



Study on the use of *Piliostigma reticulatum* as a sorbent for remediation of crude oil from water

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Abstract Oil activities over the years have negative impacts on the environments, hence effective and ecofriendly cleanup is required. This study investigated the use of *Piliostigma reticulatum* as a sorbent for remediation of crude oil polluted water. The crude *Piliostigma reticulatum* (CPR), retted *Piliostigma reticulatum* (RPR) and bleached *Piliostigma reticulatum* (PFPR) 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 that increase in sorbent weight led to increase in sorption capacity from 4.22 -5.20g/g, 5.20- 7.92g/g, and 4.70- 6.75g/g in CPR, RPR and PFPR respectively. Increased in Initial oil concentration also increased the oil sorption capacity by 25-32%. Sorption time was varied from 10-70 minutes and the highest sorption capacity was recorded at 30 minutes. The effect of temperature was investigated from 30-60⁰C and increased in temperature causes decreased in sorption capacity with about 28-31%. The sorbent exhibited good reusability after 8 cycles, with less than 50 % reduction in sorption capacity and RPR showed better retention capacity when compared with the other sorbent. This study will place *Piliostigma reticulatum* as a promising sorbent for crude oil remediation.

Keywords crude oil, sorption, sorbent, *Piliostigma reticulatum*, remediation

Introduction

Development in oil production and transportation has led to increase in oil spill accident which has generated not only environmental problems but lost in energy resources. Contaminated water cannot be used for municipal water supply, irrigation or industry because oil endangers all organisms in the water and effective decontamination and clean-up is required after a spill for the protection of the environment and human health [1, 2].

Ukpala [3] reported that oil spill is a common event in Nigeria and it occurs as a results of corrosion of pipeline and tankers (accounting for 50% of spills) sabotage (28%), oil production operation (21%) inadequate and nonfunctional production materials (11%). United nation reported in 2017 that the world estimated population of 7.6 billion people are placing more pressure on the world's limited water resources, while more water is being consumed on a daily basis, the quality of water keeps declining due to the significant amount of pollutants being discarded into our water systems [4]. Oil spilled in water or land changes the physical and chemical property of the environment, because it makes the water to have undesirable taste and odour causing severe environment damage.

Recently oil spills in the Niger delta region of Nigeria have risen to serious dimensions due to pipeline vandalization and other activities in the oil industry. Most water bodies in this region are contaminated with different hydrogen carbon fraction. Oil industry spills incident rate has increase to 80% between 1976 and 2008



[5]. In Nigeria oil spills have resulted to loss of many lives, destruction of farmlands, fishing activities, tourist's sites and cultural areas. The greatest issue is even that of long term effect of both acute and chronic contamination that can cause diseases like cancer and also rashes in children [6].

Crude oil be removed from water using physical or chemical methods, treatment is usually expensive and difficult to maintain [7, 8]. Hence adsorption is seen as an attractive treatment method for oily wastewater because it is environmentally friendly and has high removal efficiency. But there is need to sort for a sorbent that is effective and economical [9].

Natural and synthetic sorbent are used for oil removal from water. Several sorbents have reported as been used for remediation of oil water [10, 11, 12]. There is great interest in plant fiber due to their classification as renewable sources, combined with low cost availability, low density, non-toxicity and recyclability. Bast of vegetable plant are now being studied as possible replacement for some synthetic fiber [13]. Sorption techniques are accepted due to its simplicity and relatively lower processing cost when they are compared to other oil removal techniques.

The plant *Piliostigma reticulatum* (DC) Hochst is an evergreen shrub or small tree with a twisted bole widely distributed in Northern, Western and Eastern Africa. It is reported to possess medicinal properties similar to *Piliostigma thonningii*. A fiber obtained from the bark is used to make clothes and rope. The bark is commonly used for tying, for instance in hut, fence and bridge building. Fibers extracted from the bark are widely used for making string, rope and cloth.

