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CRUDE OIL SORPTION CAPACITY OF MODIFIED AND UNMODIFIED BIOWASTE

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ABSTRACT

Contamination of water by crude oil is ubiquitous particularly in developing countries. The governments of affected countries raise a lot of concern because of the associated economic and environmental impacts which are not in tandem with the United Nations Sustainable Development Goals (SDGs), 6, 8, 11, 13 & 14. Remediation of crude oil-impacted water and soil become imperative. However, the exercise is expensive; hence the search for cheaper and local alternatives. In this study, unsegregated wood sawdust was collected from the various wood sawdust dump sites within the main Timber shade in the vicinity of Udu Bridge, Udu LGA of Delta State, Nigeria. One half of the sample was chemically modified by acetylation using acetic anhydride while the other half wasn't. They were characterized using Fourier Transformed Infra-Red Spectrophotometer (FTIR) and Scanning Electron Microscope coupled with Electron Dispersive X-ray (SEM/EDX) analyzer before employing them in the removal of the crude oil from the simulated crude oil spill- water media. The obtained results indicated that the modified sorbent (wood sawdust) sorbed more crude oil than the unmodified sorbent (wood sawdust). Experimental process control factors limited to time, sorbent dosage, particle size, was investigated using the modified sawdust. The modified sorbent gave maximum oil absorption capacity of 5.228 gg^{-1} for sorption time, 4.759 gg^{-1} for sorbent dosage and 5.838 gg^{-1} for particle size analyses. The study contributes to the knowledge of sorption studies using unsegregated wood sawdust for remediation of crude oil-contaminated media.

Keywords: sorption, chemical modification, crude oil, wood sawdust, FTIR.

INTRODUCTION

Oil contaminants are some of the major sources of pollution in water bodies because of their threat to aquatic life and man (Al-Majed et al. 2012, Oliveira et al. 2014, Sammarco et al. 2014). Oil pollution may occur as a consequence of accidental discharge, intentional (such as vandalization of pipeline) and uncontrolled anthropogenic sources such as corrosion of oil pipes, over pressure failure and over flow process components; poor maintenance of infrastructure, spills or leaks during processing at refineries, tankers accidents, human error, sabotage and oil theft activities

(Kinako and Awi- Waadu, 2014). Most oils float on water, and because of this, organisms that live in the water are harmed (Barron, 2012). Other organisms and land animals are also affected if the oil comes to the shore (Brody et al. 2012). These oil contaminants affect marine species negatively; and lead to their high death rate and physical smothering because of their exposure to the toxic effects of the oil (Dicks, 1998). The oil on the fur and feathers destroys the insulation value of the wild-life and then causes them death by hypothermia. More so, these spilled oils also affect humans through inhalation, skin and eye irritation. These environmental damages can be avoided if the spilled oil is promptly and effectively removed from the water.

Numerous techniques have been put in place to tackle pollution caused by crude oil. These measures include the use of adsorbing materials (Zadaka- Amir, 2012, Sayed and Zayed, 2006), in-situ burning (Erit – Rasmussen and Brandvik, 2011), dispersing agents (Inouye, 2005) enhanced bioremediation (Ron and Roseberg, 2014), use of skimmers, oil booms, porous materials, oil sorption resins, etc. However, these measures inevitably have some drawbacks, such as high cost, non-biodegradation, low separation efficiency, secondary pollution and co-absorption of water, etc (Zang et al. 2016). Most dispersants can lead to secondary pollution which can affect fish, fowl, and various mammals, while skimmers and oil booms are not effective for removing trace oil from the oil-water mixture (Wang et al. 2013). Therefore, there is a need for the application of simple, biodegradable, non-toxic, cost-effective and environmentally friendly sorbents from organic materials to clean up pollution caused by oil spills. At a trace or light thickness of oil on water, sorbents are the best agent to clean the oil from water as skimmers will suck up oil and water. Indeed, sorbents can pull together liquids and transform them into a semisolid or solid state. Hence, when a sorbent is employed to tackle pollution caused by oil spill, no secondary pollution occurs (Jmaa and Kallel, 2019). The process of the cleanup is also simpler and cost-effective (Yati et al. 2016). Thus, biodegradable natural materials with hydrophobic property and high separating capacity are needed.

