[1.]K. W. SALEEMAN, [2.]H. K. IBRAHIM, [3.]Y. O. BUSARI, [3.]Y. L. SHUAIN–BABATA,
[1.]A. G. ADUAGBA, [3.]A. S. ADEGBOLA, [3.]K. S. AJAO, [3.]I. O. AMBALI

DEVELOPMENT OF FUNCTIONAL OIL–ADSORPTION MATERIAL FROM WATERMELON RIND FOR REMEDIATION OIL SPILL RELATED POLLUTION

[1.]National Agency for Science and Engineering Infrastructure (NASENI) Abuja, NIGERIA
[2.]Department of Mechanical Engineering, University of Ilorin, Ilorin, NIGERIA
[3.]Department of Material and Metallurgical Engineering, University of Ilorin, Ilorin, NIGERIA

Abstract: The cost of common sorbent materials are relatively low but have poor recyclability, absorption capacity and are not environmentally friendly. Agricultural products are now been utilized as a substitution for environmentally friendly and low-cost oil sorbent materials. The present study aimed to develop and investigate the oil adsorption efficiency of modified watermelon rind for remediation of oil-contaminated water pollution. The particle size of the adsorbent was determined using mesh sizes of 0.3-1 mm. The pyrolysis experiments of Watermelon rind were carried out within the temperature range 250–700 ^{O}C. The composition of the pyrolyzed watermelon rind (PRA) was determined and functionalized with Methyltrichlorosilane (MTCS) and Methyltrimethoxysilane to form an improved oleophilic and hydrophobic surface. The adsorption properties and morphology of both adsorbents were characterized using FTIR and SEM respectively. Adsorption capacity, dose, and recyclability tests were carried out on three adsorbates (Motor oil and diesel fuel, and vegetable oil). The result showed that functionalizing the PRA improved the performance of the adsorbent with increasing oil adsorption capacities and oil selectivity. The findings also showed that oil recoverability and sorbents recyclability is possible with this material especially recovering with hexane.
Keywords: Functionalized, Pollution, Adsorption, Recyclability, Pyrollised

INTRODUCTION

The frequent occurring environmental pollution during accidental discharge of oil resulted in severe environmental damages. This can be sited from the recent devastating oil spillage at Venezuela, Mauritius, and Canada across the ocean. In the quest to provide an effective control of oil contamination from water, the use of adsorbents has been gaining attention (Asadpour *et al* 2015). The cost of common sorbent materials are relatively low, most of which the raw materials are from natural organic fibers (straw, wool, cotton), inorganic materials (zeolites, activated carbons, clays), and synthetic organic polymers (polypropylene, alkyl acrylate copolymers). Application of these materials have been reported with poor recyclability, absorption ability, degradability, and not eco-friendly. Some synthetic sorbent materials with excellent sorptive properties (graphite foams, graphene, carbon nanotubes sponges, aerogels or hydrogels, and hydrophobic polymer coatings on polyurethane sponges) are very expensive and harmful due to the required fabrication process with complex equipment (Zamparas *et al.,* 2020).

Currently, agricultural waste materials that also contributed to the environmental hazards are considered for sorption process due to its abundancy, eco-friendliness, and biodegradability properties. The materials have been reported with poor sorptive properties and can be enhanced by modification techniques using chemical, mechanical, and thermal modification approaches that include pyrolysis treatment of sorbent materials which involved treatments at temperatures above 700 °C, or functionalizing the pre-processing or post-processing modified sorbent materials with coating additives (Stolz *et al.,* 2016).

