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RESEARCH ARTICLE **AJHSE010202**

MODELLING AND OPTIMIZING THE APPLICATION OF WASTE TYRE POWDER (WTP) AS OIL SORBENT, USING RESPONSE SURFACE METHODOLOGY (RSM)

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ABSTRACT

The rapid growth of the automobile industry has led to the abundance and indiscriminate disposal of waste tyres which causes environmental pollution and also lead to serious health problems. The absorption of crude oil using waste tyre powder (WTP) was investigated. A three variable Box-Behnken design was used to study the effect of particle size, contact time and temperature on the oil sorption capacity of WTP. Optimization was carried out using Response Surface Methodology (RSM). A quadratic model was obtained to predict the oil sorption capacity of WTP as a function of particle size, contact time and temperature. The optimum conditions of the sorption process obtained from RSM gave a temperature of 30.19oC, contact time 59.04 mins and particle size 0.15mm. A maximum oil sorption capacity of 4.71 g/g was obtained at these optimized conditions. Also, a comparison between the oil sorption efficiency of fresh tyre powder and regenerated tyre powder subjected to the same conditions of particle size, contact time and temperature were carried out. It was shown that the oil sorption capacity of the fresh tyre powder was higher than that of regenerated tyre powder.

Keywords: *waste rubber, tyre, oil spill, energy efficiency, waste management, technical performance*

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INTRODUCTION

Nigeria can be considered as a densely populated nation and with it comes acquisition of automobiles of different grades. Connected with these automobiles are waste tyres that have raised so much environmental concerns due to its negative impact on the environment (Adhikari *et al*., 2000; Kumaravel *et al*., 2016). Waste tyres are not biodegradable and take up landfill spaces (Williams *et al*., 1990; Kar, 2011). A lot of damaged and worn out tyres are present in Nigeria and this constitutes environmental nuisance to the environment. Ebewele and Dzong (1990) estimated the volume of waste tyres to be about 5 million in a study they carried out between 1979 and 1983. It is envisaged that the volume of waste tyres would quadruple.

When these tyres are discarded at dumpsites, they create problems of space for the overwhelming growing population and these sites become habitat for mice and other rodents (Shu and Huang, 2004; Yilmaz and Degirmenci, 2009). These waste tyres also collected water in their grooves after rainfall and these become breeding grounds for mosquitoes. Some tyres can be ignited by accident and when they are burnt, some of the tyres emit a characteristic smell and toxic vapors (Ewadinger and Steuteville, 1996).

Oil spillage will continue to be a major concern not only due to the negative effect on the environment but also to health hazards associated to it. Globally, the high demand of energy coupled with the activities that accompany production, exploration, transportation and marketing of crude oil and petroleum products have led to increased number of spills in terrestrial and aquatic bodies. Some of these spills occur during loading and unloading of oil, transportation on sea and land, damaged oil pipes caused by worn out pipe material or vandalization of these pipes by oil thieves and ocean oil drilling. Therefore, the need for an immediate and important need to develop advanced materials and technologies for the cleanup of oil and water-insoluble organic liquids from the surface of water become imperative *(Wu et a*l., 2014).

The use of sorbent materials has become more attractive for oil-spill cleanup because of the possibility of collection and complete removal of the oil while causing no side effects to the environment. Some of the adsorbents used for oil spill clean -up includes but not limited to modified organophilic clays, lime, silica, exfoliated graphite, hydrocarbon and plastic polymers, cellulose-based materials and elastomers (Daughney, 2000; Gitipour *et al*., 1997; Lee *et al*., 1995; Hyung-Min and Cloud, 1992; Meininghaus and Prins, 2000; Reynolds, *et al*, 2001; Teas *et al*., 2001). Other adsorbents in use are zeolites, silica, perlite, graphite, vermiculites, sorbent clay, diatomite (Kumaravel *et al*., 2016; Melvold *et al*, 1988).

Ebewele and Dzong, (1990) discovered and reported that recycled rubber from waste tyres possesses a characteristic ability of absorbing hydrocarbon liquids and gases with relatively low density in water. Aisien *et al*., 2003, also reported that rubber from waste tyres absorbs four to five times its weight of oil. In this study, the use of recycled rubber from waste tyres was applied for oil spill clean-up experienced in the oil rich Niger Delta region of Nigeria. The essence of this work will not only reduce the growing menace caused by these waste tyres in the environment but will also decrease the rate of death of aquatic organisms in the Niger Delta region of Nigeria caused by oil spillage.

