

REMOVAL OF CRUDE OIL FROM AQUEOUS SOLUTION BY ZINC CHLORIDE MODIFIED *DIOSCOREA ROTUNDATA* PEEL CARBON: EQUILIBRIUM, KINETIC AND INTRA-PARTICLE DIFFUSIVITY

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Received June 19, 2018; Accepted September 28, 2018

Abstract

This study evaluated the potential of unmodified yam peel carbon (UYPC) and Zinc chloride activated yam peel carbon (AYPC) as low cost sorbent for remediation of crude oil from aqueous solution. Their efficiencies on the adsorption of crude oil from the water were investigated through batch adsorption studies. The crude oil obtained was characterized, and properties such as viscosity (kinematic and dynamic), pH, density, specific gravity, and API gravity were determined. Thermal properties such as heat of combustion, thermal conductivity, specific heat capacity and latent heat of vaporization were also determined. Proximate analyses were carried out on the raw yam peels, and the physicochemical properties were examined after carbonization. The AYPC was found to be more efficient than UYPC in removing oil from water under the varied conditions of pH (3 to 13), concentration (2.0 to 5.0 g/L), time (0 to 80 min) and dosage (0.2 to 1.4g). AYPC sorption of crude oil required a much lesser dosage compared to UYPC to obtain maximum removal. The adsorption experiment gave the optimum conditions of pH 7, and contact time of 40 mins for both sorbents. The equilibrium adsorption test conducted showed that the Langmuir isotherm is a better fit for the adsorption of the crude oil on AYPC and the Freundlich model was a better fit for UYPC. The kinetics studies showed that Pseudo-second order kinetics is a better fit for both sorbents than the pseudo first order model. The intra-particle diffusion evaluation revealed the influence of film diffusion and external mass transfer. The sorbents were found to be very effective as low cost materials for treatment of oil spill waters.

Keywords: Oil spill; sorption; crude oil; remediation.

1. Introduction

The oil spill is the main source of oil contamination in water which is a result of the unintentional introduction of oil into the environment during the extraction, production, transportation or storage processes. When the oil is explored, transported and stored and its derivatives are used, there is a risk for spillage with the potential to cause significant environmental impact [1]. In recent times, major environmental pollution of soil and water is due to hydrocarbon contamination resulting from petrochemical industries activities. It can be caused by accidental discharge of petroleum industries products into the environment; it can also be caused by human activity. Hydrocarbon compounds are known for its carcinogenic and neurotoxic behavior. The overall annual intake of petroleum hydrocarbon around the world is very high, and it is approximately about 1012 US gallons [2]. Higher concentration of the hydrocarbon molecules, which are the main constituent of crude oil and petroleum products are highly toxic to living beings, including humans. Petroleum products also comprise trace amounts of sulfur and nitrogen compounds, which are hazardous and can react with the environment to produce secondary poisonous chemicals [3].

The oily wastewaters and produced waters generated from oil and gas exploration activities, crude oil refinery, petrochemical and petroleum refining plants, steel manufacturing and metal works, vehicle repair, and various types of manufacturing plants are other sources of water contamination by oil. The discovery of oil has so much influenced the world's economy that it appears to be one of the major sources of energy in homes and industries. At the same time, it has constituted a huge source of environmental disaster. Contaminated water cannot be used as a municipal water supply in industry, or for irrigation [4]. With oil continuing to pour into the Gulf of Mexico, the Deepwater Horizon drilling rig explosion, which occurred on April 20, 2010, has been described as the worst environmental disaster in U.S history. Every year approximately 5 million tons of petroleum oil is shipped across the oceans in shipping containers which place the marine life and the ecosystem at high risk [5]. Oil spills can also affect marine life, birds, and plantation. In 2010 the BP Deep Horizon rig was lit in flames leading to the death of 11 workers and released approximately 5 million barrels of oil into the Gulf of Mexico. The oil spills lasted for three months which led to the death of a huge population of marine animals and polluted 320 km of shorelines [5]. Oil spills are a common event in Nigeria. Half of all spills occur due to pipeline and tanker accidents (50%), other causes include sabotage (28%) and oil production operations (21%), with 1% of the spills being accounted for by inadequate or non-functional production equipment. Corrosion of pipelines and tankers is the rupturing or leaking of old production infrastructures that often do not receive inspection and maintenance [4]. Oil in water constitutes a major contaminant and research are ongoing to develop techniques for removing these contaminants, especially from low cost agricultural waste. There are many techniques available for oil-in-water separation regardless of being physical, biological or chemical such as gravity separation, chemical treatment methods, flotation system, coagulation, filtration, hydrocyclone, electrical process, reverse osmosis and membrane reactor which offer advantages and drawbacks over others [6]. Some existing methods are expensive, and sometimes some supplementary treatments are needed in order to achieve the stipulated environmental standards. Among these methods, the sorption process has emerged as a highly efficient treatment method due to its simplicity of the design, ease in operation and inert to toxic substances in removing dissolved organic components from water.