Furthermore, the increasing population rate has led to a large amount of bio-wastes produced globally. This easily biodegradable bio-wastes or biomass can be used as sorbents to tackle oil spills, instead of using hazardous chemicals like dispersants (Doshi et al. 2018). The technique of cleaning up water containing oil with organic natural materials is economical because of the low cost of natural renewable materials.

When organic materials undergo effective modification, their ability in tackling pollution caused by the oil spill can improve. Local sorbents gotten from these bio-wastes are mostly cheap and eco-friendly because of their availability and biological compositions.

Hydrophobicity is one of the most important properties of oil sorbents. Sorbents with high hydrophobicity tend to be more efficient for sorbing oil than sorbents with low hydrophobicity (Fang et al. 2015; Wang et al. 2015 and Yang et al. 2015b). The sorbent's hydrophobicity improves the sorption capacity of oil and prevents the uptake of water.

Some workers (Schanninger et al. 2011) investigated the chemical modification of Spruce wood of various sample shapes, by applying acetic anhydride with and without catalysts. The latter showed enhanced absorption

Another worker (Nwufo et al. 2014) investigated the use of an untreated, raw and mixed variety of hardwood and softwood sawdust in oil pollution remediation. Adsorptive capacities of raw hard and softwood sawdust were studied and compared. Softwood sawdust was seen to have better oil adsorptive capacities. The adsorptive capacities are related to the mesh size of the sawdust, the oil concentration, the quantity of the sawdust, and the contact time

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between the oil and the sawdust. Maximum uptake of the oil by both adsorbents occurred at 120 and 150 minutes for all the determinations.

Other researchers (Jonoobi et al. 2010) and (Sun et al. 2002) have investigated the crude oil absorption capacity of sawdust of different wood species by different treatments with different results.

However, the unsegregated sawdust of different woods as heaped in most dumpsites in Nigeria has not been investigated. Hence, this work investigated the sorption capacity of such unsegregated mounds of sawdust which are usually heaped at the banks of nearby rivers with its consequent ecotoxicological threat to the littoral and neritic environments of the rivers as earlier investigated by Ibe and Ogeleka, 2019.

MATERIALS AND METHODS

Collection and Preparation of Samples

Sawdust was collected from some dumpsites within the main Timber shade in the vicinity of Udu Bridge, Udu Local Government Area, Delta State, Nigeria.

Fig. 1: Udu Sawdust Dunes

They were sorted to remove debris and unwanted particles, sun-dried to remove moisture, then sieved to get uniform particles of various sizes. Thereafter, the sawdust of various sizes was oven-dried at 100^0C for about 3 hours. Portions of the samples were taken out for chemical modification (acetylation using acetic anhydride).

Acetylation Concept

Acetylation is a method of treating natural fibers, making them more hydrophobic. The principle of acetylation is to substitute the fiber's hydroxyl groups (OH), which are responsible for their hydrophilic behaviour, with acetyl groups (O-CO-CH_3) that have a more hydrophobic nature (Lepetit et al., 2017) through reaction with acetic anhydride.

Modification by Acetylation

5g of sawdust was added in a flask containing 100mL of acetic anhydride, heated at 120°C under reflux for about 90 minutes. The product was filtered and washed with acetone to remove unreacted reagents, and then dried in an oven at 60°C to a constant weight.

Crude oil was obtained from an oil field in Warri, Delta State.

Characterization of the Modified Sawdust

Weight percent gain (WPG)

WPG is the difference between the oven-dried weight of the sample before and after the acetylation process. This was calculated using equation (1)

$$WPG\ (\%) = \frac{m_2 - m_1}{m_1} \times \frac{100}{1} \quad \text{.....(1)}$$

where m_1 and m_2 are the masses of unmodified and modified fiber

Morphology analyses

The morphology of modified and unmodified sawdust was investigated using a Scanning electron microscope coupled with Energy

Dispersive X-ray analysis (SEM/EDX). It was recorded using JOEL Scanning Electron Microscope, JSM-7600F. SEM produced images from the sample by scanning the surface with a focused beam of electrons.