A study by Uzunov *et al.* (2012) reported the effects of the pyrolysis process on the oil sorption capacity of rice husk. The results show that the oil sorption capacity at a moderate temperature, 480 ◦C, is moderately influenced by the porous structure and promoted the sorption of oil and oil products in aqueous media. Behnood *et al.* (2016) examined the crude oil sorption capacity with raw sugarcane bagasse and acetylated sugarcane bagasse. It was concluded that the acetylated bagasse was significantly more oleophilic than the raw bagasse and the acetylation reaction increased bagasse oil sorption capacity by about 90%. Tang *et al.* (2018) modified wheat straw with palmitic acid as an efficient oil spill adsorbent. The capacity of pretreated wheat straw (PWS) and esterified wheat straw (EWS) was only 7.13±0.86 g/g and 14.00±0.50 g/g and this showed strong hydrophobicity and rapid oil absorption during the initial 20 s. Bayık and Altin (2018) utilized industrial waste to produce an oil sorbent by coupling it with functional silane coatings. The percentage of water sorption for uncoated and surface coated sorbent with (3-Aminopropyl) triethoxysilane (APS) and 10% for 3-(Trimethoxysilyl) propyl methacrylate (MPS) were 38.24% and 9.27% respectively. The results showed that industrial waste is promising oil sorbing material by silane coupling. El Gheriany *et al.* (2020) evaluated the oil sorption capacity of dried raw orange peel waste (OP) and thermally modified (300 ^{O}C and 500 ^{O}C) orange peel waste (TMOP). The results have indicated the water uptake of the TMOP is significantly higher than OP with better oil retention characteristics. The use of watermelon rind has been utilized

as adsorbent for the removal of heavy metal ions. It has been reported WR contained pectin, citrulline, cellulose, proteins, and carotenoids which are rich in functional groups such as hydroxyl (cellulose), amine (proteins), and carboxylic (pectin) that are hydrophilic groups, making it to adsorb organic pollutants. However, no research has consider the use of modified Watermelon rind for oil contaminated water sorption process. The present study aimed to develop and investigate the adsorption behavior of modified functional watermelon rind for remediation of oil contaminated water pollution.

MATERIALS AND METHODS

— Preparation of Modified Adsorbent Material

The Watermelon rind were obtained from local stores in Coventry city UK. The rind was shredded, transferred to a tray and washed severally with distilled water to remove physical impurities. The washed samples were dried at 60°C for 48hours in vacuum to less than 5% moisture content and grinded and mechanically sieved to 0.3-1 mm particle size and lower as shown in Figure 1. The dried sample was put in an evaporating basin of 14.5cm x 7cm hollow furnace for pyrolysis treatment. The pyrolysis of was carried out in two batches as reported by Jiao, Wan and Li (2016). The first batch of the dried sample was held at temperature of 500°C for 1 hour and raised to 700°C for 2 hours and allow cool to room temperature and a black ash-like substance was obtained. The second batch of the dried sample was held at temperature of 700°C for 1hour, then raised to 1000°C for 2hours and white ash-like substance (PRA) was obtained.
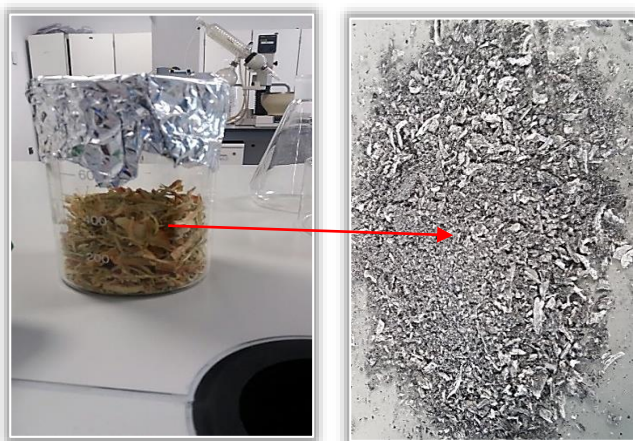

Figure 1: dried and Pyrolysed (Modified) watermelon rind

— Preparation of Functionalized Samples

The modified Watermelon rind was coated with Methyltrichlorosilane (MTCS) and Methyltrimethoxysilane, and then poured into a tea bag to improve the functional hydrophobic properties. Then 200 mL of toluene was added to the particles as the reaction solvent. Besides, a beaker of MTCS solution was prepared with addition of 2, 3 and 4 mL of MTCS and MTMS in10 mL of ethanol. The obtained solution was added to the tea bag dropwise under a nitrogen atmosphere and agitated for 5 h. After reaction, the sample was dried in an oven at 60°C. The pyrolised watermelon rind was functionalized with 10 ml MTCS and 10 ml MTMS was briefly named as functionalized samples (FRA) shown in Figure 2.