The high commercial sorbent costs and stringent environmental regulations have resulted in the need to develop low-cost sorbents derived from biomass materials, such as wheat straw, sawdust, rice residues, corncob, coconut husk, kapok fibers, cotton, wool and wood [7-9]. The advantages of these materials include being low cost, biodegradability, and non-toxicity. Among the main existing oil removal techniques, the sorption process has gained high popularity as it is one of the easiest and most cost effective physiochemical methods for removing contaminants from aqueous solution [10]. Synthetic organic polymers such as polypropylene, polyurethane, and polyethylene have been widely used to remove oil. Synthetic polymers have a high sorption capacity, but their major drawbacks are non-biodegradability and high cost. Over the years, researchers have tried to use adsorption as a technique for the control of environmental pollution from heavy metals and oil spills. Intensive researches have been done on various sorbents for oil spills control, and both their thermodynamic and kinetic studies have been reported. The highly hydrophobic characteristics of biomass combined with its high porosity develop a capillary force towards the adsorption of oils. Vegetal tissues, with the large surface area and big pores, tend to adsorb organic contaminants through physical and chemical mechanisms, in a similar way to charcoal. Several researchers, while studying the adsorption of oil by-products using different types of biomass, have observed the promising character of these materials as adsorbents [11]. It was observed that the application of carbon in the form of commercial activated carbon or charcoal (ACC), which had proved to be very effective in treating most complex industrial wastewater, is exorbitantly expensive and about 10–15% of it, is lost during regeneration [12]. Therefore there is a need to source for a cheap alternative form of effective carbon-based adsorbents. However, the use of yam peel carbon and its zinc chloride activated form for crude oil removal hasn't been established. The aim of

this study was therefore to utilize yam peel carbon, and its zinc chloride activated derivative as low cost sorbents for crude oil removal from aqueous solution.

2. Experimental

2.1. Sampling

Waste yam peels were collected, washed and dried in sunlight for one week. The sundried peels were further dried in an oven at 110°C for one hour. The crude oil was obtained from Warri refinery and petrochemical company (WRPC) in Warri, Delta state of Nigeria.

2.2. Characterization of crude oil and proximate analysis of yam peels

The physical properties of the crude oil such as density, specific gravity, viscosity, and thermal properties were determined using ASTM methods (ASTM D1298-95, ASTM D445-01). The proximate analysis of the yam peels such as moisture content, ash content, crude fibre, crude protein, carbohydrate, and crude fat were determined using standard methods [13].

2.3. Carbonization of the yam peels

The dried yam peels were carbonized in a muffle furnace at 400°C for 1hr in the absence of air. The charcoal obtained was allowed to cool, grounded, and sieved to obtain a uniform particle size. This was then stored in a plastic container as Unmodified Yam Peel Carbon (UYPC).

2.4. Chemical activation of the carbonized yam peel

Two hundred grams (200g) of the carbonized material was divided into two equal portions. 100g was transferred into a beaker mixed with 150mL 0.1M ZnCl₂ until the mixture formed paste. The paste was transferred into a dry crucible, oven dried for 1hr at 105°C and finally introduced into the muffle furnace and heated at 520°C for 2 hrs in the absence of air so as to increase the surface area of the sample for the adsorption process. It was then cooled at room temperature, washed with distilled water until the filtrate obtained a pH of about 7. The washed activated carbon was dried for 3hrs in an oven at 105°C and then stored in a plastic container as the Activated Yam Peel Carbon (AYPC) [14].

2.5. Fourier Transform Infrared spectroscopy analysis

The Infra-Red spectra of the samples were obtained before and after the adsorption analysis for the activated and inactivated portion of the sample using a Fourier transform infra-red spectrometer. This was carried out to identify the functional groups that were responsible for adsorption.

