Studies on the Properties of an Oil Sorbent Material Produced from *Hyphaene thebaica*

Dimas, B. J*[1], Osemeahon, S. A[2]

[1]*Department of Science Education, Taraba State University, Jalingo, Nigeria*
[2]*Department of Chemistry Modibbo Adama University of Technology, Yola, Nigeria*
**Corresponding author*

Abstract: **This study investigated the use of Hyphaene thebaica as a sorbent for remediation of crude oil polluted water. The crude Hyphaene thebaica (CHT), retted Hyphaene thebaica (RHT) and bleached Hyphaene thebaica (PFHT) were subjected to sorption studies to optimize their sorption capacity. The results revealed that the efficiency of sorbent to remove crude oil from water is related to the sorbent weight, contact time, initial oil concentration and temperature of sorption. It was found out that increase in sorbent weight led to increase in sorption capacity from 3.71-4.87g/g, 4.30-5.77g/g, and 4.56-6.23g/g in CHT, RHT and PFHT respectively. Increased in Initial oil concentration also increased the oil sorption capacity by 18-24% until it reach equilibrium. Sorption time was varied from 10, 20, 30, 40, 50, 60 and70 minutes and the highest sorption capacity was recorded at 30 minutes before a gradual decreased was observed. Sorption capacity decreased with increased in temperature above 40^0C. The sorbent exhibited good reusability after 8 cycles, with less than 50 % reduction in sorption capacity. These properties introduce Hyphaene thebaica as a potential sorbent for oil cleanup.**

Keywords: **crude oil, sorption, sorbent, *Hyphaene thebaica*, remediation**.

I. INTRODUCTION

Spillage of oil can occur anytime there is exploration, transportation and storage of oil and its derivatives; this can cause significant environmental impact. An area can be contaminated once an oil spill. Cleaning of oil from surfaces becomes more tasking when oil comes in contacts with something to cling to (e.g., beach, rocks, feathers of a duck or a bathers' hair). Sea life, economy, tourism and leisure activities are all affected by pollution whenever there is oil spillage. Report has shown that about 10 million barrels of oil have been spilled both onshore and offshore in the Niger delta region of Nigeria [1, 2].

Water is a necessity to sustain life. The world approximated population of 7.6 billion is mounting pressure on the world's limited water resources [3]. While more water is use, the quality of water is reducing due to the large amounts of pollutants that are been released into the world water system on daily basis [4]. Development of environmentally friendly and cost-effective techniques to minimize petroleum-related pollution is greatly required. Oil absorbents are very promising materials to deal with oil spills, as they typically have high cleanup efficiencies. Oil sorbents can solidify and transform liquid oil into a semi-solid or solid phase, which can be removed from water and handled in properly without the oil draining out [5].

Management of oil spill can be achieved through sorption techniques. This method has been used effectively for cleaning of oil from the surfaces of water. Adsorption is a preferred oil removal technology because of its simplicity and lower processing cost in comparison to other oil removal technologies [6]. Experiment have been carried out using sugarcane bagasse [7], other agricultural waste such as rice straw [8], activated husk [9], Dialium guineense seed husk [10] and Codiaeum variegatum stem powder [11].

Hyphaene thebaica (Common Names: Egyptian doum palm, gingerbread palm) is a dioecious palm whose name is derived from the Greek word 'hyphaino' referring to the fibers from the leaves. The tree is found in countries such as Egypt, Senegal, Sudan, Central Africa, Nigeria, Tanzania and Mauritania. *Hyphaene thebaica* belongs to the family Palmae (Arecaceae) and subfamily Barassoideae. The leaves are regarded in many communities as the most important, being useful as fuel in the dried form and provide the raw material used in basketry, making mats, brooms, coarse textiles, ropes, thatching and string. The root fibers are also useful in making fishing nets [12, 13].

This work explores the use of *Hyphaene thebaica* fibers as sorbents in oil spill cleanup across various factors. The effect of variations in sorbent weight, contact time, initial oil concentration and temperature was examined. Water sorption capacity, sorbent reusability and oil retention were also investigated.

II. METHODOLOGY

Sorbent Preparation

The fibrous plant *Hyphaene thebaica* were obtained from around Girei Local Government Area, Adamawa State, Nigeria. A Botanist from Modibbo Adama University of Technology, Yola identified the plant sample. A knife was used to cut the plant part from the stem and subsequently the bark was removed and washed with distilled water. The fibrous plant was dried in the laboratory for one week.