Figure 2: Settling Unfuctionalised and Floating Functionalised PRA in Water

— Preparation of the Adsorbate

50mL of deionized water was measured in a 100mL measuring cylinder and transferred into a 200mL glass beaker. The motor and diesel oil SAE 20W/50 of low and medium viscosity as reported by Senanurakwarkul *et al.* (2013) with physical properties (Density and Viscosity) in Table 1 were purchased from PTT (Thailand) Co., Ltd. The 5ml of diesel oil and machine oil stained with Sudan red were mixed separately with the measured deionized water by agitating the mixture on a flask shaker for 30 minutes as carried out by Husin *et al* (2011). Each beaker was labelled accordingly.

Table 1: Adsorbate Physical Properties

Oil Type	Density (g/cm^3)	Viscosity (cP)
Mortor Oil	0.92	133
Diesel Oil	0.832	210.4

— Experimental Analyses

The morphology of the surface of the PRA and that of FRA were observed by scanning electron microscopy (SEM) using JEOL JSM 6390 microscope applying the appropriate magnification. The amounts of the dispersed fluids were determined by extraction with acetone. The determination of the organic compounds composition of the acetone extract from raw watermelon rind has been carried out using a GCMS-QP2010 PLUS, SHIMADZU, JAPAN equipped with a mass spectrometric detector (GC/MS). The obtained organic compounds were also characterized using Fourier transformer infrared (FTIR) spectroscopy (FTIR- 8400S instrument). The infrared spectra were recorded on a Nicolet Avatar 360 spectrometer in a tablet KBr at a spectral resolution of 2 cm^{-1} and scanned in the 4000–400 cm^{-1} range.

Batch adsorption experiments for the samples were carried out using pure oil and oil-water solution. Ranges of adsorbent mass (0.5g, 1.0g, 1.5g and 2.0g) were prepared in tea bag, weighted and dipped in the pure oil at interval of 10minutes. At drip time of 1minutes, the mass of the adsorbent was measured using ASTM F 716–09 standard for oil sorption capacity of the pyrolysed and functionalsed samples to calculate sorption capacity by dividing the final mass after oil sorption by the initial mass before sorption.

The adsorbent was then taken out with a tweezer and the final concentrations were calculated using equation 1. The experiment was conducted at 30 $^\circ$C room temperature and at 45–55% relative air humidity.

Mechanical squeezing was first conducted for recyclability on the two sorbents by squeezing with two pairs of tweezers to recover the different oil and reuse material. Thereafter, the adsorbent was weighed before reapplying immediately for another sorption cycle. This process was repeated until each adsorbent could no longer undergo more adsorption. A second method adapted from EPA method 9071B was applied by washing the adsorbents in n-hexane for four hours using soxhlet. This was done by putting the adsorbent in an extraction thimble and thereafter measuring 150mL of n-hexane into a 250mL round bottom flask which had boiling chips put in it. The soxhlet apparatus was set up to get the heating to a cycling rate of 20 cycles per hour and this was done for four hours to extract the oil out of the sorbent. The n-hexane washed adsorbent was then placed in an oven to dry. The adsorbent was reweighed before using for another cycle of adsorption. This was repeated for every cycle of recyclability. The oil used in this test was motor oil. Subsequently, the extract was decanted to another round bottom flask and attached to a rotary evaporator for 15 minutes in order for the n-hexane to evaporate. Thereafter, the residue oil was recycled for reuse.