2.6. Batch sorption experiment

The adsorption analysis was carried out by a batch process. A definite concentration of the crude oil was prepared (18g in 2.5L of distilled water). The adsorption experiments were carried out by agitation of AYPC and UYPC in required amounts with 50mL of the oil/water mixture at room temperature in an orbital shaker. Several parameters affect the uptake on the adsorbent, so studies were undertaken to choose the best conditions. The adsorptions were carried out at various pH (3.0 -13.0), the pH of the working mixtures was adjusted to the desired value with 0.1M HCl or 0.1M NaOH, contact time (0-80min), initial crude oil-water concentrations (2.0 – 5.0 g/L) and adsorbent dosage (0.2 – 1.4g). At the end of a preliminarily determined period of time, samples were withdrawn by vacuum filtration and analyzed using UV adsorption spectrophotometer to obtain the final concentrations of the oil in the sample. The amount of crude oil adsorbed per unit mass of adsorbent at equilibrium, (q_e mg/g), or at time t , (q_t) were calculated according to the following relations;

$$q_e = (C_o - C_e) \frac{v}{m} \quad (1)$$

$$q_t = (C_o - C_e) \frac{v}{m} \tag{2}$$

where C_o and C_e are the initial and final concentrations of crude oil (g/L), v is the volume of the crude oil-water mixture (L), m is the amount of adsorbent used (g). The percentage of oil removed was determined using the equation;

$$\% \text{ Removal} = (C_o - C_e) \frac{100}{C_o} \tag{3}$$

Adsorption data obtained from the effect of initial concentration and contact time were employed in testing the applicability of adsorption isotherm and adsorption kinetics respectively.

3. Results and discussion

3.1. Characterization of the yam peel and sorbents

The result of the proximate analysis of the yam peel as well as the characterization of UYPC and AYPC are shown in Table 1. The proximate analysis presented the low amount of moisture content, ash content, crude fiber, crude protein, and crude fat. The low crude fiber value of the yam peel obtained indicated its usefulness in producing a good activated carbon [15]. Crude protein is an organic compound, and the low organic material is necessary to produce activated carbon with low ash content [16]. The result for the characterization of the sorbents showed a lower bulk density of AYPC than that of UYPC. Generally, the lower the bulk density, the higher the porosity and surface area. This suggests that AYPC would be more efficient as a sorbent for crude oil remediation than UYPC.

Table 1. Proximate analysis of yam peel and sorbents characterization

Parameter	Yam peel	UYPC	AYPC
Moisture content (%)	5.81	0.50	0.48
Ash content (%)	4.99	14.0	13.2
Crude fibre (%)	11.40	-	-
Crude protein (%)	10.58×10^{-5}	-	-
Crude fat (%)	0.61×10^{-5}	-	-
Carbohydrate content (%)	78.01	-	-
Bulk density (g/cm ³)	-	0.210	0.192
Carbon yield (%)	-	69.1	71.3

3.2. FTIR analysis

Fourier Transform Infra-Red (FTIR) analysis determined for UYPC and AYPC before and after sorption of crude oil and the absorption bands are presented in Table 2.

Table 2. FTIR spectra absorption bands of the sorbents before and after crude oil uptake

Sample	Functional groups			
	O-H _{str}	C-H _{str}	C-O _{str}	C-N _{str}
UYPC before sorption	3404 cm ⁻¹	2930 cm ⁻¹	1388 cm ⁻¹ 1122 cm ⁻¹ 1049 cm ⁻¹	2381 cm ⁻¹
UYPC after sorption	3420 cm ⁻¹	2926 cm ⁻¹	1383 cm ⁻¹ 1267 cm ⁻¹ 1157 cm ⁻¹	2371 cm ⁻¹
AYPC before sorption	3431 cm ⁻¹	N.D	1434 cm ⁻¹ 1050 cm ⁻¹	2359 cm ⁻¹
AYPC after sorption	3430 cm ⁻¹	2929 cm ⁻¹	1437 cm ⁻¹ 1050 cm ⁻¹	2364 cm ⁻¹

Some functional groups were revealed which were responsible for the sorption characteristics of the sorbents. The band at 3404 cm⁻¹ (broad peak) was indicative of a hydroxyl functionality of absorbed water which became more intense after activation giving a peak at 3431 cm⁻¹ (narrow and sharp). The C-O_{str} peaks observed for UYPC became sharper and more intense after activation, showing two distinct peaks at 1434 cm⁻¹ and 1050 cm⁻¹. The peaks

observed for the Nitrile functionality also became more intense and distinct after activation. The peaks around 2900 cm^{-1} indicate C-H_{str} of alkane which was not detected after activation. The C-H_{str} was again observed after adsorption which is most likely from the adsorbed crude oil. Slight differences in absorption bands after crude oil sorption showed that the functional groups of both adsorbents were utilized in the removal process.