Functional group analysis

The Fourier transform infrared analysis (FTIR) spectra were recorded using FTIR bulk scientific m530 USA equipment. Infra-red spectroscopy was performed on the modified and unmodified sawdust to investigate the functional groups present in both of them.

Properties of the crude oil used

Some properties of crude oil used for the sorption process are presented in Table 1.

Table 1: Crude oil properties

Crude oil	
Density @ 26^0C	0.8344g/mL
Specific gravity @ 15^0C	0.86
API gravity	32.6
Kinematic Viscosity @ 26^0C	5.857 mm^2/s

Experimental Procedure

Batch sorption experiments were carried out using the unacetylated (unmodified) and acetylated (modified) sawdust (sorbent) to determine sorption capacity. The effect of varying parameters like particle size, contact time, and sorbent dosage were also investigated. The sorbent was neatly enclosed in a permeable material, giving it a pillow-like shape. The enclosed material containing 2g of the sawdust was dipped in a beaker containing 15mL of crude oil and 150mL of water at room temperature without shaking. The initial weight of the sorbent material was taken before it was deployed in the oil - water mixture. On completion of the sorption experiment, the soaked sorbent was removed and allowed to drain for a specified time and weighed.

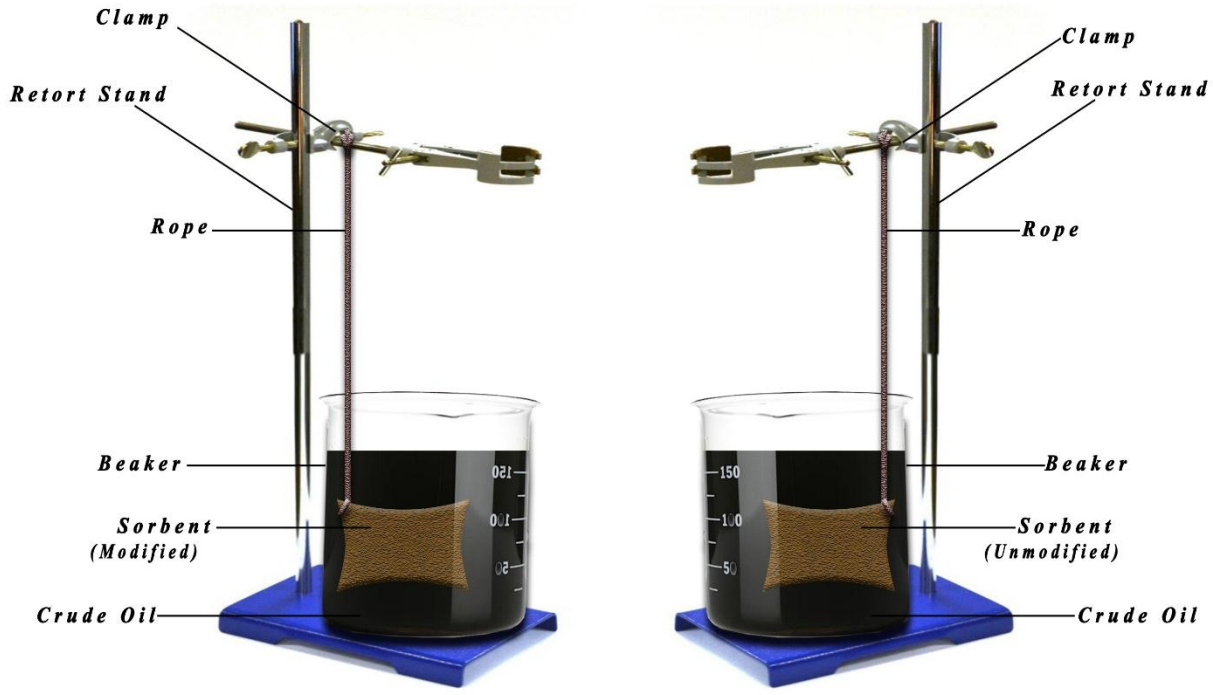

Fig. 2: Sorption Set- Up

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The oil sorption capacity (OSC) was calculated using the expression (Zang et al. 2015).

$$OSC = \frac{X_a - X_b}{X_b} \quad \ldots\ldots (2)$$

where X_a is the total weight of the sorbent after sorption while X_b is the total weight of the sorbent before sorption.