Extraction of fiber Procedure

The fiber was extracted from the fibrous plant stem using chemical retting extraction process, giving fiber of different

lengths and diameters. The fibrous plant (Sample) was treated with 6% NaOH solution in accordance with work done by Cai *et al.* [14]. 15g of the sample was submerged in 6% NaOH solution and heated at 100^0C for 30 minutes in a water-bath. The fiber was rinsed in cold water to free fibers strands. It was neutralized with acetic acid and washed with distilled water repeatedly until all sodium hydroxide is eliminated. Finally, the fiber was dried at room temperature for 48H.

Bleaching of Fibers

Retted fibers were scoured in 2% NaOH solution at 100 ^{0}C for 30 minutes. Scouring of the fiber was carried out before bleaching. Dry scoured fibers were measured and submerged in a solution of 3% H_2O_2, with sodium pyrophosphate/sodium oxalate as buffering medium at 55 ^{0}C for 30 minutes to remove any colouring matter and white fibers was obtained.

Characterization of Crude Oil Sample

The properties of crude oil sample (COS) was characterized according to the method describe by Nwabueze *et al.,* [15]. The density, viscosity, specific gravity and API gravity of the crude oil sample were investigated.

1. Density

The density of COS sample was taken by using a specific gravity bottle. The bottle was filled with oil and weighed at room temperature (28 -30 ^{0}C) and the density calculated from:

$$Density = \frac{(MS - Mb)}{Vb} \text{----------Eq. 1}$$

where MS = mass of oil plus bottle

Mb = mass of bottle

Vb = volume of bottle

The method was repeated in triplicate to obtain a mean value.

2. Viscosity

The viscosity for crude oil sample was determined using viscometer. The viscometer was cleansed with a non-toxic solvent and dried. A certain amount of crude oil sample was poured into a beaker, and then transferred to the viscometer. The viscometer was inserted into the water bath at the required temperature. The pump was used to raise the level of the crude to the starting mark on the left hand limp of the viscometer; another finger was used to close the other limp to avoid the flow of the crude due to air. The finger was removed to allow the flow of crude down the capillary at that point, the time at which the crude flow down was taken and recorded.

3. Specific Gravity

The crude oil specific gravity was determined from the results obtained for density. When the density calculated was multiple with density of water 0.998 g/cm^3, it gives the specific gravity of the sample.

4. American Petroleum Institute (API) Gravity

The API gravity was calculated using the formula**:**

$$API = (141/s.g) - 131.5 \text{ ----------Eq.2}$$

where s.g = specific gravity of crude oil calculated.

Characterization of Crude Fibers

The physiochemical properties of the sorbents were investigated according to the method described by Donatus *et al.,* [16]. The moisture Content, Ash Content, Volatile Content, Fixed Carbon, Density, Specific Gravity and Swell ability were determined.

Determination of Water Sorption

The water sorbed by the sorbent was determined in the laboratory using the method of centrifuge technique described by Al Zubaidy *et al.,* [17]. Pressing was used to desorb the crude oil from the sorbent. Petroleum ether was added to help extract the oil in the sorbent during the pressing stage. Extracted liquid was collected in a centrifuge tube and placed in a water bath to break emulsion present and then, centrifuge for 20 minutes. The amount of water taken in by the sorbent was weighed and recorded.

Test for oil Sorption Capacity by Sorbent

Factors that affect oil adsorption were investigated, namely the effect of variation in sorbent weight, contact time, oil concentration and temperature in water/oil medium and in oil medium. Tests were carried out at room temperature. The methods describe by Onwuka *et al.,* [18] was adopted for the sorption studies. The crude oil sample was held in beakers for 1 day in open air to release volatile hydrocarbon contents. This was carried out to reduced variation during the sorption studies. The crude, retted and pure fibers were subjected to sorption studies to optimize the sorption properties.

