 6 hours to remove moisture. This was sieved again, to obtain uniform particle size and then collected in a tightly sealed container for characterization.

Applying the formula, the activated carbon yield was estimated:

$$X(\%) = \frac{m}{m_0} \times 100$$

Where:

X is the activated carbon yield (%)

m is the activated carbon mass (g)

m₀ is the raw sample mass (g)

Modification of prepared Activated Carbon with Graphene Oxide (GO)

Activated carbon particle sizes of 180-200 µm was taken for modification and further experimentation. 2.0 g of graphene oxide was mixed with 30 g of activated carbon in distilled water and stirred using a magnetic stirrer at 105 °C for 90 minutes, after which a centrifuge was used to remove the excess water from the mixture at 1500 rev/min for 30 mins. The resulting graphene oxide-activated carbon (AC-GO) mix was oven-dried at 120 °C for 30 minutes to remove of the moisture. The dried graphene oxide-activated carbon (AC-GO) mix was then subjected to calcination in a furnace at 400 °C for 1 hour for proper integration. The resulting dried and well-integrated graphene oxide-activated carbon mix is ready for the adsorption experiment.

It is essential to understand the common chemical characteristics of produced water, both treated and untreated, to design efficient treatment strategies for lowering the toxicity and to set acceptable limits before disposal. It revealed that this oil production platform's untreated produced water has a significant concentration of several heavy metal ions. These values for the untreated effluent in Table 1 could be comparable to or different from those found in this research area from other sources (Erakhrumen, 2015).

Table 1 - Results of analyses for sampled untreated produced water and some permissible limits for heavy metals in effluent

Heavy Metals and their concentration level in the produced water sample (mg/L)						
	Cadmium	Nickel	Iron	Manganese	Copper	Lead
Result for initial concentration of Produced water Sample	0.9067	0.6121	0.0944	0.2374	2.0013	2.9044
Inland Effluent Limitations (Erakhrumen, 2015)	0.003	0.07	1.0 – 2.0	0.4	0.03	0.05– 0.01

Table 1 shows the concentration of the various heavy metals present in the produced water sample, which is to be adsorbed to meet the specifications of Nigeria's regulations. Although this research will focus on mainly three metals due to their possible effects, as they exceed the specifications provided.

Adsorption Process

250 cm³ of the produced water sample was mixed with 5 g of the adsorbent. The mixture was then agitated using a thermostatic shaker at a constant agitation speed of 120 rpm at room temperature. Filtration was carried out across a range of time durations. As a result, five different combinations were examined for contact times of 5, 10, 20, 30, 40, 70, 100, and 120 minutes on each. To track changes in the heavy metal concentration, the filtrate was subjected to an AAS analysis (Abdulsalam, Alhaji Yaro, et al., 2023). This was to ascertain the optimum dosage of the adsorbent. According to the equation, the removal efficiency and adsorption capacity, q_e mg/g, were determined.

$$Q_e = \frac{V(C_i - C_e)}{W} \quad (1)$$

The formula was used to calculate how much metal was absorbed from the wastewater.

Where:

Q is the amount of metal adsorbed (mg/g)

C_i is the initial concentration of metal

C_e is the concentration of metal (mg/L) at equilibrium

V is the volume of solution (L)

M is the mass of the adsorbent used (g) (Balarak et al., 2019).

The removal efficiency was then determined by the expression (Obayomi et al., 2022)

$$\% q = \frac{(C_i - C_e)}{C_i} \times 100 \quad (2)$$

Result and Discussion

The AAS results of produced water at various contact time shown in table 3

Table 2 - Effective of adsorbent contact time on heavy metal adsorption

Time (mins)	Concentration (mg/L)		
	Cadmium	Nickel	Lead
0	0.9067	0.6121	2.9044
5	0.8459	0.5937	1.415
10	0.2521	0.2569	0.9695
20	0.0984	0.3056	0.7962
30	0.0550	0.2777	0.5819
40	0.0516	0.3170	0.9601
70	0.0597	0.2750	0.9476
100	0.1251	0.2341	0.5224
120	0.0638	0.2924	0.9361