Owing to the relatively higher oil sorption capacities of the acetylated (modified) sawdust, further process control factors limited to time, sorbent dosage and particle size were investigated using modified sawdust.

Variation of sorption capacity with contact time

The sorption of crude oil using acetylated (modified) sawdust (sorbent) was investigated at various time intervals: 5, 10, 15, and 20 minutes. 2g of the modified sawdust was used. The mass of the enclosed sorbent was taken, and then dipped in a beaker containing 15mL of crude oil and 150mL of water at room temperature without shaking. On completion of each contact time, the sorbent was removed and allowed to drain for 10 minutes and re-weighed. The oil sorption capacity is then calculated using Equation 2.

Variation of sorption capacity with sorbent mass

The sorption of crude oil using acetylated (modified) sawdust (sorbent) was studied using various amounts of sorbent. 1g, 2g, 3g, and 4g of the modified sawdust each was dipped in a beaker containing 15mL of crude oil and 150mL of water at room temperature without shaking. After 10 minutes, the sorbent was removed and allowed to drain for 10 minutes. The weights of the enclosed sorbents before and after sorption were taken. The sorption capacity then calculated.

Variation of sorption capacity with particle size

The oil sorption capacity of different particle sizes of the sorbent was investigated using the following particle sizes – 1.0mm, 0.6mm, 0.425mm, and 0.106mm. 2g of the sorbent, enclosed in a permeable material was dipped in a beaker containing 15mL of crude oil and 150mL of water at room temperature without shaking. After 10 minutes, the sorbent was removed and allowed to drain for 10 minutes. The weights of the enclosed sorbents before and after sorption were taken. The oil sorption capacity was calculated using Equation 2.

RESULTS AND DISCUSSION

Characterization of the sorbent

Weight percent gain (WPG)

Table 2: Mean weight percentage gain of modified and unmodified sorbent

	Sample1	Sample 2	Sample 3	Sample4	Sample5
Wt. before Modification(g)	5.0000	5.0000	5.0000	5.0000	5.0000
Wt. after Modification(g)	5.1507	5.1509	5.1509	5.1507	5.1513
Wt. Gain(g)	0.1507	0.1509	0.1509	0.1507	0.1513
Mean Wt. Gain (WG) (g)			0.1509		
Weight % Gain (WPG)			3.018		

The weights of the unmodified and modified sorbents, the weight gain, mean weight gain and hence the percentage weight gain(WPG) are as shown on the table. Application of Equation 1 gave the calculated value of WPG as 3.018%. This shows that the weight of the sorbent increased after acetylation because of the incorporation of the acetyl group (O-CO-CH_3) in place of hydroxyl (OH) which has a lower mass.

Fourier transform infrared

The FTIR spectra of modified and unmodified sorbent are shown in Figures 3 and 4 below. The chemical modification occurred as seen by the presence of the following bands: 1381 cm^{-1} showing the C - H bond bending in -O(C=O)-CH_3 and 1260 cm^{-1} showing the C-O stretching vibration of the acetyl group (Olaru et al. 2011). More so, the decrease of the cellulose OH band at 3439 cm^{-1} also shows that a chemical modification occurred on the sorbent.

