ASSESSMENT OF THE ADSORBENT CAPACITY OF SAWDUST BRIQUETTE IN TREATING CRUDE OIL CONTAMINATED WATER

Odugbose, B. D.*[1], Dada, O. I.[1], Adeyemi, H. O.[2]., Opafola, T. O.[3] and Emeadi, C. F.[1]

[1]*Department of Agricultural Engineering, Faculty of Engineering, College of Engineering and Environmental Studies (CEES), Olabisi Onabanjo University, Ibogun Campus, P. M. B. 5026, Ifo, Ogun State, Nigeria.*
[2]*Department of Mechanical Engineering, Faculty of Engineering, College of Engineering and Environmental Studies (CEES), Olabisi Onabanjo University, Ibogun Campus, P. M. B. 5026, Ifo, Ogun State, Nigeria.*
[3]*Department of Civil Engineering, Faculty of Engineering, College of Engineering and Environmental Studies (CEES), Olabisi Onabanjo University, Ibogun Campus, P. M. B 5026, Ifo, Ogun State, Nigeria.*

*Corresponding author's email *: odugbose.babashola@oouagoiwoye.edu.ng*

ABSTRACT
In crude oil catchment areas of oil-producing countries, oil spillage is a serious environmental challenge. As a result, fertile agricultural lands get polluted and degraded, the morbidity of aquatic animals is rampant, and water is rendered unfit for drinking and general household usage. This work offers a solution to this environmental menace by utilizing sawdust briquettes as filter media in filter columns to purify crude oil-contaminated water. Subjecting both the oil-polluted water and treated water to total petroleum hydrocarbon (TPH) test and water quality test, the study investigated the effectiveness of using the filter columns and the suitability of using the treated water for irrigation purposes. From the results, the adsorption capacity was in the range of 2.86 and 4.01 g/g. It was established that the filter was very effective in treating oil-polluted water as the TPH concentration sharply reduced to about 96.1% from 32960 mg/L in the oil-polluted water to 1284 mg/L in the treated water. However, the reduced TPH, was above the EU EPA standard TPH level required for river and basin waters. The physicochemical properties for the treated water as compared with the oil-polluted water showed drastic EC_w *increase (172.13 ± 0.06 to 708.00 ± 114.59 µS/cm, depicting slight salinity), increased TDS (114.24 ± 0.01 to 479.33 ± 44.99 mg/L), reduced pH but very strongly acidic (6.50 ± 0.00 to 4.37 ± 0.06), reduced turbidity (8.96 ± 0.02 to 5.88 ± 0.24 NTU), increased alkalinity but below optimum (13.12 ± 0.01 to 21.59 ± 0.76 mg/L), and reduced colour (55.18 ± 0.03 to 15.10 ± 0.56 Hz). For the heavy metals, only Copper (Cu), Chromium (Cr) and Iron (Fe) were within the standard limit of irrigation waters while Cadmium (Cd) and Manganese (Mn) were not. It was, therefore, concluded that though it is evident that the use of sawdust briquette as a filter is effective at reducing TPH in crude oil-contaminated water, the treated water is altogether not suitable for irrigation purposes. Therefore, it was recommended that the sawdust filter be upgraded to attain TPH level standard limit.*

KEYWORDS: Absorption, Adsorption, Briquette, Total petroleum hydrocarbon (TPH), Water quality, Water treatment

1. INTRODUCTION

The menace of pollution from oil spillage is very common in crude oil catchment areas of oil-producing countries; a unique case study is the Niger Delta region in Nigeria. This oil spillage is usually as a result of blowouts of oil wells, dumping of petroleum wastes in the waters, leakages from oil tanker trucks and tanks (Inyang *et al.*, 2018), and oil bunkering. Due to this, the morbidity of aquatic animals is inevitable. Also, oil spillage is a major concern to the farmers in the Niger Delta region because fertile agricultural lands get polluted and degraded. This also makes water unfit for drinking and general household usage. As a result, crude oil spillage poses a serious environmental challenge and requires more attention. For this reason, a number of researches have been conducted, aiming to find a lasting solution to oil-contaminated lands (Solisio, 2002; Adebajo, 2003; Radetic, 2008; Korhonen, 2011).