10 g of crude oil was added to 100 ml of water in a 250-ml beaker. A portion 0.10g of the sorbent was added into the mixture in the beaker and left for 30 min. After the required time, the sorbent was removed using a spatula and placed on sieving net and left to drain by hanging the net over a beaker for 10 minutes. The drained sample was weighed and recorded. This was repeated at different weights of 0.2, 0.3, 0.4, 0.5, 0.6 and 0.7 g and results recorded. This experiment was also conducted at different times of 10, 20, 30, 40, 50, 60 and 70 minutes at constant sorbent weight/ oil concentration and results were recorded. The effect of Initial concentrations of crude oil was also studied from 5, 7.5, 10, 12.5, 15, 17.5 and 20 g /100 ml of water at constant sorbent weight, time and the results were recorded. The effect of temperature on sorption was also investigated at different temperature (30, 35, 40, 45, 50, 55 and 60 ^{0}C) at constant weight of sorbent and time. This was also repeated in oil medium. The sorption capacity of all the sorbent (CHT, RHT and PFHT) was calculated using the expression:

$$Sorption\ Capacity = \frac{New\ weight\ gain}{original\ \ weight} \ g/g \text{ ----------Eq.3}$$

The sorption capacity was recorded as g/g. This procedure was carried out in triplicates to obtain a mean value.

Sorbent Reusability

The sorbent sample was used eight times and after each time the sorbent was pressed to squeeze the oil content from the sorbent and ready for further use. The sorption performance was recorded in g/g. Reusability of the sorbent sample was studied in oil medium. 8 cycles of sorption processes was performed. After each cycle, the sorbent was squeezed and re-weighed. The difference between the weight of the wet material after drainage and the initial weight of the material gives its sorption ability.

Oil Retention

To determine the oil retention, a known weight of sorbent was placed in 20 ml of oil for 30 min. The sorbent was removed and vertically hung, where upon the adsorbed oil began to drip from the sorbent, the weight of the material was measured after 10, 20, 30, 40, 50, 60 and 70 min. of draining. The amount of oil retained was determined as the difference between the weight of the wet material after drainage and the initial weight of the material [19].

III. RESULT AND DISCUSSION

Table 1: Physicochemical Properties of Crude Oil Sample (COS)

Properties	Values – Mean and Standard Deviation
Density (g/cm^3)	0.8651± 0.01
Specific gravity (g/cm^3)	0.8634± 0.01
API^0 gravity (30^{o}C)	32.4 ± 0.02
Viscosity, 30^{o}C (cSt)	5.04 ± 0.02

Table 2: Physicochemical Properties of Sorbent

Properties	CHT	RHT	PFHT
Moisture Contents (%)	9.45 ± 0.01	11.38± 0.02	3.99± 0.02
Ash Content (%)	5.0± 0.01	0.3± 0.03	2.50 ± 0.02
Volatile Content (%)	46.58± 0.02	72.15± 0.01	47.76 ± 0.02
Fixed Carbon Content	38.96 ± 0.01	16.16± 0.01	45.75± 0.01
Density (g/cm^3)	0.9934±0.02	1.0045 ± 0.01	1.0041 ± 0.01
Swell ability (%)	215.54±0.02	132.5± 0.02	246.56± 0.02

Effect of sorbent weight

The effect of sorbent dose on the amount of crude oil removed was studied for the different sorbent weight ranging from 0.10, 0.20, 0.30, 0.40, 0.50, 0.60 and 0.70g. As shown in Figure 1, the sorption capacity efficiency was dependent on the weight of the sorbent in the entire sample studied. In CHT the sorption capacity increased from 3.81- 4.87g/g, RHT from 4.30-5.77 g/g and PFHT 4.56-6.23g/g. These indicate that increasing the amount of sorbent led to increase in oil sorption. The phenomenon here is associated with an increase in available active sites for sorption at higher sorbent dosage. Active sites are the site where a substrate binds and products are formed. The increase in active sites will results into higher percentage of oil removal, because of abundances of binding sites on the sorbent, thus leading to higher interaction between the oil particles and the sorbent [20]. The sorption capacity of the retted *Hyphaene thebaica* (RHT) and bleached *Hyphaene thebaica* (PFHT) were higher than the crude *Hyphaene thebaica* (CHT), this is as a results of alkalization, which disrupted the fiber s primary walls and exposed the microfibers causing increase in the number of reactive sites and allows better fiber wetting thereby improving the sorption capacity