RESULT AND DISCUSSION

— Characterization of adsorbent

The composition of organic substances watermelon rind was obtained from GC/MS analysis. The Relative abundance organic compounds has been reported to contained two phases that include low molecular weight of organic oxygen-containing compound (acids, aldehydes, esters, ketones, and phenols) and a phase that contained water-insoluble organics (alkenes, arenes, and cycloalkenes) of high molecular weight (Uzunov et al., 2012). The extract of watermelon rind is characterized with ten major and abundant compounds as shown in Figure 3.
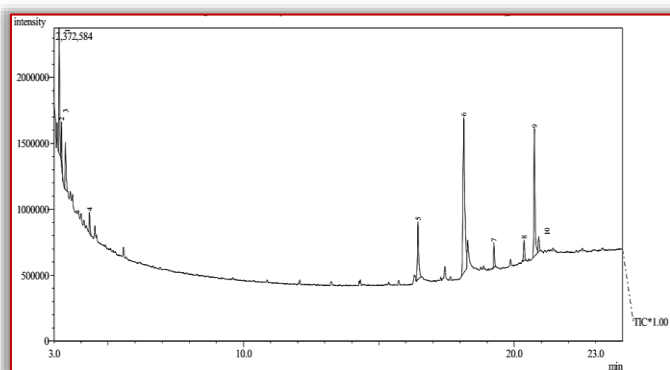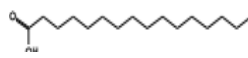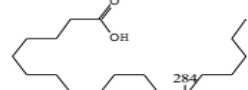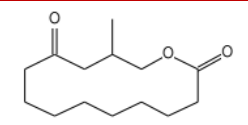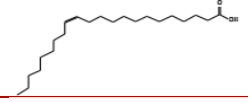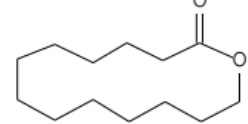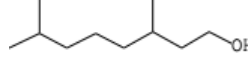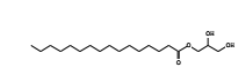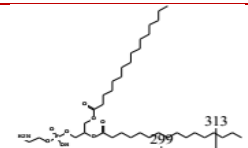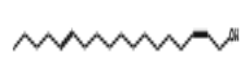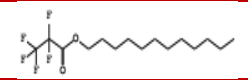

Figure 3: Chromatogram of Watermelon Rind Extract

Table 2 shows the result obtained in GCMS bands of watermelon rind which contained mainly variety of organic oxygen-containing compounds with higher molecular weight. This showed the reason for poor sorptive properties that required modification techniques such as pyrolysis and esterification to obtain water-insoluble organic compounds.

Table 2: Major Organic Compounds name and their structure obtained from GCMS

S/N	Compound Name	Formula	Molecular Weight	Compound Structure
1	Pentadecanecarboxylic acid	C16H32O2	256	
2	Octadecanoic acid	C18H36O2	284	
3	3-Methyloxacyclotetradecane-2,11-dione	C14H24O3	240	
4	Erucic acid	C22H42O2	338	
5	Oxacyclotetradecan-2-one	C13H24O2	212	
6	3,7-Dimethyl-1-octanol	C10H22O	158	
7	2,3-dihydroxypropyl ester	C19H38O4	330	
8	Hexadecanoic acid	C37H74NO8P	691	
9	Z,E-2,13-Octadecadien-1-ol	C18H34O	266	
10	Pentafluoropropionic acid	C18H31F5O2	374	

The pyrolysis process involved increasing temperature that lead to deterioration of oxygen compounds that forms large amount of high molecular weight alkanes with low temperature increment whereas at higher temperature, compounds of lower molecular weight are formed.

The adsorption characteristic of modified watermelon rind was conducted with FTIR. The FTIR showed the transmittance spectral of organic compound that displayed many peaks indicating different functional groups. The spectral for raw watermelon rind has been reported with a broad peak at 3635 cm-1 assigned to O–H stretching vibrations (polyphenolic group), 2910 to C–H stretching vibration of the methyl and methoxy groups. The peak at 1730 cm-1 assigned to C=O stretching vibration of acid derivatives, and the weak band at 1310 cm-1 is attributed to amide groups (Prasad et al., 2015). However, the intense peak corresponds to –OH (hydroxyl) stretching vibrations, for the modified WR showed in Figure 4 was obtained at 3427.62 cm–1. The peaks at 2932.86 cm–1 assigned to C–H, peak at 1631.83 cm–1 corresponds to C=O stretching of

carboxylic acid groups or esters, and asymmetric and symmetric vibrations of ionic carboxylic groups (–COO–), respectively, appeared at 1432.19 and 1080.17 cm−1. These results suggest that WR predominantly contain organic functional groups such as hydroxyl, carboxyl and carbonyl groups. Meanwhile, the change in the spectral values indicate the destruction processes during the thermal cracking of lignocellulose materials.
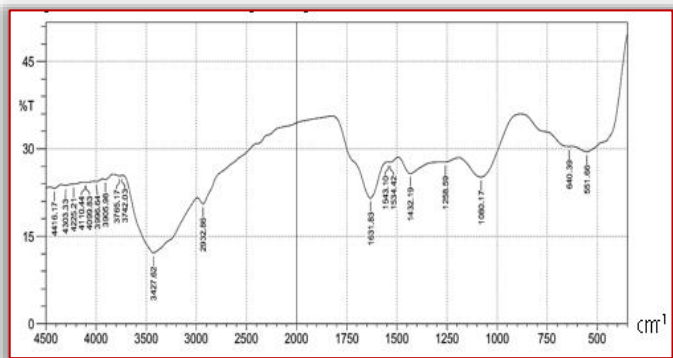