Table 3 - Removal efficiencies at various contact times of adsorbent

Time (mins)	Cadmium (%)	Nickel (%)	Lead (%)
5	6.71	3.01	51.28
10	72.20	58.03	66.62
20	89.15	50.07	72.59
30	93.93	54.63	79.96
40	94.31	48.21	66.94
70	93.41	55.07	67.37
100	86.20	61.75	82.01
120	92.96	52.23	67.77

Effect of contact time on removal efficiency

An important factor for assessing the adsorption capabilities of adsorbents is the contact time between the adsorbent and the adsorbate (Andrea et al., 2023). Results of the contact time influence on removal effectiveness were shown in table 3. There was an increase in the adsorption efficiency slowly within 10 minutes. The adsorption equilibrium was established within 15 – 20 minutes. In general, longer contact times were advantageous to promote more effective heavy metal adsorption (Cui et al., 2015). The adsorption increased very rapidly because of the active sites present in the adsorbent at the initial timing.

The prolonged process of absorption made the longer contact time advantageous for the adequate loading of Cd, Ni, and Pb on the adsorption sites of AC-GO (Abdulsalam, Ibrahim, et al., 2023). Take Cd as an example; the removal efficiency rose vividly within 10 minutes and reached its peak in 40 minutes. This is similar to the adsorption on Ni and Pb. The initial Cd adsorption sites for AC-GO were adequate. As time went on, more and more adsorption sites were occupied.

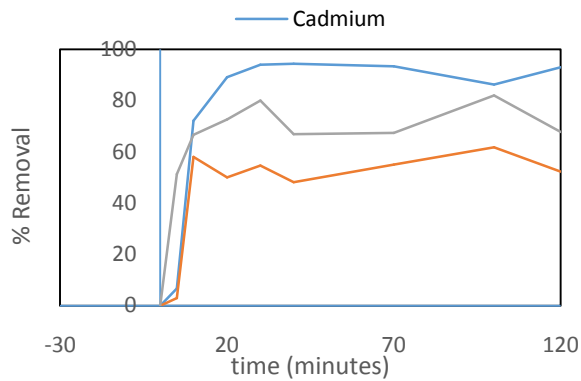


Figure 2 – Plot of % removal of Cd, Ni, and Pd against time

Removal of Cadmium: The modified adsorbent showed good adsorption capacity in the removal of Cadmium. Within 5 minutes of contact time, cadmium concentration reduced from 0.9067 mg/L to 0.8459 mg/L, 0.9067 - 0.2521 mg/L, and 0.0638 mg/L after 120 minutes. The removal efficiency at various time intervals was determined.

Removal of Nickel: The modified adsorbent showed good adsorption capacity in the removal of Nickel. The concentration of nickel reduced from 0.6121 mg/L to 0.5937 mg/L within 5 minutes contact time, 0.6121 - 0.5937 mg/L and 0.2924 mg/L after 120 minutes. The removal efficiency at various time intervals was determined.

Removal of Lead: The modified adsorbent showed good adsorption capacity in the removal of Lead. Within 5 minutes of contact time, Lead concentration reduced from 2.9044 mg/L to 1.4150 mg/L, 2.9044 - 1.4150 mg/L and 0.9361 mg/L after 120 minutes. The removal efficiency at various time intervals was determined.