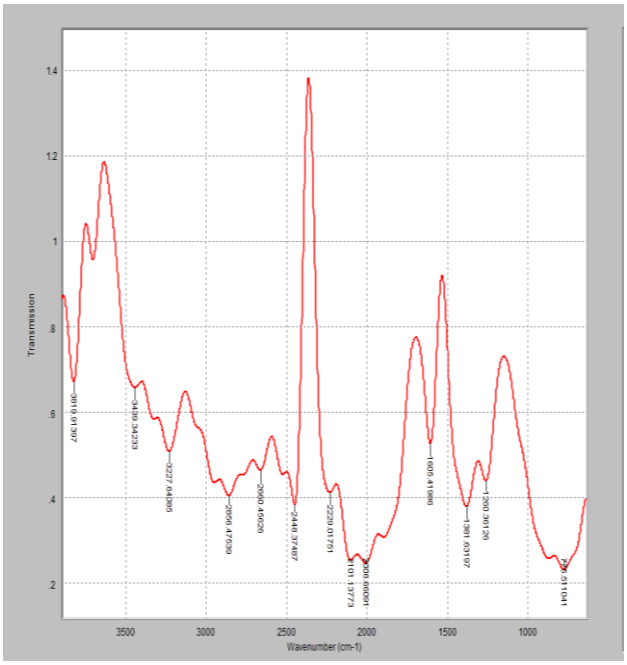

Fig. 3: FTIR Spectra of modified sawdust

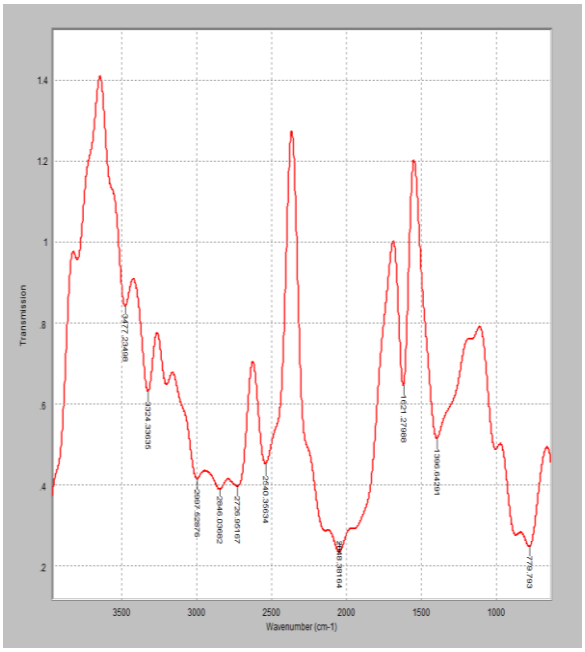

Fig. 4: FTIR spectra of unmodified sawdust

Scan electron micrograph

The micrographs and compositions (deduced from SEM & EDX) of the modified and unmodified wood sawdust are shown in Figures 5 to 8. The scanning electron micrograph (SEM) of the modified wood sawdust seems to present a better morphological appearance than that of the unmodified wood sawdust. The EDX images of the modified and unmodified wood sawdust reveal similar elemental composition in different proportion.

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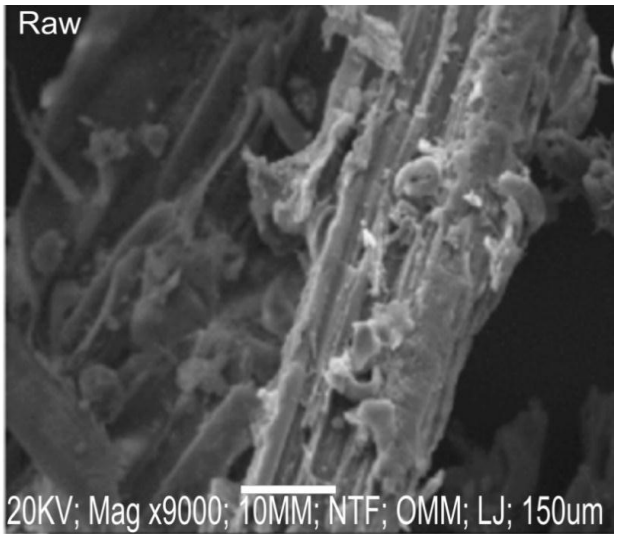