Figure 4: Spectrogram of Modified Watermelon Rind

The results from the spectral analysis showed that functional groups intensity decrease with increasing the pyrolysis temperature which resulted to formation of some volatile compounds like cellulose and lignin. It has been reported that cellulose, and lignin build a stable porous structural units that have an optimal affinity for hydrophilic and hydrophobic liquids (Mahmoud, 2020) but decomposed within the temperature interval 300–400∘C and above 400∘C for lignin. The destructive reactions has been attributed to decreasing polymerization degree like depolymerization, hydrolysis, oxidation, dehydration and decarboxylation (Uzunov *et al.,* 2012). This thermal degradation lead to the formation of more stable condensed structures with pores to accommodate oil substances.

The morphology of the different adsorbent was characterized using SEM operating at 10.00kV as shown Figure 5. The SEM image of the PRA has a unified structure compare to the FRA which shows an infinite porous structure. These differences in the functionalized materials compared to their unfunctionalized form could be attributed to the effect of salination on the materials by creating a non-polar interphase for increased oil sorption.

—— Effect of Functionalization on Absorption

Hydrophobic property and affinity of sorbent material to adsorb oil and organic solvent can be effective with modification approach like pyrolysis process which make it hydrophilic and functionalized to induce hydrophobicity. It can be observed in Figure 6 that adsorption capacity of FRA sorbent with two adsorbate is higher than the PRA material. The adsorbent adsorbed the water to a saturation point with an adsorption capacity of 3.4420g/g and the hydrophobicity increased by 70.3% with water adsorption capacity of 1.0215g/g by functionalizing the PRA with MTCS. This was as a result of the silanation reaction between chlorosilane and water molecules that are loosely combined with the surface

of the PRA. Thus, presented a great number of accessible sites and high surface energies with non-polar interphase by eliminating the hydroxyl groups (Makowski *et al.,* 2014).

Figure 5: The Morphology of Pyrolysed (Modified) and Functionalised Watermelon Rind.

The result of functionalization on the PRA is in consistent with findings of Wang *et al* (2012) where dodecyltrimethoxysilane (DTMS) was used to functionalize Kapok fibre. The oleophilicity increased by 46.6% for diesel oil sorption and 20.2% for soybean oil sorption. However, the water adsorption capacity of the MTCS functionalized PRA is lower than that functionalized with MTMS. Compared to MTCS coated samples, MTMS functionalized PRA had a water adsorption capacity of 0.3339g/g. The better performance of MTCS over MTMS may be because the latter is synthesized from the former, hence has a lower concentration (Latthe *et al* 2012).
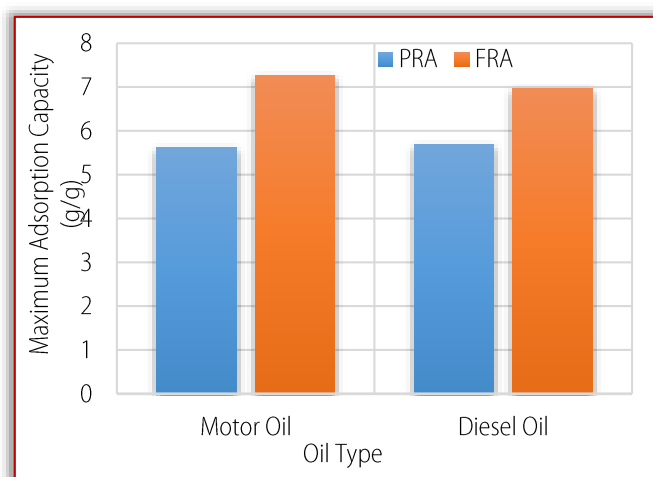

Figure 6: A comparison between PRA and FRA for motor oil, and diesel oil.