Recently, a number of methods have been established by researchers for clean-up or remediating oil-contaminated land and water. Among these include the use of activated carbon for petroleum-contaminated groundwater remediation (Ayotamuno, 2006), and using hydrophobic nanocellulose aerogels as oil absorbents (Korhonen, 2011). Others include the utilization of wool-based non-woven material as oil adsorbents (Radetic, 2008), inorganic adsorbent (Solisio, 2002), and synthetic porous materials as adsorbents (Adebajo, 2003). The materials used in these methods are, however, expensive. There is, therefore, a need to explore some natural inexpensive methods such as using barley straw (Husseien, 2009; Ibrahim *et al.*, 2010), organically modified clay (Alther, 1995; Moazed, and Viraraghavan, 2005) and bio-remediation (Bragg *et al.*, 1994). Sawdust has also been used as an adsorbent for the removal of methylene blue (MB) dye (Markandeya *et al.*, 2015). Also, Ukpala (2013) studied the physicochemical characteristics of using sawdust as an adsorbent in bioremediation of crude oil polluted environments. In this work also, sawdust was utilized as the purification medium but in form of briquettes.

Sawdust possesses high surface area with low buoyancy which allows it to efficiently adsorb oil content from the oil-water mixture (Zang *et al.*, 2015). The particle size of sawdust also affects its adsorption capacity. Adsorption capacity has been determined to increase directly with reduced adsorbent particle size (Ibrahim *et al.*, 2010) giving rise to an increase in effective contact area (Ngah and Hanafiah, 2008), consequently, resulting in increased adsorption capacity (Fanta *et al.*, 1987). Owing to these properties of sawdust, modified sawdust in form of briquette was used in this study.

In this study, sawdust was made into column briquettes and utilized as the purification medium to treat crude-oil contaminated water. This study further determined the suitability of using sawdust in form of briquettes to treat crude-oil contaminated water by analysing both contaminated and treated waters against the total petroleum hydrocarbon (TPH) level. In addition, the study determined the suitability of the treated water for irrigation purposes by analysing the treated and oil-polluted waters against water quality parameters.

2. MATERIALS AND METHODS

2.1 Materials

The materials used included sawdust of *Sacoglottis gasbonensis* (Baill.) Urb. locally called Itara, binder (cassava starch), crude oil, water, plastic pipes, and metre rule. Equipment used were electric oven (NS Biotec Model ODS-060, SN: 1705018, Power: 1100W 10A 185-240V AC, 40/60Hz), sieve (650micron), sieve shaker (W.S Tyler Ro-Tap, Model: RX-29, No: 890218030010, Power: 150W 220V AC, 50Hz), electronic weighing balance (MP10001

Electronic Balance, Serial: SH100911378, Error: 0.1g), conical flask, mould, and manual briquetting machine.

2.2 Sample collection

Sawdust sample from hardwood bark (*Sacoglottis gasbonensis* tree), was gotten from a sawmill in Ibogun, Ogun State, Nigeria, and located at the coordinates 6°48'41.6"N 3°6'27.0"E. Crude oil sample was obtained at NNPC Depot Ejigbo, Lagos State, Nigeria. The materials were then transferred to the laboratory where the experiment was carried out.

2.3 Particle preparation

The sawdust was sundried in order to remove the moisture present. Since adsorption capacity of sawdust has been determined to be highest in small particle size (Ibrahim *et al*., 2010; Fanta *et al*., 1987), the sawdust was thereafter passed through a 0.65 mm sieve to obtain small particle size and remove foreign materials. The sawdust was treated by washing with water to remove muddy materials and impurities. The water was drained thoroughly by decantation and then oven-dried using an electrical oven at 105°C for 6 hours to remove the remaining water, taking cue from Ikenyiri and Ukpaka (2016). The sawdust was then used to produce briquettes.

2.4 Briquettes production

The treated sawdust was soaked in water at room temperature for about 5 days until it began to decompose. The sawdust required no pounding due to its small particle size. Water was, thereafter, drained off, leaving behind a pulpy mixture. Local starch made from fermented cassava, which served as a binder, was prepared by mixing the starch with cold water and stirring thoroughly after which hot water was added until it became jellylike. The prepared starch was then added to the pulp and mixed thoroughly.

Three cylindrical moulds of 10.3 cm and 5 cm in diameter and height were used to make 2 sets of briquettes; the first set for adsorption experiments and the second set for the filter columns. The moulds had small holes, which functioned as drainage outlets, at the base. The starched pulp was poured into one of the moulds in excess. The water was allowed to drain a bit after which it was compressed with the use of a manual briquetting machine. The briquette was then ejected from the mould after no more water drained from the mould which is an indication of full consolidation from compression conforming with procedure used by other researcher Ngumbi *et al* (2013). After this, the briquette was allowed to air-dry for 10 days.