Fig.5: SEM of unmodified sawdust

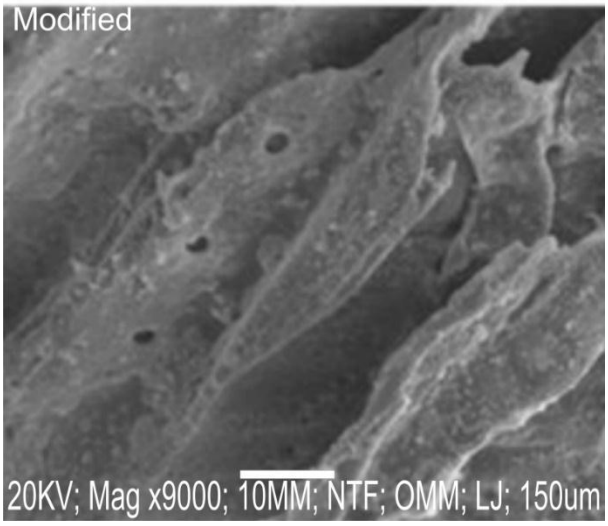

Fig 6: SEM of modified sawdust

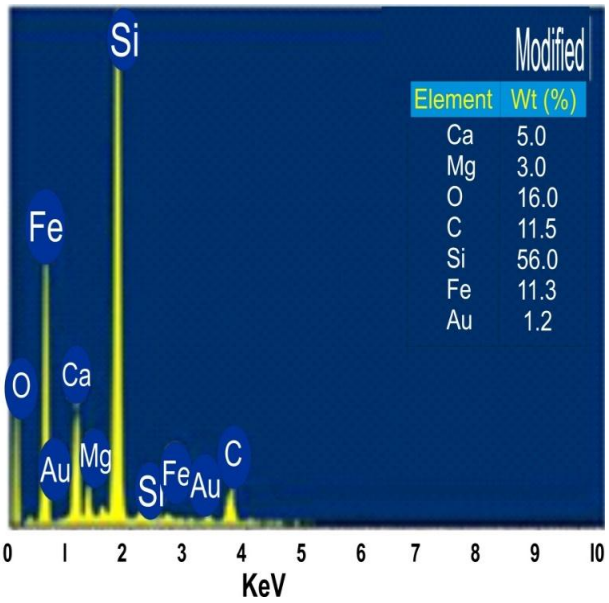

Fig.7: EDX micrograph of modified sawdust

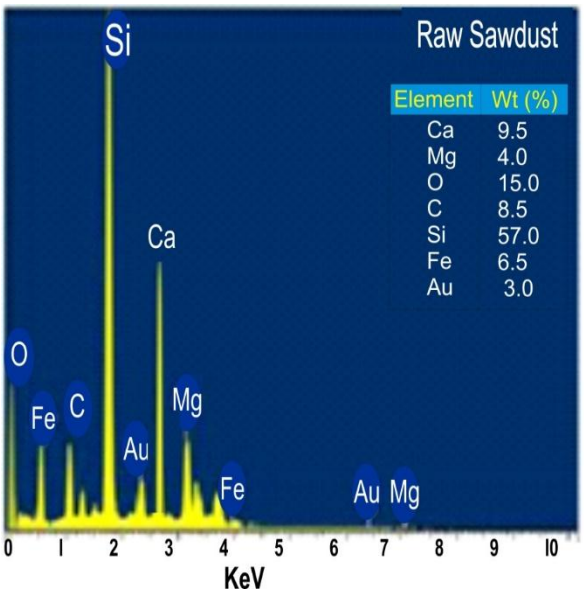

Fig. 8: EDX micrograph of unmodified sawdust

Calcium in the unmodified sample (9.5%) was reduced to 5.0% in the modified samples. Reduction in Mg (from 4 to 3%), Si (57 to 56%) and Au (from 3 to 1.2%) were also observed as a result of the acetylation. However, oxygen and carbon contents were observed to increase after acetylation. It is obvious that in the two samples, silicon is the major element. The Au peak seen may be attributed to the sample cover for the EDX scanning (Melena and Lazaro, 2011). In the case of the modified saw dust sample, the amount of Au was reduced which showed that the modification could have obscured the Au of the sample cover, the source of the Au. The modification of the saw dust with acetic anhydride increased the O and C content which may further be improved by the extent of the modification.

Singh *et al.*(2011) have reported the elemental constituents of their raw saw dust as C 45.52%, O30.65%, Ca 0.61%, Cu 2.39%, Zn 1.8%,Au 19.04%, S 0% and the sulphuric acid modified saw dust as C

46.98%, O 34.06%,Ca 0.5%, Cu 1.36%, Zn 1.34%,Au 15.24%, S 0.52%. it can be seen that when their results are compared with the present study, that the constituent elements differ. C was seen to be of the highest composition in their study whereas Si was seen to be the major element in ours. However, Au was seen to reduce with modification in the different studies due to the reason earlier adduced.

We can therefore conclude that the composition of saw dust cannot be the same but depends on the source of the wood.