The dependence of Nigeria oil industry on foreign expert for spill management will not add value in development and transfer of technology hence there is a need to develop a cost effective method for oil cleanup which be based on cheap agro products found readily within the environment. There have been many major oil spills that have resulted in environmental damage despite cleanup effort in the last decade; therefore, more work is required to discover more available material for oil spills removal on the surface of water.

The study investigates the use of *Piliostigma reticulatum* as sorbent for remediation of crude oil in water. The effect of variations in sorbent weight, contact time, initial oil concentration and temperature were studied. Sorbent reusability and oil retention were also investigated.

Methodology

Sample Collection and sorbent Preparation

The fibrous plant *Piliostigma reticulatum* was collected from a farmland located in Girei Local Government Area, Adamawa State, Nigeria and identified by a Botanist from ModibboAdama University of Technology, Yola. The plant part obtained was cut from the stem with a knife, the bark removed and washed with distilled water. It was spread on a clean polyethene and allowed to dry 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 100⁰C 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 100⁰C for 30 minutes. Scouring of the fiber was carried out before bleaching. Dry scoured fibers were measured and submerged in a solution of 3% H₂O₂, with sodium pyrophosphate/sodium oxalate as buffering medium at 55⁰C 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 was 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 °C) and the density calculated from:

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

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 and the viscosity was recorded in poise and converted to centistokes. This was carried out in triplicate to obtain a mean value.

3. Specific Gravity

The specific gravity of crude oil was determined from the results obtained for density. The specific gravity, being a more standard measurement was obtained by multiplying the density calculated with density of water 0.998 g/cm³.

4. American Petroleum Institute (API) Gravity

The API gravity was calculated using the formula:

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

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

Characterization of Crude Fibers

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

Determination of the Amount of Water Sorption

The water content of the sorbent was determined in the laboratory using the method of centrifuge technique described by Al Zubaidy *et al* [17]. The sorbent was subjected to pressing to desorb the crude oil. During the pressing stage, petroleum ether was added to help extract the oil in the sorbent; the 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 sorbed 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* [6] was adopted for the sorption studies. To simulate the situation of oil spill and minimize experimental variation, the crude oil sample was held in beakers for 1 day in open air to release volatile hydrocarbon contents. The crude, retted and pure fibers were subjected to sorption studies to optimize the sorption properties.

To 100 ml of distilled water in a 250-ml beaker, 10 g of crude oil was being added. A portion 0.10g of the sorbent was added into the mixture in the beaker and left unperturbed for 30 min. After 30 min, 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 and time and results recorded. The effect of temperature on sorption was also investigated at different temperature (30, 35, 40, 45, 50, 55 and 60 °C) at constant weight of sorbent and time. This was also repeated in oil medium. The sorption capacity of the sorbent samples was calculated using the expression:

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

and recorded as gram per gram of sorbent. The procedure was carried out in triplicates and the mean of the results reported.

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 were 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 [18].

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°C)	32.4± 0.02
Viscosity, 30°C (cSt)	5.04 ± 0.02

Table 2: Physicochemical Properties of Sorbent

Properties	CPR	RPR	PFPR
Moisture Contents (%)	12.69 ± 0.01	6.46 ± 0.02	3.89 ± 0.02
Ash Content (%)	11 ± 0.01	7.01± 0.01	9.02 ± 0.03
Volatile Content (%)	11.40 ± 0.02	61.33± 0.01	45.77 ± 0.02
Fixed Carbon Content	64.91 ± 0.01	25.15± 0.01	41.32± 0.01
Density (g/cm ³)	1.0004±0.03	1.0056 ± 0.01	1.0047± 0.01
Swell ability (%)	156.04±0.02	200.56± 0.03	200.58± 0.02