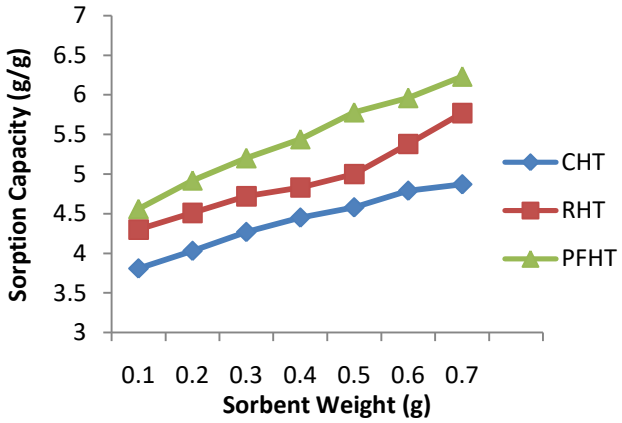

Figure 1: Effect of sorbent weight on oil sorption capacity.

Effect of contact time

Figure 2 shows the effect of contact time on sorption capacity. The effect of contact time in relation to sorption capacity of sorbents was studied at different time 10, 20, 30, 40, 50, 60, 70 minutes with other experimental variables kept constant. The sorption capacity increased gradually from 10-30 minutes before a steady decreased was observed from 40-70 minutes. The sorption process increased rapidly during the first 30 minutes. This might be due to the adsorption of crude oil on the surface of sorbent (fiber pores) before the oil begins to break though into the macroscopic voids. Then the values decrease regardless of soaking time [22, 23]. This can happen as a result of the formation of water in oil emulsion that cause increases in water pickup and decreases in oil sorption as contact time is increased.

It can also be explained to be as a result of saturation of sorbent surfaces with oil particles as well as the equilibrium between sorption and desorption process that occurred after saturation [24].

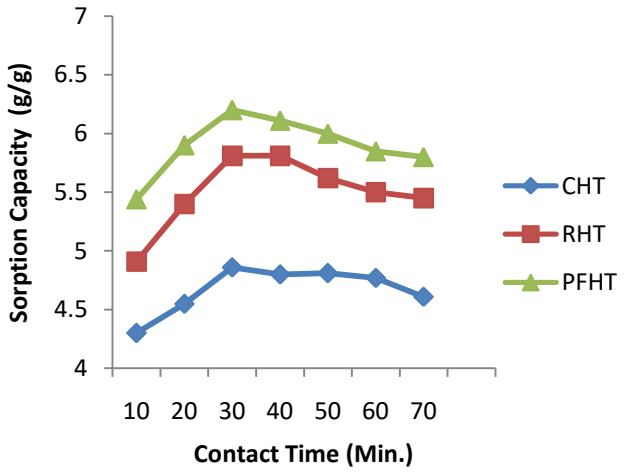

Figure 2: Effect of contact time on sorption capacity

Effect of initial oil concentration

The study of initial oil concentration is important as it strongly affect the kinetic sorption process. The effect of initial oil concentration and oil sorption capacity was studied by varying the oil concentration from 5, 7.5, 10, 12.5, 15, 17.5 and 20g. The experimental data from figure 3 revealed that increased in oil concentration enhanced oil sorption capacity before it reaches equilibrium. The sorption capacity was low at the beginning due to limitation of the oil molecules available for attachment on the sorbent surfaces [25]. The sorption capacity increased by18-24% with increased in oil concentration, because increasing the initial oil concentration provides an important driving force to overcome all resistance of oil between the aqueous and solid phases, which led to increases in the number of collisions between oil and the adsorbent, which enhances the adsorption process [26]. Then the sorption capacity became steady when it reaches equilibrium due to saturation of the binding sites [18].