3.3. Physical and thermal characterization of crude oil

The physical and thermal properties of the crude oil are presented in Table 3. Watson and Nelson [17], depicted a relationship between crude oil gravity and characterization parameter: that lower gravity crudes tend to be more naphthenic, while higher-gravity crudes tend to be more paraffinic. From the experimental data obtained for the crude oil sample, it indicates that it contains more paraffinic components and less naphthenic components.

Table 3. Physical and thermal properties of the crude oil

Parameter	Value
pH	6.203
⁰ API gravity (Degrees)	37.6
Density (g/cm ³)	0.814
Kinematic viscosity (cSt)	1.1
Dynamic viscosity, (cP)	0.895
Heat of combustion, Q _v (cal/g)	10,642.3
Thermal conductivity, K (BTU. ⁰ F ⁻¹ hr ⁻¹ ft ⁻¹)	0.0427
Specific heat, c kcal/(kg ⁰ C)	0.495
Latent heat of vaporization, L (kcal/kg)	229.35

3.4. Effect of contact time on crude oil sorption

The effect of contact time on the removal of crude oil from water was studied as shown in Fig.1.

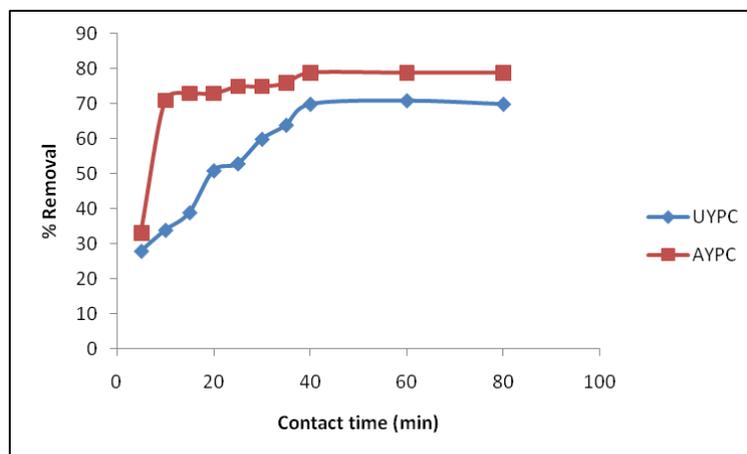


Fig.1. Effect of contact time on crude oil sorption on the sorbent

An initial concentration of 5.0g/L of the crude oil was prepared. 0.3g of UYPC and AYPC was utilized by varying the time from 0 to 80 min at 5 min interval and 20 min interval after 40 min. The adsorption was carried out in the 15mL crude oil-water mixture. The concentrations of the filtrate were obtained by running the absorbance in a UV-Visible spectrophotometer at 320nm and extrapolating from the calibration curve. For UYPC, an increase in the percentage removal from 28 to 71 as the time increased from 5 to 60 min was recorded after which the % removal decreased to 70 at 80 min. This is as a result of desorption of the oil into the solution. Also for AYPC, an increase in the percentage removal as the time increased from 5 to 40 min was obtained with the maximum removal of 79%. During the sorption process, the particles of

the crude oil attach to the surface of the adsorbent by forces of attraction and occupy/ clog the available sites. The longer the time of contact, the more the available sites would be occupied until all the adsorbent sites are filled up. This is when the breakthrough time is achieved and no more adsorbate can be sorbed. The plot of percentage removal against time thus became constant.

3.5. Effect of sorbent dosage on crude oil sorption

The effect of sorbent dosage on the removal of crude oil from water by UYPC and AYPC was investigated as shown in Figure 2.