Effect of Sorbent Weight

Figure 1 showed the effect of sorbent weight on the sorption capacity of CPR, RPR and PFPR. AN increased in sorption capacity was observed as sorbent weight increased. The sorption capacity increased from 4.22- 5.25g/g CPR, from 5.20-7.92 g/g RPR and 4.70-6.75g/g PFPR. These indicate that increasing the amount of sorbent led to increase in oil uptake. The phenomenon here is associated with an increase in available binding sites for sorption at higher sorbent dosage. At higher sorbent dosage, oil adsorb are higher due to availability of more empty binding sites as compared to lower dosage which has less binding sites to adsorb the same amount of oil in the adsorbate solution [19]. The trend revealed a progressive increase in the sorption capacity as the adsorbent mass increased from 0.1 to 0.7 g/g. Higher dosage of sorbent permanently binds the oil, which facilitates the



storage and transport of oil molecules [20]. RPR and PFPR exhibit higher sorption capacity due to more active sites as a result a result of changes in surface chemical functionality due to pretreatment which enhanced interaction with oil molecules. The hydroxyl binding force is weakened by pretreatment and this improves the diffusion velocity of the oil to the inside of the sorbent [21]. Sorption is a surface phenomenon that is directly related to surface area. Therefore, increasing the surface area increases the oil uptake. As the basal spacing of the pretreated sorbent sample (RPR and PFPR) expands, it increases the surface area of the materials and as the surface area increases, it increases the capillaries that are being formed and hence increases/favours absorption.

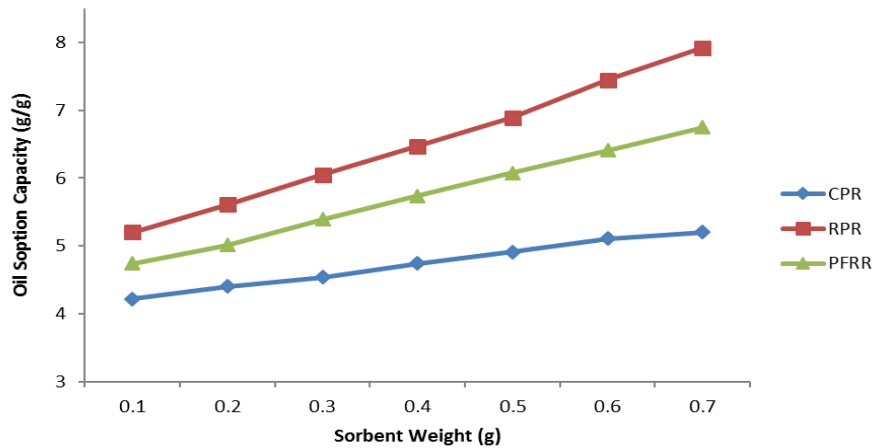


Figure 1: Effect of sorbent weight on oil sorption capacity

Effect of Contact Time

Sorption is a time dependent process and it is crucial to know the rate of sorption for designing and evaluating the sorbent performance in oil removal. Figure 2 shows the effect of contact time on sorption capacity. The oil sorption increases gradually as sorption time increases up to 30 minutes when the maximum value is reached. This can 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 [22].