2.5 Experimental methods

2.5.1 Dry weight and adsorption capacity of the briquettes

The dry weight (W_1) of the briquettes was determined by putting the first set of 3 air-dried briquettes in separate watch glasses of known weight (W_2), and was oven-dried at 110°C for 2 hours taking cue from Jain *et al.* (2014). It was then removed and air-cooled for 3 hours. The combined weight of the glass and briquettes was weighed (W_3) using an electronic weighing balance. The determination of adsorption capacity followed.

2.5.2 Adsorption capacity of the briquettes

The contaminated water was synthesized by mixing crude oil with water in a ratio of 1:5. Therefore, 200 ml of crude oil was poured into 1 litre of water in a bowl. To determine the adsorbent capacity (A_C) of the briquettes, the briquettes were submerged in the oil-water solution and allowed to stand for 150 minutes, for higher adsorption of oil as determined by Nwufo *et al.* (2014). After this, the briquettes were removed and adsorbent weight (W_{AD})

measured using an electronic weighing balance. The adsorbent capacity was then determined using Equation (1).

$$A_C = \frac{W_{AD} - W_1}{W_1} \quad (1)$$

where:
A_C = Adsorbent capacity of briquette (g/g)
$W_1 = W_3 - W_2$ = Oven dry weight of briquette (g)
W_2 = Weight of watch glass (g)
W_3 = Weight of watch glass and oven dried briquette (g)
W_{AD} = Weight of briquette after adsorption (g)

2.5.3 Purification column and process set-up

The exploded view and assembly of the purification column are shown in Figure 1. Two plastic pipes of 10.3 cm and 30 cm in diameter and heights, respectively, were used. One of the pipes was drilled near the end for a plastic tap fitting (Figure 1) and damp-proofed to avoid leakage. A cap was also fitted tightly to the pipe end after the tap fitting to make a closed end (Figure 1), while the other end remained open. On the second pipe, one of the second sets of briquettes, which serves as a filter, was gently inserted into an open end of the pipe, while the other end served as a hopper where the contaminated water would be fed into the purification chamber. After this, the second pipe, which has the briquette, was then corked into the open end of the first pipe to make up a complete purification column (Figure 1).

To initiate the process of purification, the tap was closed and the contaminated water was poured into the purification chamber from the hopper. The tap was opened to collect the purified/treated water (Figure 2). The water was then analysed for water quality and total petroleum hydrocarbon (TPH) tests.

2.5.4 Determination of contaminated water quality and total petroleum hydrocarbon (TPH) level

The following water quality parameters – pH, total dissolved solids (TDS), electrical conductivity (EC), turbidity, colour, and heavy metals (Copper (Cu), Chromium (Cr), Iron (Fe), Cadmium (Cd), Manganese (Mn)). Total petroleum hydrocarbon (TPH) was carried out using a standard gravimetric analysis – EPA Method 1664A. Both water quality and TPH tests were conducted on the contaminated and treated water in order to determine the purification capacity of the purifying medium – sawdust briquette, and the suitability of the treated water for irrigation purpose.

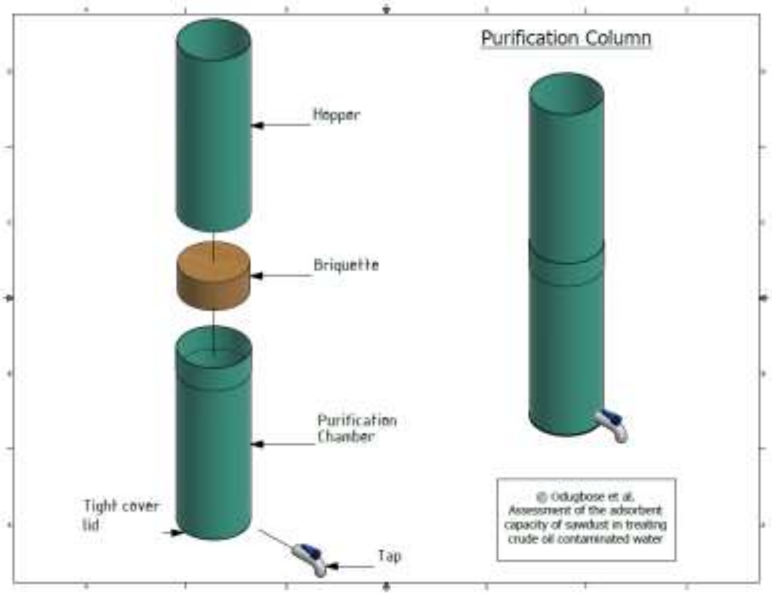

Figure 1: Exploded and assembly views of the purification column set-up

Figure 2: Experimental process showing the purifying columns with the treated samples

3. Results and Discussion

3.1 Absorption and adsorption capacity of the briquettes

The briquettes produced are shown in Figure 3. The results of the oven dry weights of the briquettes are shown in Table 1.