Sorption Studies

Effect of contact time on the sorption capacity of crude oil

The effect of contact time on the sorption of crude oil using the acetylated sawdust is shown on table 2 & figure 8 below:

Table 3: Effect of contact time on the sorption capacity of crude oil

Time (minutes)	Weight before sorption (g)	Weight after sorption (g)	OSC (gg^{-1})
5	2	12.210	5.105
10	2	12.455	5.228
15	2	12.230	5.115
20	2	12.051	5.026

The maximum value of oil sorption capacity was at 10th minute, this shows that the sorption phenomenon is fast, and then decreased with increasing sorption time. This fast sorption indicates the presence of sorption sites that can be easily accessed, causing the sorbent and the oil to interact very well. The reduction in sorption capacity as the contact time increases above 10 minutes may be because of the saturation of the available sorption sites (Sokker et al., 2011).

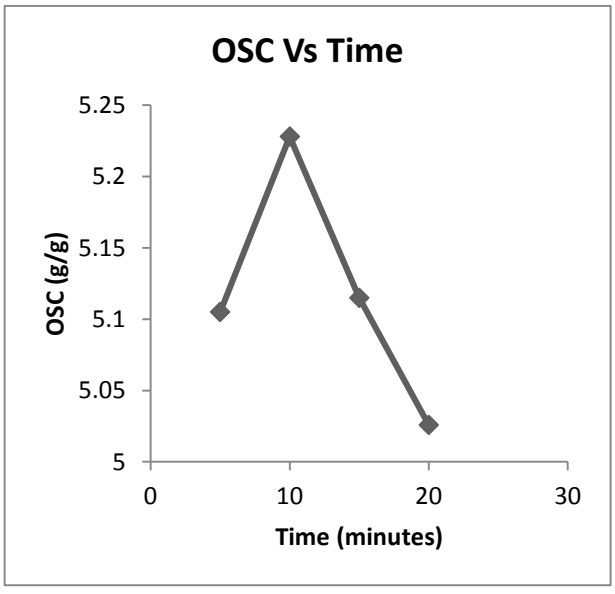

Fig. 9: OSC versus Time

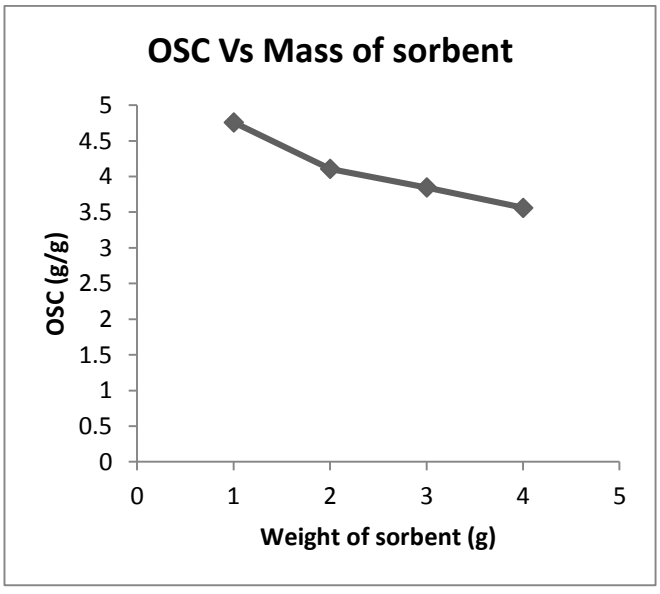

Fig. 10: OSC versus mass of sorbent

Effect of sorbent mass on the sorption capacity of crude oil

The effect of sorbent weight on the sorption capacity of crude oil was investigated and the result is clearly seen in table 4 & figure 10

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Table 4: Effect of sorbent mass on the sorption capacity of crude oil

Weight of sorbent (g)	Weight before sorption (g)	Weight after sorption (g)	OSC (gg^{-1})
1	1	5.759	4.759
2	2	10.213	4.107
3	3	14.542	3.847
4	4	18.243	3.561

The oil sorption capacity of the sorbent decreases as the sorbent mass increases. This is because of higher unsaturated oil binding sites (Ibrahim et al. 2010) and unoccupied surfaces remaining during the sorption process. Similar sorption results were obtained by Pauzan and Ahad, 2018 when they studied the sorption capacity of palm-based cooking oil by surfactant-modified sago hampas, Jmaa and Kallel, 2019, when they studied the assessment of the performance of Posidona oceanica (L.) as bio sorbent for crude oil-spill cleanup in seawater.