— Effect of Contact Time on Oil Sorption Capacity

The experimental data were measured at 60 minutes, with an interval of 10 minutes and the effect of time on sorbents with oil-contaminated water was established as shown Figure 5. From the result for all the two oil types (motor oil, and diesel oil), there is an increment in adsorption capacity with increasing contact time for PRA, and FRA. The fastest rate was observed between the first 10minutes and 20 minutes. This is in line with the result reported by Behnood *et al* (2016) that fastest sorption was achieved at an early stage due to the high porosity of the sorbent. Thereafter, the adsorption capacity continued to increase but at a slower rate till the 50th-60th minute. This adsorption trend could be due to plain active sites on the adsorbent surface which then got to saturation phase later with time as more oil molecules are taken up by the initial plain sites and led to slower rate of adsorption and this is consistent with the findings of Mahmoud (2020); El Gheriany *et al.*, (2020).
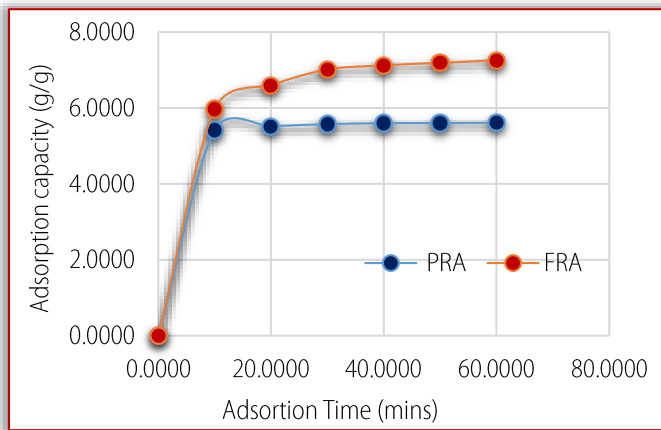

Figure 7: Effect of adsorption time of PRA and FRA on Motor Oil
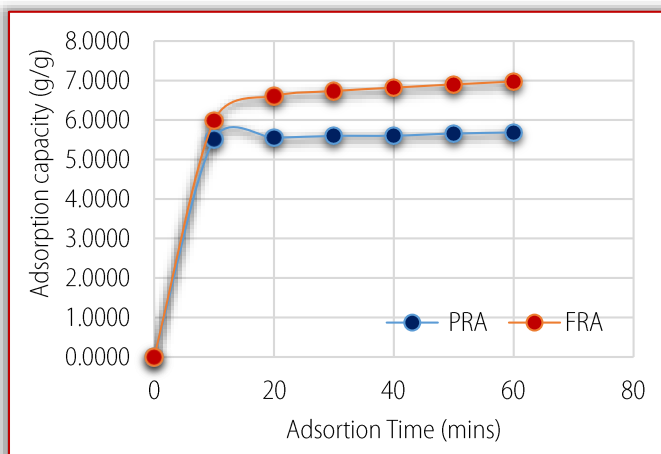

Figure 8: Effect of adsorption time of PRA and FRA on Diesel Oil

It can be revealed in Figure 7 and 8 that PRA and FRA had their optimum adsorption at 60 minutes contact time. For motor oil, an optimum adsorption capacity of 5.6108g/g, and 7.2560g/g for PRA, and FRA respectively were observed. Whereas for diesel oil, maximum adsorption capacities of 5.6860g/g, and 6.9760g/g were obtained for PRA, and FRA respectively as shown in Figure 8.

— Effect of Initial Concentration of Oil

The initial oil concentration was used to study the percentage of adsorbed oil concentration under optimized condition of 60 minutes contact time and 0.5g adsorbent. Using 100mL of water, varying initial oil concentrations of 25%v/v-45%v/v at an interval of 5%v/v were used to evaluate the effect of oil concentration on adsorption. From table 3, it showed that sorption capacity increased with increasing initial oil concentration; however, the percentage of oil adsorbed decreased by increasing initial oil concentration.