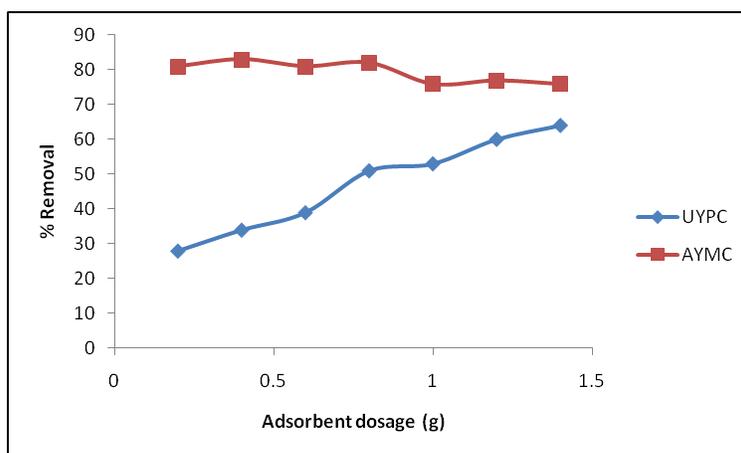


Fig.2. Effect of sorbent dosage on the sorption of crude oil on the sorbents

An initial concentration of 5.0g/L of the crude oil was prepared. The adsorbent dosage was varied from 0.2 to 1.4g at an interval of 0.2g. 20mL of the crude oil- water solution was used and the time was kept constant at 60 min. The percentage removal of crude oil increased steadily UYPC as the adsorbent mass was increased from 0.2 to 1.4g, with the optimum removal of 64%. The percentage removal of crude oil increased steadily as the adsorbent mass was increased from 0.2 to 0.4g for AYPC where the highest percentage removal was attained at 83%. Beyond this, there was a decrease in the % removal depicting desorption. This shows that a smaller adsorbent mass of activated carbon is needed compared to the inactivated carbon. The decrease in the percentage removal after attaining the highest adsorption percentage as the adsorbent increases may be due to the aggregation of adsorption sites resulting in a decrease in the surface area available for adsorption and an increase in the diffusion path-length. The decrease in percentage removal at 1.0g dosage was due to lack of achieving equilibrium between the amounts of oil adsorbed by the sorbent and the amount of oil remaining in the solution. As a result, active sites of the adsorbent were not fully bound to the oil molecules as a result of the weak van de Waal's force of attraction between the adsorbate and adsorbent.

3.6. Effect of crude oil concentration on the percentage removal

The effect of initial concentrations was studied in order to analyze the sorption concentration dependence, and thus the sorption isotherm which allows describing the nature of interactions between the sorbate and sorbent. The study on the effect of initial concentration on oil sorption onto UYPC and AYPC was carried out by varying the initial oil concentration from 2.0 g/L to 5.0 g/L at 0.5 g/L interval, while other parameters sorbent dosage (20 mg/mL), contact time (60 min) and pH (6.5) were kept constant. The result is shown in Figure 3. It was observed that for UYPC, an increase in the initial concentration of the crude oil- water solution resulted in an increase in the percentage removal of crude oil. As the initial concentration increases, the percentage removal increased from 25% at 2.0g/L to 52% at 4.5g/L

after which there was a decrease in the percentage removal. For AYPC it was discovered that an increase in the initial concentration of the crude oil- water solution resulted in an increase in the percentage removal of crude oil. As the initial concentration increases, the percentage removal increased from 35% at 2.0g/L to 73% at 4.5g/L, where there was maximum sorption capacity after which there was a decrease in the percentage removal. This is because all sorption sites on the sorbent surfaces were completely occupied by oil, and this hindered more oil to be sorbed. Increasing the initial concentration would increase the mass transfer driving force, and, therefore, the rate at which molecules pass from the bulk solution to the particle surface. This would result in a higher adsorption capacity. When the sites at the sorbent are occupied, there would be opposition to the mass transfer driving force which would lead to desorption [18].