Adsorption isotherms used for the analysis

The Langmuir Model

The relationship between the amount of extracted material and its equilibrium concentration is described by the isotherm model. The basis of the Langmuir model is the presumption of monolayer adsorption on a uniform surface with similar active sites (Obayomi et al., 2022). The model considers homogeneous adsorption on the surface of the adsorbent.

$$\frac{C_e}{q_e} = \frac{1}{q_m K_L} + \frac{C_e}{q_m} \quad (3)$$

Where;

- K_L (L/g) is the Langmuir constant,
- q_e (mg/g) is the amount of solute adsorbed
- q_m (mg/g) is the maximum capacity at equilibrium
- C_e (mg/L) is the equilibrium concentration

A plot of C_e/q_e against C_e on a Cartesian graph is used to express the model

Freundlich isotherm

Adsorption processes that occur on heterogeneous surfaces are covered by this isotherm. The Freundlich model is predicated on the idea that the surfaces of the adsorbent and adsorbate are heterogeneous (Obayomi et al., 2022). It provides an equation for both the exponential distribution of functional adsorption sites and surface heterogeneity. The isotherm's mathematical expression is given as

$$\log q_e = \log K_F + \frac{1}{n} \log C_e \quad (4)$$

Where;

K_F (mg/g) is the Freundlich constant,

$\frac{1}{n}$ is the heterogeneity factor or adsorption intensity

q_e (mg/g) is the amount of solute adsorbed

C_e (mg/L) is the equilibrium concentration

A plot of $\log q_e$ against $\log C_e$ on a cartesian graph is used to express the model

Table 4 – Analysis of Cadmium using Langmuir and Freundlich model for the adsorbent

Time (minutes)	Metal (Cadmium)				
	C_e	q_e	C_e/q_e	$\log C_e$	$\log q_e$
0	0.9067				
5	0.8459	0.0030	278.257	-0.0727	-2.5171
10	0.2521	0.0327	7.702	-0.5984	-1.4851
20	0.0984	0.0404	2.435	-1.0070	-1.3935
30	0.0550	0.0426	1.292	-1.2596	-1.3707
40	0.0516	0.0428	1.207	-1.2874	-1.3690
70	0.0597	0.0423	1.410	-1.2238	-1.3732
100	0.1251	0.0391	3.201	-0.9027	-1.4080
120	0.0638	0.0421	1.514	-1.1952	-1.3753

Comparison between Langmuir and Freundlich models

A plot of $\log q_e$ versus $\log C_e$ for Lead adsorption shows that the process follows the Langmuir sorption model. The gradient and intercept of the plot were used to calculate q_m and K_L . The R^2 value was calculated to be 0.9699 which is higher than the R^2 value for the Freundlich equivalent (which was estimated to be 0.9309). The maximum adsorption capacity (q_m) for Lead also found to be 62.4 mg/g. This shows the suitability of the Langmuir model for the adsorption equilibrium study of Lead. The values of q_m and K_L (Table 5) indicate that the adsorption process is favorable.

Table 5 – A comparison of parameters for the adsorbent treatment

		Metals		
		Cadmium	Nickel	Lead
Langmuir	R^2	0.9500	0.9524	0.9699
	q_m	0.0028	0.0005	0.0624
	K_L	-11.396	-3.7208	-3.2689
Freundlich	R^2	0.7788	0.9298	0.9309
	n	-1.0704	-0.2877	-2.2722
	K_F	4.18E-03	1.8E-04	9.3E-02

A plot of C_e/q_e versus C_e for lead adsorption and a plot of $\text{Log } C_e$ against $\text{Log } Q_e$, are shown in