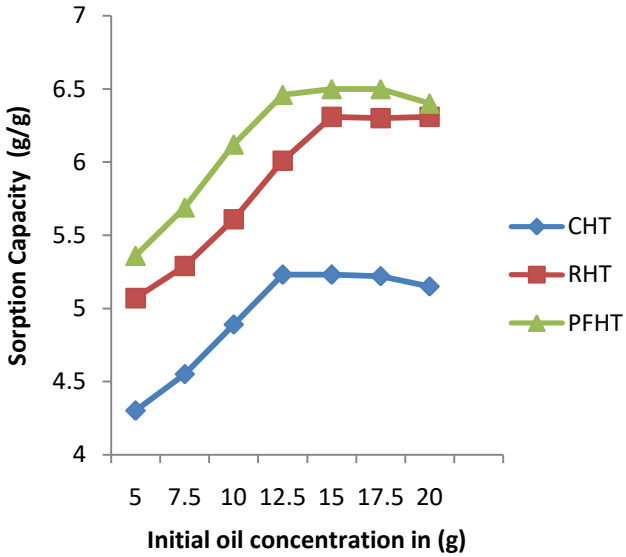

Figure 3: Effect of initial oil concentration

Effect of Temperature

The effect of variation in temperature on sorption capacity of the sorbents was studied, from 30, 35, 40, 45, 50, 55, and 60 ^{0}C. The effect of temperature on the sorption capacity of the sorbent as shown in figure 4 indicates that sorption capacity decreased by 35% for CHT, 31% for RHT and 26% for PFHT with increase in temperature indicating the dependence of adsorption on temperature. This decreased in sorption capacity is as a result of reduction in viscosity of oil. Viscosity depends on how fast or slow the fluids flows, as temperature increases the particles of liquid move faster and viscosity decreases making the oil light. As the oil becomes lighter, it is easily release from the particle. This finding is typical of sorption processes, whereby at higher kinetic energy of the oil at higher temperature, retention of oil becomes difficult resulting in decreased in sorption capacity [27]. It may also be due to decrease in adsorptive forces between the oil molecules and the active sites of solid phases as temperature increased.

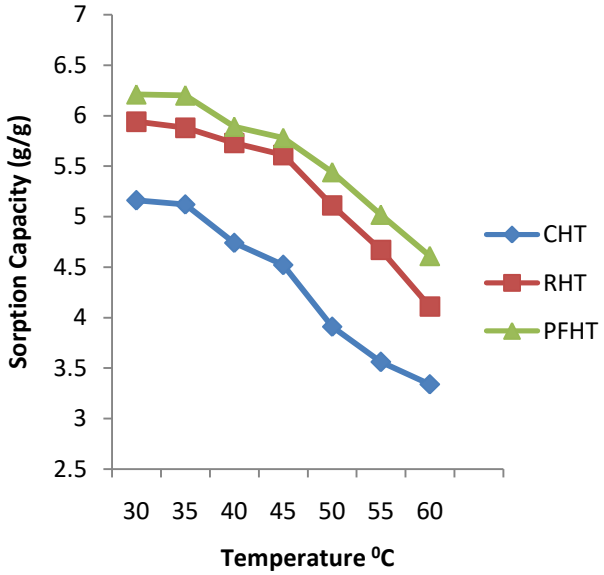

Figure 4: Effect of Temperature on oil sorption capacity

Water sorption capacity

Figure 5 showed the water sorption capacity of the sorbent in percentage at different sorbent weight (0.1, 0.2, 0.3.0.4.0.5, 0.6 and 0.7g). The water sorption capacity increased with increase in sorbent weight. While increasing the amount of sorbent, large amount of oil are removed narrowing the interface between oil and water leaving the sorbent to adsorb high value of water [28]. RHT has a lower value of water sorption of 147% at 0.7g of sorbent. This may be as a result of reduction in polar hydroxyl group in the fiber by replacement of OH group on the surface of the fibers by NaOH during retting [21].

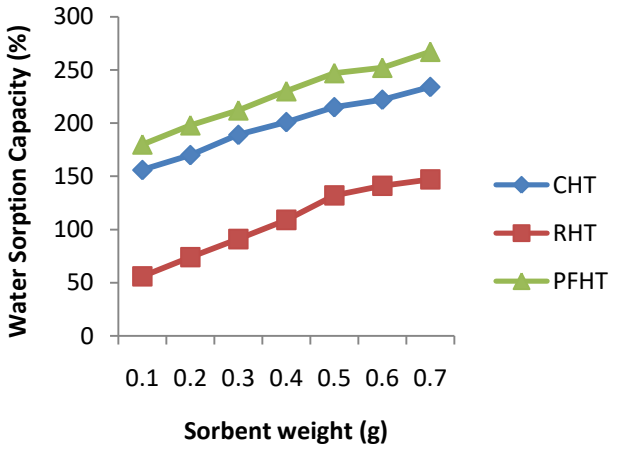

Figure 5: Water sorption capacity (%)