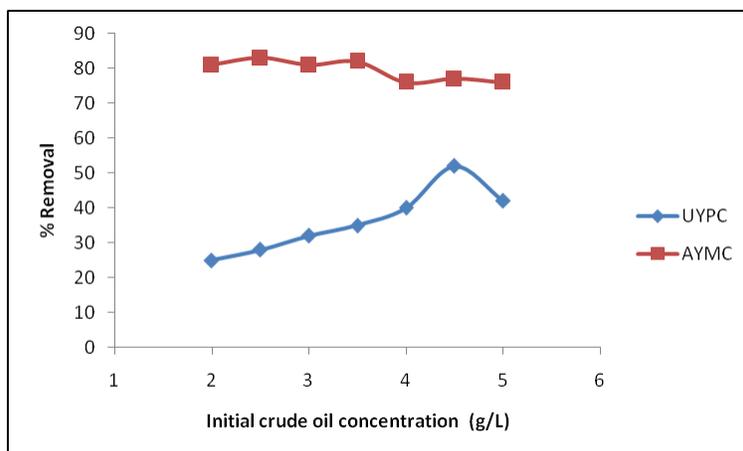


Fig.3. Effect of crude oil concentration on sorption by the sorbents

3.7. Effect of solution pH

The effect of pH on the removal of crude oil from aqueous solution was studied as shown in Figure 4.

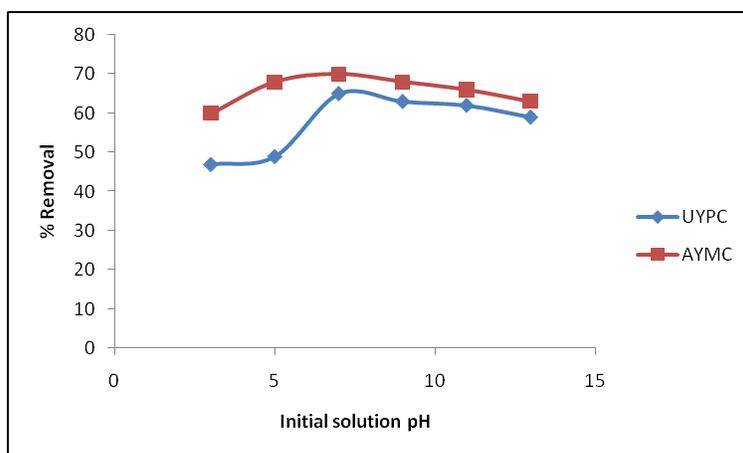


Fig.4. Effect of pH on crude oil sorption on the sorbents

An initial concentration of 5.0 g/L of the crude oil was prepared. The pH was varied from 3.0 to 13.0 at an interval of 2.0 while sorbent dosage (20 mg/mL) and contact time (60 min) were kept constant. An increase in the pH of the crude oil- water solution resulted in an increase in the percentage removal of crude oil for UYPC. As the initial pH increases, the percentage removal increased from 47 % at pH 3 to 65 % at pH 7 after which there was a

decrease in the percentage removal. This indicates that under acidic conditions, the carbon could remove oil till a neutral pH where maximum sorption capacity was achieved. In the case of AYPC, it was discovered that an increase in the pH of the crude oil- water solution also resulted in an increase in the percentage removal of crude oil. As the initial concentration increases, the percentage removal increased from 60 % at pH 3 to 70 % at pH 7 after which there was a decrease in the percentage removal. The higher the pH of an adsorbent, the higher the removal efficiency of organic materials by the adsorbent from wastewater. The surface of the adsorbent functional group is usually repulsively associated with hydroxonium ions (H₃O⁺) at low pH which invariably reduces the removal efficiency of organics during adsorption. Interestingly, it was observed that a higher percentage crude oil removal was achieved for AYPC than UYPC under all conditions of pH, contact time, sorbent dosage and concentration which indicates the effectiveness of zinc chloride modification.

3.8. Equilibrium isotherm and kinetic modeling of sorption

In this study, Langmuir and Freundlich's models were used to describe the equilibrium between the crude oil adsorbed onto the carbon, and the adsorbate solutions (Table 4).