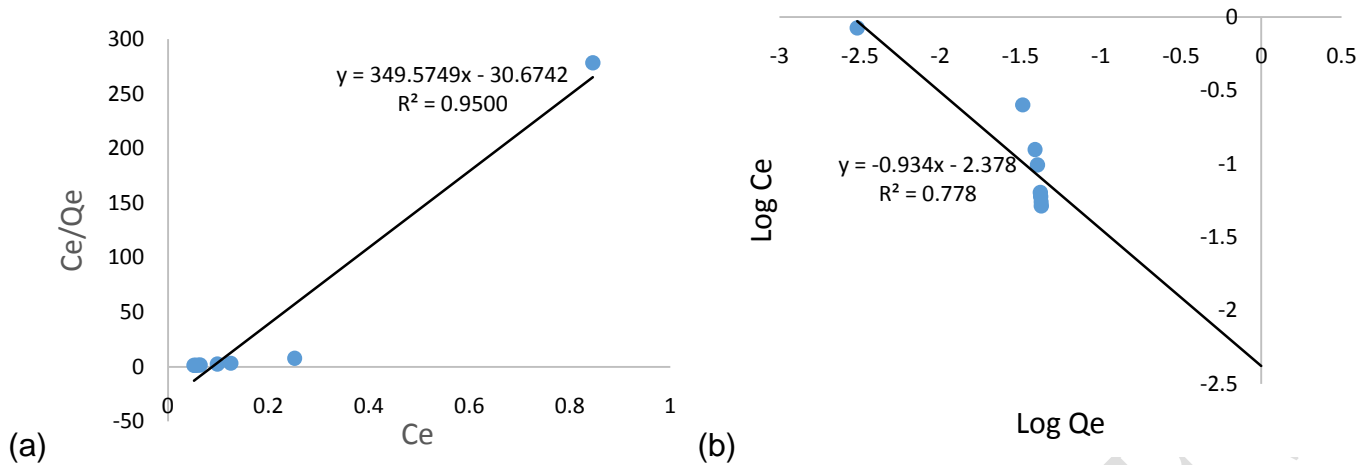


Figure 3 – Analysis of Cadmium using (a) Langmuir and (b) Freundlich Isotherm

According to Figure 1, the plot of C_e/q_e vs C_e for cadmium adsorption, the process follows the Langmuir model. The gradient and intercept of the line in figure 1 were used to get the values of q_m and K_L . For Cd (Langmuir), the correlation coefficient (R^2) was determined to be 0.9500, maximum adsorption capacity at equilibrium (q_m) was obtained to be 2.86 mg/g, while for the same Cd, it was 0.7788 (Freundlich). The Freundlich sorption isotherm model is not appropriate for investigating Cd, as shown by the poor correlation coefficient from the Freundlich isotherm lot of $\text{Log } Q_e$ vs $\text{Log } C_e$ in Figure 1(b). The R^2 value indicated that the data is fitted to the Langmuir isotherm model.

Kinetic Studies

Adsorption kinetics is important for determining the amount of time and rate at which solutes are absorbed throughout the adsorption process (Abdullahi et al., 2018). In the current study, heavy metal adsorption kinetics were investigated at various time points utilising pseudo-first and second-order kinetics model.

The model for pseudo-first-order kinetics: The $\log (C_e - C_t)$ against time interval was plotted to estimate the values of k and Q_e for the pseudo-first-order kinetic model. The initial sorption rate was calculated using the equation shown below:

$$\log (C_e - C_t) = \log Q_e - (K_1/2.303)t \quad (5)$$

where K_1 is the rate constant of adsorption (min^{-1}), which is determined from the slope, and C_e and C_t are the quantity adsorbed in mg/g at equilibrium, time 't' in minutes.

The equation provides the pseudo-second-order kinetic model:

$$\frac{t}{C_t} = \frac{1}{K_2 C_e^2} + \frac{1}{C_e} t \quad (6)$$

where K_2 is the pseudo-second-order adsorption process's rate constant. At various initial concentrations, the linear graphs of t/C_t vs t demonstrate good agreement between experimental and computed C_e values.