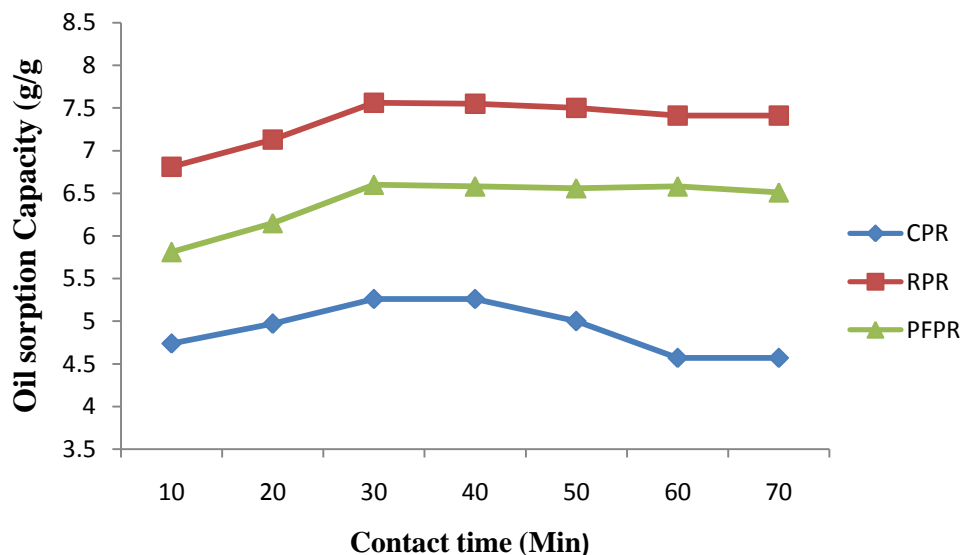


Figure 2: Effect of time of contact

This initially high rate of oil uptake maybe attributed to the presence of a large number of vacant voids accessible for the oil sorption on the sorbent. Increase in contact time provide enough time for molecules of oil to bind on the surface of sorbent sites. This might also be due to the adsorption of crude oil on the surface of sorbent before the oil begins to break though into the macroscopic voids. The first 30 minutes was governed by fast diffusion on the external surface, followed by fast pore diffusion into the intra particle matrix to attain rapid



equilibrium. Further increased showed gradual decrease in the oil removal until equilibrium is reached [23]. As the contact time was increased, less adsorption sites were available, hence oil uptake remained constant [24]. This may be due to the fact that the more time oil spends in an open area environment the less viscous it likely becomes hence leading to release of oil from the sorbent [15]. Then the values decrease regardless of soaking time. Similar findings were reported by Onwuka *et al* [6] and Thompson *et al* [25].

Effect of Initial Oil Concentration

Figure 3 showed the sorption capacity of the sorbent at different initial oil concentrations ranging from 5- 20g. An increase in oil concentration enhanced oil sorption capacity. The sorption capacity of all the sorbent increased with 25-32%. Increased in oil sorption capacity can be attributed to adsorption of crude oil molecules at the reactive sites and diffusion into pores or hollow structure of the sorbent as more oil is added, thus reducing water uptake. As initial oil concentration increases, the thickness of the oil layer on the water surfaces increases and the sorbent reach the water surfaces to less extent; therefore, lowering the amount of water sorption and enhance oil uptake. With increase in oil concentration, the oil molecules in the adsorption layer converge and form a saturated layer so adsorption increases. At high initial concentration, the gradient between the solution sample and the center of the particles enhance oil diffusion through the film surrounding the particles and in the porous network of the sorbent [26]. Then the sorption capacity became steady when it reaches equilibrium due to saturation of the binding sites [6]. Surface treatment also improves the affinity towards oil sorption that is why RPR and PFPR showed higher sorption capacity than CPR.