Table 3: Effect of Initial oil concentration on Motor and Diesel oil

Adsorbent	Initial oil concentration (%) v/v	Final oil concentration % (v/v)	Motor Oil removed (%)	Diesel Oil removed (%)	Motor Oil Adsorption capacity (g/g)	Diesel Oil Adsorption capacity (g/g)
PRA	25	21.5	14.0	12.4	5.5322	4.9000
	30	26	13.3	11.7	6.3226	5.5322
	35	30.7	12.3	10.6	6.7968	5.8484
	40	35.7	10.8	10.5	6.8491	6.6387
	45	40.6	9.8	10.4	6.9548	7.4290
FRA	25	20.2	19.2	16.0	7.5871	6.3226
	30	25.1	16.3	13.7	7.7451	6.4806
	35	29.8	14.9	12.0	8.2193	6.6387
	40	34.6	13.5	10.6	8.5355	6.6703
	45	39.5	12.2	10.5	8.6935	7.4448

The percentage of oil removed for PRA on motor oil decreased by 4.2% as initial oil concentration increased from 25%v/v to 45%v/v. There was 7% reduction in oil removed percentage for FRA on motor oil from the lowest initial oil concentration to the highest initial oil concentration. The same trend was encountered for diesel oil where oil removed for PRA reduced by 2%, and FRA by 6.5%. A similar trend was reported by Tiwari (2011) and attributed the reduction in percentage of oil removed to an increase in number of oil molecules while adsorbent amount remains the same. However, increasing adsorption capacity per unit mass of the adsorbent with increasing initial oil concentration could be as a result of available active sites that were not possible at low concentration and utilization of adsorption surface (Razavi *et al.*, 2014).

— Oil Recoverability and Recyclability

Adsorption recyclability is another vital property to investigate the beahviour of an adsorbent. This contributed towards saving cost, reduction in waste generation and energy saving in production process. The sorbent materials showed some contents of oil adsorbed in sorbent material and some incomplete removal of oil as the mass of the reweighed sorbents increased by 20-25% of the initial weight of the dry sorbent. The optimum capacity of the PRA and FRA decreased by 40% and 45% respectively showed in Figure 9. The decrease in adsorption capacity can be attributed to the damage of porous structure and also the increased remnant mass (Brindha *et al.*, 2019).

Secondly, the motor oil-adsorbed material was washed with hexane and oil was recovered in a rotary evaporator. After six cycles, the PRA could no longer undergo recyclability as observed in Figure 10. The adsorption capacity of hexane washed FRA at the fifth cycle was even slightly higher than that recorded at the second cycle when mechanical squeezing was applied.
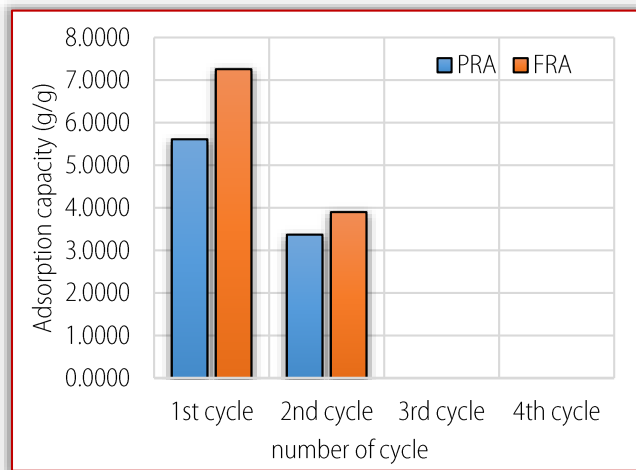

Figure 9: Recyclability of PRA, and FRA by squeezing

This could be due to its hydrophilicity which allowed FRA to move with two more times up to the eighth cycle. There was insignificant weight gain in the reweighed sorbent samples after being washed with hexane and dried in an oven. This showed excellent oil recoverability which was confirmed when the recovered oil was weighed after separation from hexane in a rotary evaporator.
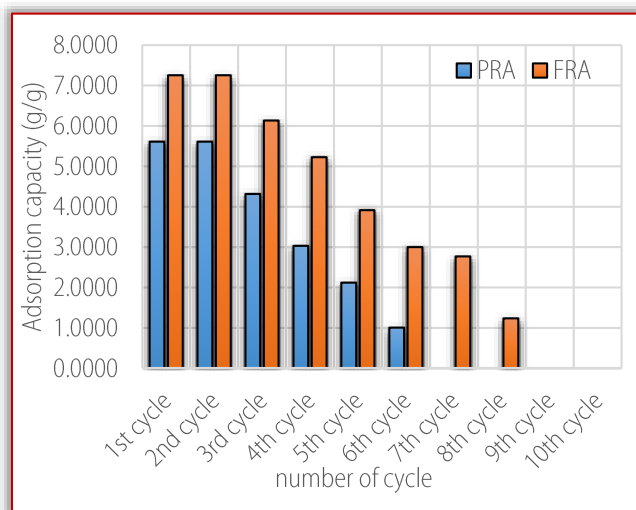