3.1.1 Adsorption capacity

The adsorption capacity of the briquette was between the range of 2.86 and 4.01 g/g (Table 1). As seen from the results, the Sample 3 briquette was able to adsorb about 4 times oil contaminant of its dry weight. On average, the briquettes have the capacity to adsorb oil about 3.53 times contaminants of the briquettes dry weight.

Table 1: Results table showing weight and adsorption capacity of briquettes

Samples	Initial weight of dried adsorbent (g)	Weight after adsorption (g)	Adsorption capacity (g/g)
1	107.40	506.00	3.72
2	95.00	366.40	2.86
3	79.10	396.30	4.01
Mean	93.83	422.90	3.53

Figure 3: A set of three briquettes used in the filter columns

3.2 Total Petroleum Hydrocarbon (TPH)

The presence of TPH indicates the health and quality status of the water as well as a measure of contaminants in the water (Zaghden *et al.*, 2005; Zrafi *et al.*, 2013). The TPH values for both treated and contaminated water are presented in Table 2. From the table, it is observed that there is a sharp reduction in TPH concentration; about 96.1% decrease from contaminated water (32960 mg/L) to treated water (1284 mg/L). Though not totally purified, TPH has reduced considerably. However, there is a need for further treatment as the treated water may not still be useful. Comparing the treated water to river and basin water TPH standard, the treated water is way too short of the EU EPA (2009) standard limit level of TPH 0.3 mg/L for petroleum hydrocarbons in river and basin waters (Adeniji *et al.*, 2017; Inyang *et al.*, 2018).

Table 2: Total Petroleum Hydrocarbon Test Results

Parameters	TPH (mg/L)
Treated Water	1284
Oil contaminated water	32960

3.3 Physicochemical properties of crude oil contaminated water and treated water

The physicochemical properties results of both treated water and crude oil contaminated water are presented in Table 3. As specified by the FAO irrigation water standard (Ayers and Westcot, 1985), the optimum range of pH in irrigation water is between 6.5 and 8.4. From the results gotten, the pH reduced after treatment from 6.50 ± 0.00 in the contaminated sample to 4.37 ± 0.06 in the treated sample. As pH is a vital parameter used in evaluating the acid-base balance of water, it is deduced that the treated sample is very strongly acidic. The treated water is therefore not suitable for irrigation purposes since it is not within the optimum pH needed for irrigation water. It should also be noted that the treated water, due to its low pH, will expedite the corrosion of irrigation lines (Jeong *et al.,* 2016). Thus there is need for further purification measures in order to remediate the water sample for irrigation purposes.

The water salinity status is determined by the level of electrical conductivity (EC_w). A higher EC_w means a high level of salt in the water, which translates to a less fresh portion of water for plant development and transpiration, resulting in physiological drought (Bauder *et al.*, 2014; Fipps, 2003). Water with EC_w 700 µS/cm is considered none saline and typical of drinking water while waters having EC_w between 700 - 2000 µS/cm are considered slightly saline and typical of irrigation waters (Rhoades *et al.*, 1992). In general, irrigation water with EC_w up to 700 µS/cm is best for irrigation with no restriction of usage and no effect on crop growth. However, EC_w greater than 3000 µS/cm is considered unsuitable for irrigation purposes (Ayers and Westcot, 1985). The results (Table 3) show that the treated water is slightly saline, however, the contaminated water is non-saline. From the results (Table 3), the drastic increase of EC_w from 172.13 ± 0.06 µS/cm in the contaminated water to 708.00 ± 114.59 µS/cm in the treated water can be attributed to the dissolution of Na content of the cassava starch as the water penetrates through the briquettes. A 100g of raw cassava contains 14 mg Na (USDA, 2019), and Na is about 39% of the mass of typical NaCl salt. Since the amount of water transpired directly influences yield (Bauder, 2014), the treated water, in this case, can be said to slightly affect crop growth and consequently, its yield if used for irrigation.