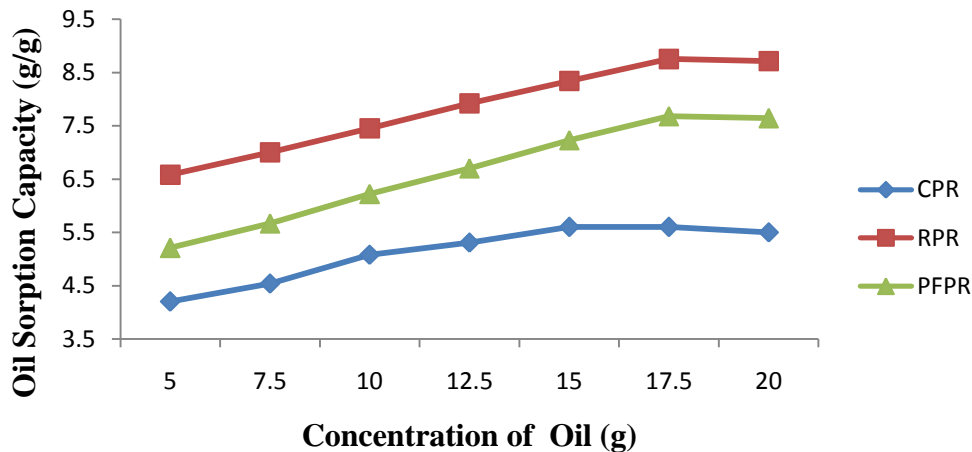


Figure 3: Effect of initial oil concentration

Effect of Temperature

The effect of temperature on the sorption capacity of the sorbent is shown in figure 4. The sorption capacity increased from 30-40°C. Other increased in temperature result in decreased in the sorption capacity by 31%, 28% and 28% in CPR, RPR and PFPR respectively. It is clear that adsorption decreases gradually with temperature increase at above 40°C. This result may be due to decreased in viscosity at high temperature making the oil it drains off the sorbents surface easily. Molecules of adsorbed material are in constant motion (Brownian motion) and when they are very close to the surface of sorbent they get attached and connected with the surface of sorbent. When temperature raises, the movement of molecules increase (motion is higher) and the interactions between sorbent and molecules are more intense. With higher temperature the possibility for molecules to attach to the sorbent surface is lower, hence lowering the ability of oil droplets to be attached on the sorbent surface, requiring more energy to stick to the surface of the sorbent material [27]. Initially the viscosity was good enough to penetrate the pores of the sorbent and halts within the surface roughness. This means that adsorption is directly proportional to the capillaries diameter and inversely proportional to the oil viscosity [28]. In general, oil sorption capacity increases with increasing temperature, until an upper threshold where the surface characteristics (e.g., particle size, porosity) of sorbent become limiting [29, 30]. At higher temperature the



efficiency of the sorbent to adsorb oil is decreased. This is expected due to fibers degradation at higher temperature since their structure and form may be damaged.

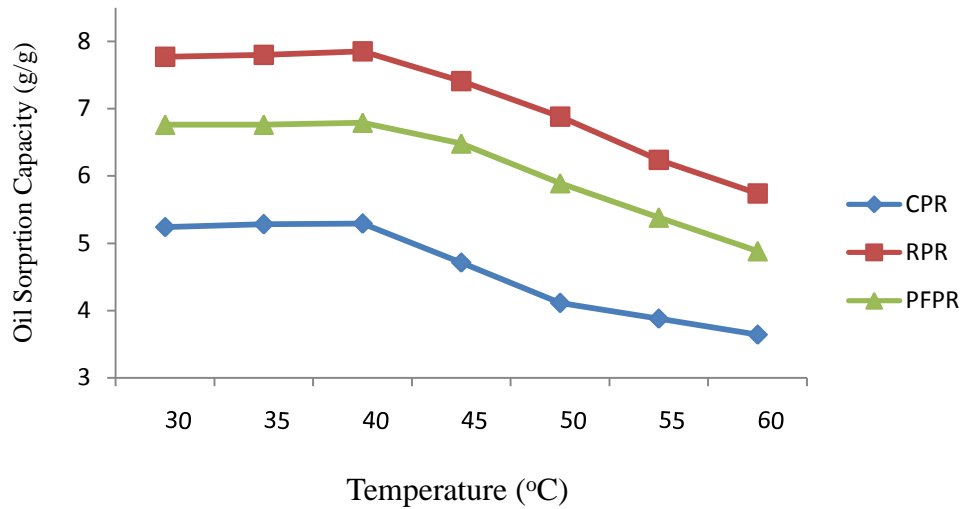


Figure 4: Effect of Temperature on oil sorption capacity

Water Sorption Capacity

Figure 5 showed the water sorption capacity of the sorbent in percentage at different sorbent weight. The water sorption capacity increased with increase in sorbent weight with RPR having a lower value of 154%. Water can penetrate cellulose network and move into the capillaries and spaces between the fibrils. Water molecules tend to force the cellulose molecules apart reducing the forces that hold them together. Reduction in water absorption result from pretreatment, which give rise to increase in its potentials to sorb more oil in an aqueous medium [1]. Water molecules attract the hydrophilic group of the sorbent and react with hydroxyl group (-OH) of the cellulose to form hydrogen bond [31]. Replacement of poly-hydroxyl groups with Na are likely the cause for the observed reduction in water sorption capacity values [32]. When chemicals are added to clean the sorbent, it improves adsorption capacity and stability of the bio sorbent making it more effective [4].