Figure 10: Recyclability of PRA, and FRA using hexane as solvent

The oil recovered was about 0.2% less of the initial mass of oil that was adsorbed by the adsorbent. The excellent recyclability could be attributed to high oil recoverability which gave room to more sites for adsorption compared to mechanical squeezing. From Figure 10 below, it can be seen that reduction in adsorption capacities tends to be higher as recyclability increase; this reason could be attributed to damage of porous structures as reusability increases (Alaa El-Din *et al.,* 2017). Although squeezing the oil is very energy efficient but eventually, the free space for adsorption is progressively restricted as a result of damaged porous structure and retained oil. In comparison, El Gheriany *et al.* (2020) established that the process enhanced removal of oil leaving behind not more than 0.5% of the residual oil.

— Influence of Adsorbent Dose

The influence of adsorbent quantity was studied using ranging mass of adsorbent from 0.5g to 2.0g at an interval of 0.5g. This was studied in motor oil, and diesel oil using PRA and FRA adsorbents.
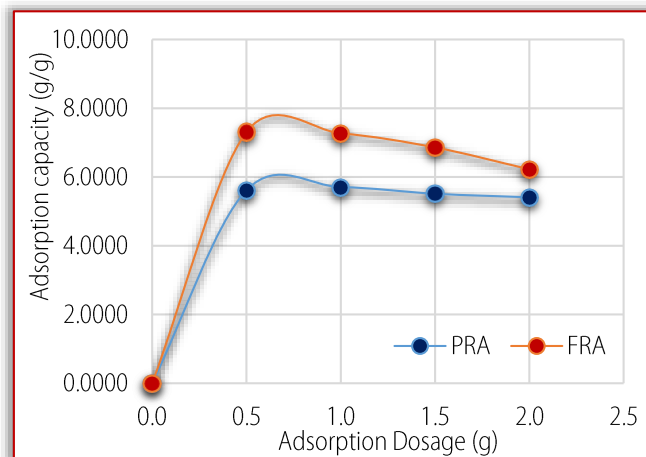

Figure 11: Influence of adsorbent dose on PRA and FRA.

From the Figures 11, it can be observed that adsorption capacity initially increased with increasing adsorbent dose. However, this decreased with further increase in adsorbent dose. An optimum adsorption capacity of 8.1206g/g and 8.4995g/g at 1.0g adsorbent dose was observed for the PRA and FRA respectively in vegetable oil. This same attribute was obtained for diesel oil. A similar trend was observed by Aljeboree *et al.* (2014) in the study on the adsorption of textile dyes using activated carbon from coconut shell. Though the adsorption capacities tend to reduce with increasing adsorbent dose, the amount of oil adsorbed continued to increase with increasing dose of adsorbent till the last dose of adsorbent was applied. The initial increase in adsorption capacities could be explained to be attributed to the presence of higher active sites at a lower concentration of adsorbent dose (Padmavathy *et al.,* 2015). Consequently, the further reduction in adsorption capacities with an increase in adsorbent dose could as well be attributed to the unsaturated adsorption sites during adsorption reaction. Further increment in concentration of adsorbent dose may cause aggregation of adsorbent. The increase in the amount of oil adsorbed could be understood from the fact that increase in adsorption dose increases the number of available adsorption site (Shahul *et al.,* 2017).

CONCLUSION

It can be deduced that the adsorption capacities of sorbents is affected by a lot of factors; ranging from the concentration of the spilled oil to the time of contact of adsorbent with the adsorbate. The experimental analyses showed that functionalizing the pyrolysed watermelon rind with MTCS improved the performance of the two adsorbents by

increasing the oil adsorption capacities and oil selectivity. The findings also showed that oil recoverability and sorbents recyclability is possible with these materials especially recovering with hexane. Therefore, MTCS functionalized watermelon rind can be proposed as a sorbent material for oil spill clean-up in wetlands and aquatic areas.

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