Effect of Reusability

Reusability is one of the major factors for selection of sorbent materials. The reusability of a sorbent usually depends on the ability of the oil fill sorbent to be regain its size and shape after the oil is removed and the sorbent is prepared for reused [29]. The relationship between the number of times the sorbent was reused and its sorption capacity are shown in figure 6. The sorption capacity of all the sorbent decreased with repetitive use with 32%, 24% and 25% for CHT, RHT and PFHT respectively. This decline in oil sorption capacity with increase in number of cycle can be due to incomplete desorption of oil molecules and this result in retention of some oil molecules on the sorbent surface reducing the number of active sites available for sorption [30]. The reduction in sorption capacity can also be as a result of deformation factor on the sorbent due to wearing and tearing during desorption after each used.

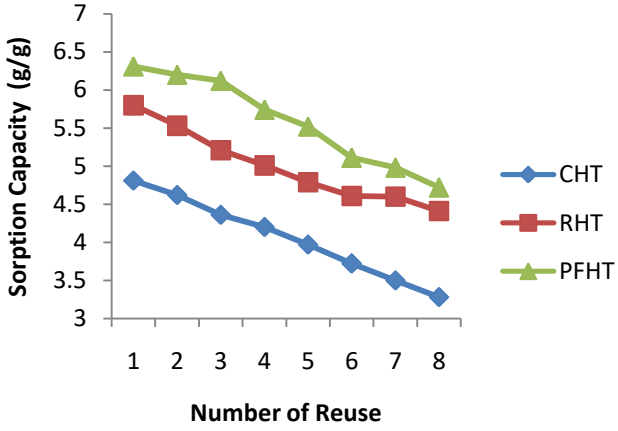

Figure 6: Effect of Sorbent Recyclability

Oil retention

The effect of oil retention with time 10, 20, 30, 40, 50 60, and 70 minutes for CHT, RHT and PFHT were studied. The quantities of adsorbed oil as remains in sorbent were shown in Figures 7. CHT, RHT and PFHT losses 48%, 33% and 32% respectively of the initial adsorb oil. This could be as a result of reduction of capillary retention pressure between the fiber assembly due to the filled oil in the lumen and interstices which was drain during the period under observation [31]. The draining of oil occurs first in the external surface of the sorbent assemblies and the surface of the reacting system before the liquid from the extra lumen drained out because of insufficiency of capillary pressure to hold the weight of the oils that are held inside the sorbent [32]. All the sorbent retained more than 50% of the sorbed oil after 70 minutes with PFHT having the highest retention value of 68%.

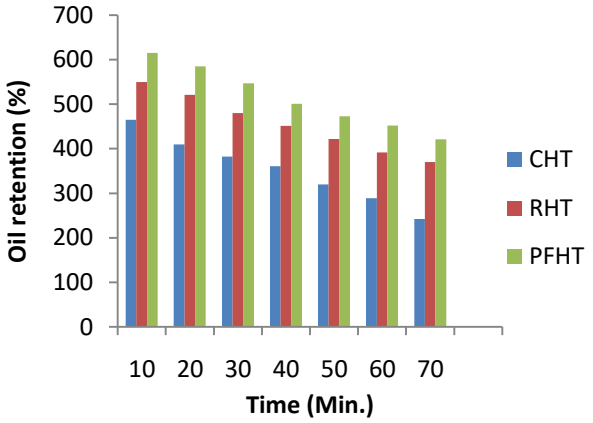

Figure 7: Oil Retention (%).

IV. CONCLUSION

This work presents a new material to separate crude oil and water by using *Hyphaene thebaica* fiber as natural sorbent. The sorption capacity of *Hyphaene thebaica* was investigated. It was found out that variation in sorbent weight, contact time, initial oil concentration and temperature all affect the sorption capacity of the sorbent. The experiments above showed that increased in sorbent weight and initial concentration led to better sorption capacity. Contact times of 30 minutes gives higher sorption capacity and temperature of less than 35^0C was chosen for the sorption studies. CHT, RHT and PFHT sorbent exhibited good reusability and oil retention. *Hyphaene thebaica* fibers with their desirable qualities have the potential to be an effective sorbent for oil-water separation.

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