Total dissolved solids (TDS) describe the content of inorganic salt and organic matter present in a solution. From the results (Table 3), it is evident that treating the contaminated water with sawdust briquettes caused the particles dissolved in the treated water to increase greatly; increase from 114.24 ± 0.01 mg/L to 479.33 ± 44.99 mg/L. This increase can be attributed to the briquette composition; since starch has high affinity with water (Hossain *et al.*, 2018), the passage of oil-water solution through the briquettes could have made the starch particles dissolve, dissolving along the briquette sawdust particles. As the treated water is above the <450 mg/L standard limits of TDS for irrigation water (Müller and Cornel, 2017; Ayers and Westcot, 1985), the treated water is not useful for irrigation purposes. Using this water for irrigation could cause clogging in the irrigation equipment which in turn would reduce the efficiency, and in some cases could cause total damage, to such equipment.

Turbidity is another important parameter in the analysis of water. It is defined as the cloudiness of water caused by a variety of particles. A high level of turbidity has been attributed to causing lower hydraulic conductivity (Ragusa *et al.*, 1994; Vinten *et al.*, 1983), soil surface pollution and aiding carriage of bacteria and germs from one point to another (US EPA, 2012; Jeong *et al.*, 2016). As seen in Table 3, ≤2 NTU is the standard limit of turbidity in irrigation water for directly consumed crops and unrestricted irrigation (US EPA, 2012; Agrafioti and Diamadopoulos, 2012; Jeong *et al.,* 2016). From the results (Table 3), the turbidity level reduced to 5.88 ± 0.24 NTU after the water was treated. However, it is still above the required limit for irrigation water, hence the treated water is not suitable for irrigation purposes.

The ideal range of total alkalinity in irrigation water is between 30 to 100 mg/L (Swistock, 2016). Alkalinity level below 30 mg/L is considered low and problematic for acid fertilizer applications. In this case, an alkaline water needed to restrict the effect of the acid in the fertilizer. Low alkalinity can lead to issues with micronutrient toxicity and allow the pH of the substrate to gradually decrease (Argo and Fisher, 2018; Swistock, 2018). From the results of both oil-contaminated and treated waters, both have low alkalinity; 13.12 ± 0.01 and 21.59 ± 0.76 respectively. However, the treated water edged closer to the standard limit of 30 mg/L. The result is consistent with the pH result of treated water, which shows strong acidity.

The reduction of the colour property from 55.18 ± 0.03 Hz in the crude oil contaminated water sample to 15.10±0.56 Hz in the treated water sample (Table 3), is evident in Figure 5. The treated water can easily be identified by its lighter colour. This is equally in line with the reduced turbidity; reduced cloudiness of the treated waters.

As seen in the results (Table 3), heavy metals all reduced in concentration after treatment of the contaminated water, except for manganese which increased from 3.11 ± 0.01 mg/L to 6.41 ± 0.65 mg/L. From the standard limit of heavy metals concentration in irrigation water by Ayers and Westcot (1985), Cu, Cr and Fe are within standard limit. The others, which are Cd and Mg are above the acceptable standards.

(a) (b)

Figure 5: (a) Crude oil contaminated water samples (b) treated water samples

Table 3: Physicochemical properties of treated and crude oil contaminated waters

S/N	Parameter	Polluted	Treated	Standard Limit of Irrigation Water
1	pH	6.50±0.00	4.37±0.06	6.5-8.4[a]
2	Colour (Hz)	55.18±0.03	15.10±0.56	N/A
3	Total Dissolved Solid (mg/L)	114.24±0.01	479.33±44.99	<450[a]
4	Electrical conductivity (µS/cm)	172.13±0.06	708.00±114.59	700-2000[d]
5	Turbidity (NTU)	8.96±0.02	5.88±0.24	≤2[b c]
6	Alkalinity (mg/L)	13.12±0.01	21.59±0.76	30-100[e]
7	Copper (mg/L)	0.28±0.01	0.23±0.07	0.2[a]
8	Chromium (mg/L)	0.00±0.00	0.00±0.00	0.1[a]
9	Iron (mg/L)	5.63±0.01	1.22±0.04	5.0[a]
10	Cadmium (mg/L)	1.04±0.00	0.16±0.03	0.01[a]
11	Manganese (mg/L)	3.11±0.01	6.41±0.65	0.2[a]

[a]Ayers and Westcot (1985), [b]US EPA (2012), [c]Agrafioti and Diamadopoulos (2012), [d]Rhoades *et al*., (1992), [e]Swistock (2018)

4. CONCLUSION AND RECOMMENDATION

From this research, it is evident that the use of sawdust as a purification medium is effective at reducing the Total Petroleum Hydrocarbons (TPH) in crude oil contaminated water. Nevertheless, the treated water is altogether not suitable for irrigation purposes. Therefore, it is recommended that the purification set-up be upgraded to include additional purification media for removal of TPH to standard limits.

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