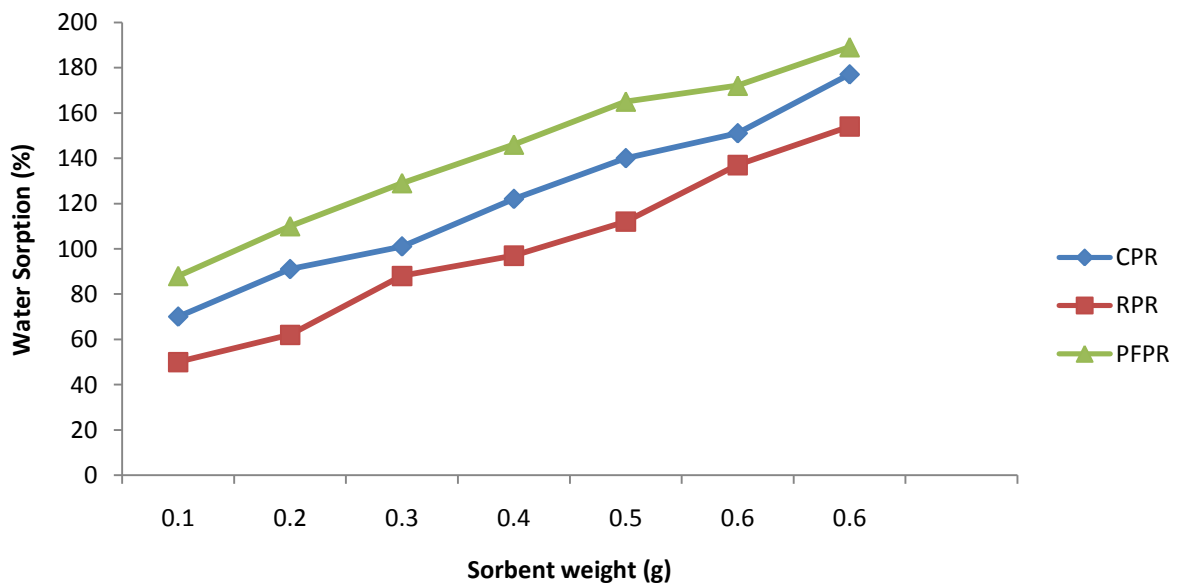


Figure 5: Water sorption capacity (%)

Effect of Reusability

Reusability is one of the major factors for selection of sorbent materials. Effect of initial oil concentration on oil sorption capacity is shown in figure 6. The amount of oil sorbed decreases with recycling effect but did not exceed 50% of the initial value after eight sorption cycles in oil without water. Perhaps it can be due to tearing of the micro fibrils, disruptive pores and traces of oil entrapment might have led to the decreased in amount of oil sorbed. This also supports, the findings that a sorbent is considered reusable if a loaded sorbent can easily compress or squeezed to its original size and shape even if there was tendency toward decrease in sorbent efficiency with repeated sorption and desorption. The decreased oil sorption capacity with number if reused can be considered to be as a consequence of the irreversible deformation of sorbent by pressing Particularly during continuous cycles [33, 34]. The void fraction may not be recovered even after desorption thus preventing more oil from entering the fiber assembly. Pretreated sample retained more oil than the raw sample because the sorbent surfaces is very effective to improve its reusability due to more stable liquid bridge between pretreated sorbent compared to the raw sorbent.