Effect of particle size on the sorption capacity of crude oil

Table 5: The effect of particle size on the sorption capacity of crude oil.

Particle size (mm)	Weight before sorption (g)	Weight after sorption (g)	OSC (gg^{-1})
1.0	2	13.094	5.547
0.6	2	13.555	5.778
0.425	2	12.210	5.105
0.106	2	13.675	5.838

As shown above, 0.106mm, the finest particle size gave the highest value for oil sorption capacity. This suggests an increased available surface area of the sorbent's particle size. It can be inferred that decreasing the sorbent's particle size increases the oil sorption capacity.

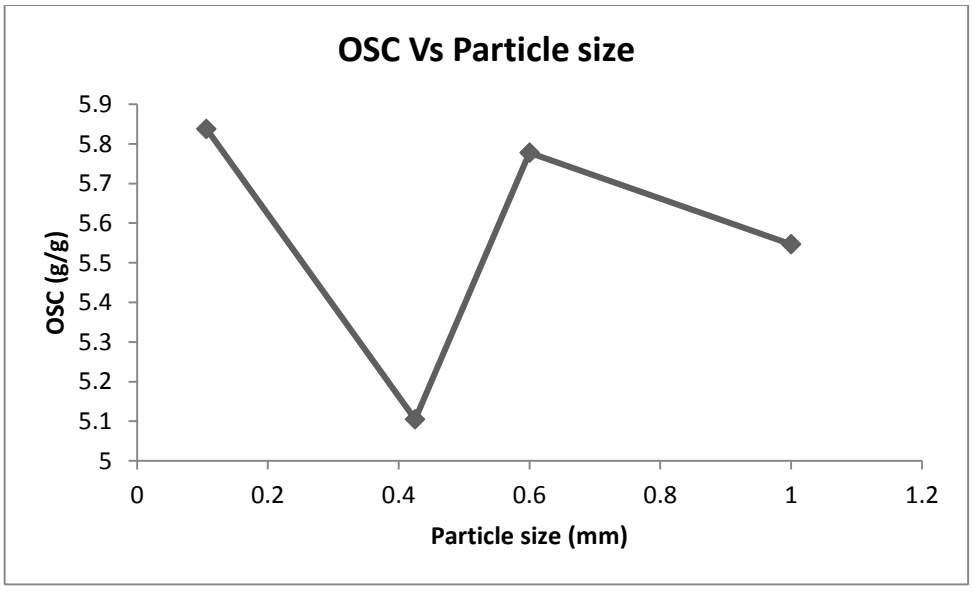

Figure 11: OSC versus Particle size

Comparison between modified and unmodified sorbent

The effect of sorbent modification was investigated using particle size of maximum sorption capacity (0.106mm) to compare their capacities in the uptake of crude oil. The result is shown below:

Table 6: Mean of the relative sorption of modified and unmodified sorbent

Sorbent type	Weight before sorption (g)	Weight after sorption (g)	OSC (g/g)
Modified	2	13.675	5.838
Unmodified	2	12.829	5.415

The sorption capacity of a modified sorbent is 5.838 g/g while that of unmodified sorbent is 5.415 g/g. The chemical modification of the sorbent raised its sorption capacity higher than the unmodified sorbent. This is because the hydroxyl groups in the structure of the cellulose are changed to hydrophobic acetate groups (O-CO-CH_3) through a reaction with acetic anhydride (Onwuka et al. 2018), thus enhancing the sorbent's ability to remove crude oil.

CONCLUSION

The chemical modification of sawdust, a biowaste by acetylation process, has shown to enhance crude oil sorption capacity than the unacetylated one. This is as a result of the displacement of some of the hydroxyl groups and the incorporation of acetyl groups to the sorbent (sawdust) as shown by FTIR characterization. This reduced hydrophilicity and enhanced oleophilicity as evidenced from the WPG. Oil sorption capacity showed dependency on variables like sorbent particle size, sorption time and sorbent dosage.

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