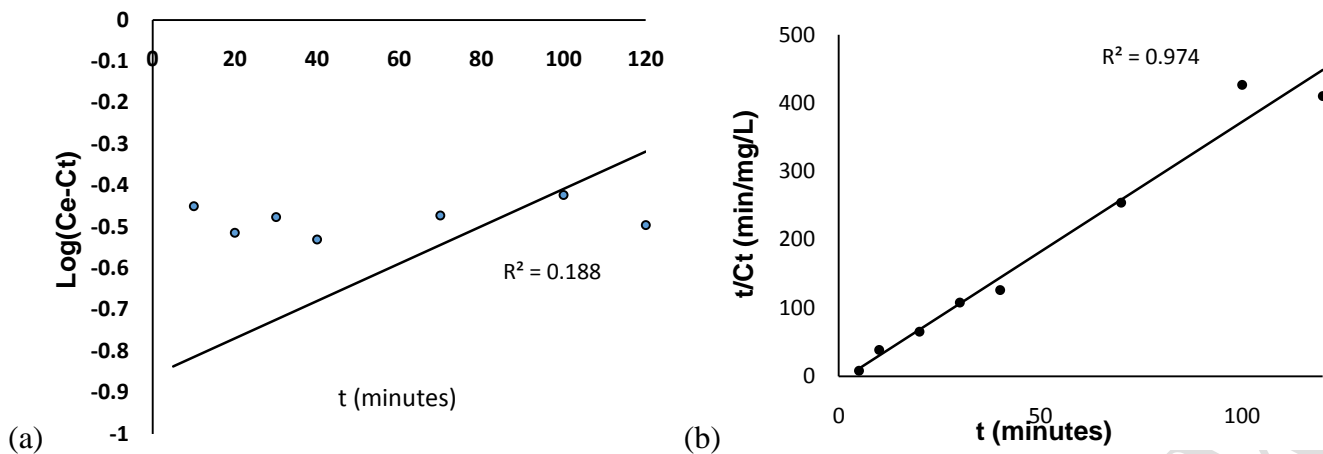


Figure 4 – Adsorption Kinetic Analysis of Nickel using (a) Pseudo-first order and (b) Pseudo-second order

Table 6 – A comparison of parameters for the Kinetic studies of the adsorbent treatment

	Metals			
		Cadmium	Nickel	Lead
Pseudo-first order	R ²	0.2032	0.1980	0.1641
	Q _e	0.6424	1.7461	1.3111
	K ₁	9.67E-3	4.83E-3	-1.38E-3
Pseudo-second order	R ²	0.8324	0.9743	0.8271
	C _e	0.0601	0.2629	0.7294
	K ₂	2.6165	-1.8414	-0.9493

For the sorption of Nickel onto graphene oxide-modified coconut husk, a poor correlation was obtained for the linear form of pseudo-first order with a low value of R² (0.1883) as compared with pseudo-second-order kinetics with a value of R² (0.9743) which is a suitable approach for this study. The computed C_e values, obtained from the linear plots, and the correlation coefficient values do not match the experimental C_e values. This same conclusion was also reached by (Batool et al., 2018). The experimental C_e value (0.0638) closed to the calculated C_e (0.0601)

Table 7 – Pseudo-second order calculated C_e for the heavy metal adsorption.

Cadmium	Nickel	Lead
0.0601	0.2629	0.7294

Conclusion

In this work, the effectiveness of modified coconut husk with graphene oxide in the removal of heavy metals (Cadmium, Nickel, Iron, Manganese, Copper, and Lead) from produced water was investigated. Heavy metal adsorption was carried out in discontinuous mode, and the effect of contact time were studied. Although, the studies were limited to Cadmium, Nickel and Lead. Also, the adsorption process was evaluated using Langmuir and Freundlich isotherm models. Metal concentrations (Cd, Ni, Pb) were found to be reduced by 92.96%, 52.23%, and 67.77 respectively, for 120 minutes. Hence, the graphene oxide modified coconut husk adsorbent is effective in the treatment of produced water. The adsorption method of treatment is highly recommended for industrial adoption. The adsorbents are inexpensive, available, and eco-friendly.

Recommendation

This adsorbent should have longer contact time intervals and a reduced adsorbent dosage to give a better description of the adsorption process which would also improve the isotherm modelling and also enable the adsorbent to adsorb the heavy metals to its surface. Adsorbents should be modified to prevent early degradation, and the lifespan of the adsorbent should be determined. Other agricultural waste products, apart from the coconut husk, should be modified with graphene oxide and used for the studies.

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