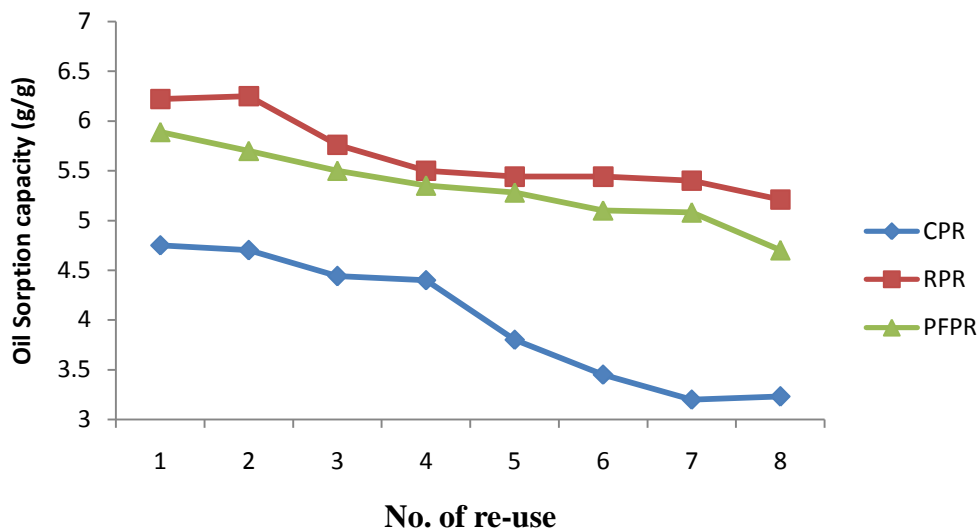


Figure 6: Sorbent Recyclability

Oil retention

The oil retention with time for CPR, RPR and PFPR was studied. The quantities of adsorbed oil as remains in sorbent were shown in Figures 7.

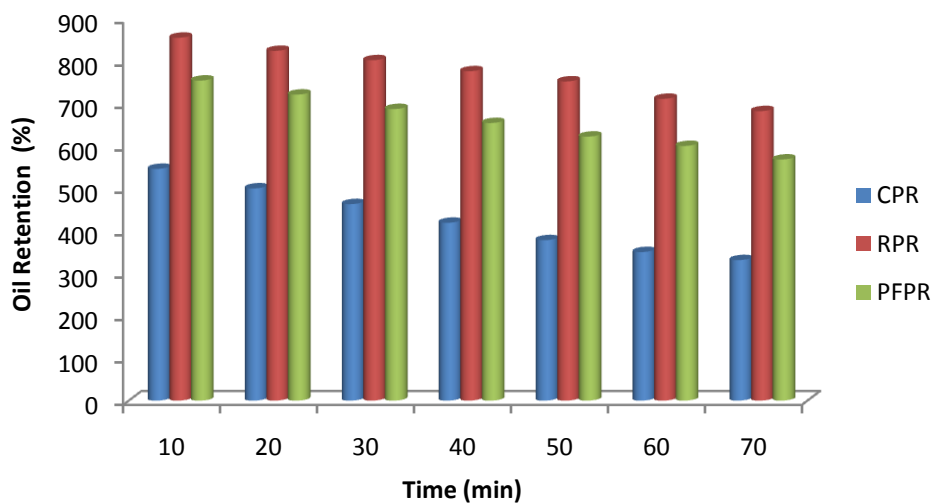


Figure 7: Oil Retention (%).

The ability of sorbent to retained oil decreases with time as seen above. Decreases in oil retention capacity may be because of escape of excess oil which had adhered superficially to the surface fiber bundles [35]. The oil contained in the fiber lumen and the inter-fiber interstices drained out slowly as the capillary pressure and Vander forces were insufficient to hold the weight of the oil [36]. The sorbent all showed good retention with RPR having the highest retention value of 79%. Improvement of fiber surface roughness can also lead to better locking-oil capacity which will give rise to good adhesion of oil in the interstice of fiber assembly of the pretreated samples compared to the smooth surface of the raw fiber [37].

Conclusion

The sorption capacity of *Piliostigma reticulatum* 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 time of 30 minutes give higher sorption capacity and temperature of less than 40⁰C as better for this sorption. This study will present a novel method for crude oil remediation in water.

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