Table 4. Equilibrium isotherm, kinetic and Intraparticle diffusion constants for sorption

Isotherm Model	UYPC	AYPC
Langmuir		
q _m (g g ⁻¹)	0.0393	0.0605
K _L (L g ⁻¹)	0.2728	5.3184
R ²	0.6076	0.6698
Freundlich		
K _F (g g ⁻¹)	0.0100	0.0570
n	0.4202	-12.94
R ²	0.7569	0.0152
Pseudo first order		
K _i (min ⁻¹)	0.0269	0.0131
q _e (g g ⁻¹)	0.0798	0.0378
R ²	0.246	0.022
Pseudo second order		
K ₂ (g g ⁻¹ min ⁻¹)	0.3391	1.1654
q _e (g g ⁻¹)	0.03166	0.3282
R ²	0.9693	0.994
Intraparticle diffusion		
K _d (g g ⁻¹ min ^{-1/2})	0.0338	0.0327
C	0.0327	0.101
R ²	0.9126	0.6653

Equilibrium kinetic study on adsorption provides information on the capacity of the adsorbent. An adsorption isotherm is characterized by certain constant values, which express the surface properties and the affinity of the adsorbents and can also be used to compare the adsorptive capacities of the adsorbent for different pollutants. Equilibrium data can be analyzed using commonly known adsorption isotherms, which provide the basis for the design of adsorption systems. The Langmuir isotherm is valid for monolayer sorption onto a surface with a finite number of identical site, represented by the following equation:

$$q_e = \frac{q_m K_L C_e}{(1 + K_L C_e)} \tag{4}$$

Where, q_m and K_L are Langmuir parameters related to maximum capacity and free energy of adsorption, respectively; C_e is the equilibrium concentration in the aqueous solution and q_e is the equilibrium adsorption capacity of adsorbent. The linearized form of Langmuir equation can be expressed as [18]:

$$C_e/q_e = 1/q_L K_L + C_e/q_L \tag{5}$$

The Langmuir constants q_L and K_L can be evaluated by plotting C_e/q_e versus C_e [18]. The low values of the linear regression coefficient obtained showed this model was not appropriate in the description of the process.

The Freundlich isotherm is an empirical equation based on sorption on heterogeneous surface energy systems given as:

$$q_e = K_f C_e^{1/n} \quad (6)$$

where K_f and n are the Freundlich constants that indicate adsorption capacity and adsorption intensity. The linear form of Freundlich isotherm can be written as:

$$\log q_e = \log K_f + 1/n \log C_e \quad (7)$$

The value of K_f and n can be calculated by plotting $\log q_e$ versus $\log C_e$. The best isotherm model in describing the sorption process must have the determination coefficient (R^2) closer to 1. According to these criteria on the R^2 results, it was observed that Langmuir isotherm showed a good fit to the experimental data with the highest R^2 for AYPC and Freundlich isotherm was the best fit for the UYPC. In addition, the Langmuir model for AYPC indicates the formation of monolayer coverage of oil onto the homogeneous sorbent surface [19].

The pseudo first order and pseudo second order kinetic models were also applied, and the equation and model constants have been discussed extensively in a previous report [18]. The model constants are given in Table 4. The pseudo second order model was shown to give clearly a better fit than the pseudo first order model as R^2 values obtained were close to one. This pseudo second order model assumes a chemisorptions mechanism between the sorbents and crude oil removal process.

3.9. Intraparticle diffusion model analysis

It is generally known that the sorption process is the rate- controlled process in which the slowest step determines the process rate limiting step. The kinetic data were further analyzed assuming that the mechanism of oil sorption can generally be described by four consecutive rate controlling steps which are external mass transfer (transport from the bulk solution to the sorbent surface), film diffusion (diffusion across the liquid film from the sorbent surface), intraparticle diffusion (pore diffusion, surface diffusion or combination of both mechanisms) and surface interactions at active sites [20]. Most of the time, only film and intraparticle diffusion are considered as the rate limiting steps as the process of external mass transfer, and chemical surface interaction are generally rapid. The rate limiting step of the sorption can be qualitatively determined by analyzing kinetic data using the Weber-Morris model as given by:

$$qt = K_d t^{1/2} + C \quad (8)$$

where k_i is the diffusion coefficient ($\text{g/g/min}^{0.5}$), and C is a constant that gives an indication of the thickness of the boundary layer. The constants obtained are presented in Table 4. The sorption process is said to be intraparticle diffusion controlled if the straight line plot passes through the origin, while some boundary layer diffusion (external mass transfer or film diffusion) may be present if it does not pass through the origin [20]. The plot for both UYPC and AYPC did not pass through the origin indicating some external mass transfer or film diffusion in the removal process.

4. Conclusion

The carbonized yam peel and zinc chloride activated yam peel carbon was found to be very efficient low cost materials for the remediation of oil spill polluted waters. The activated of the yam peel carbon by zinc chloride was very effective in improving the removal of crude oil from aqueous solution.

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