METHODOLOGY

MATERIALS COLLECTION

Crude oil of specific density 0.84 was obtained from Warri Refining and Petrochemical Company (WRPC), Ekpan-Warri, Nigeria. Car waste tyres were collected from a local vulcanizer, in Benin City, Nigeria.

PRE-TREATMENT OF WASTE TYRES

The waste tyre was washed using detergent and dried. The cleaned side of the tyre free from steel ‘threads’ was cut into thin long sections with a hacksaw and later into very small pieces that could fit into an electric grinding machine with the aid of sharp knives. These tyre chips were then transferred into a clean electric grinding machine. The electric grinding machine was used to reduce the waste tyre chips to tyre powder. The grounded mixture was then mechanically sieved into different particle sizes using mesh sizes of 0.150mm, 0.212mm and 0.300mm. The different sizes of tyre powder were collected and kept in separate plastic containers.

PROCEDURE

100 cm^3 of crude oil was measured into a beaker with a measuring cylinder. Ten grams of a given particle size of the WTP was measured with the aid of an electrical weighing balance (Mettler PM 4800 Delta range balance). The temperature of the crude oil was kept constant at the desired temperature of 30, 35 and 40 ^oC by the use of a water bath and the 10 grams of the WTP were added to the already measured crude oil and allowed to be in contact for a specified period of time of 10, 35 and 60 mins.

After the required contact time, the contents of the beaker were sieved, until there were no further drops of crude oil observed. The new weight of the waste tyre powder was measured using the weighing balance. The difference between the new weight and the original weight of the WTP gave the value of the weight of the crude oil absorbed for that particular contact time.

Crude oil absorption capacity (g oil/ g waste tyre rubber) =

$$\frac{\text{Mass of crude oil absorbed by waste tyre rubber}}{\text{Mass of waste tyre rubber}} \tag{1}$$

DESIGN OF EXPERIMENT

A three variable Box-Behnken design (BBD) with categoric factor of 0 for RSM was utilized to develop a statistical model and optimize the oil absorption process. The design comprised of three levels (low, medium and high, being coded as −1, 0 and +1) and a total of 17 runs were carried out in duplicate to optimize the level of chosen variables, such as particle size, temperature and contact time. For the purpose of statistical computations, the three independent variables were denoted as X_1, X_2 and X_3 respectively. The level of variables optimized are shown in Table 1. The design is capable and efficient for the exploration of quadratic response surfaces and building a model that is a polynomial of second order (Amenaghawon and Ogbeide,2014). A number of experimental runs were utilized during the response of the surface to the various variables to optimize the process. The number of experimental runs is denoted by the expression below:

$$N= k^2+k+c_p \tag{2}$$

Where k is the factor number and c_p indicate the replicate central point. The coded and actual values of the variables are derived using the expression:

$$x_i = \frac{X_i - X_o}{\Delta X_i} \qquad (3)$$

where xi and Xi represents the coded and actual values of the independent variable respectively. Xo indicates the actual value of the independent variable at the centre point and ΔXi represents the step change in the actual value of the independent variable. For RSM, the widely used second-order polynomial equation developed to fit the experimental data and determine the predicted response can be written as:

$$Y_{predicted} = \beta_0 + \sum \beta_i x_i + \sum \beta_{ii} x_i^2 + \sum \beta_{ij} x_i x_j + \varepsilon \qquad (4)$$

Where $Y_{predicted}$ represents the predicted response i.e. the oil absorption capacity by the waste tire rubber (g/g), β_0 represents the constant coefficient, β_i indicates the ith linear coefficient of the input factor x_i, β_{ii} is the ith quadratic coefficient of the input factors x_i, β_{ij} the different interaction coefficients between input factors x_i and x_j (i = 1–3, j = 1–3 and i is not equal to j), and ε, the error term of the model (Liu *et al.*; 2012)

Table 1: Coded and actual levels of the factors for a three factor Box-Behnken Design

Variables	Units	Symbols	Coded and actual levels		
			-1	**0**	**+1**
Particle size	mm	X_1	0.15	0.212	0.30
Contact time	minutes	X_3	10	35	60
Temperature	°C	X_2	30	35	40

RESULTS AND DISCUSSION

STATISTICAL ANALYSIS

Box Behnken was used to develop a polynomial regression equation for the analysis of the correlation between the oil sorption capacity and the operating parameters i.e. temperature, particle size and contact time. the coded and actual values of the factors X_1 (particle size), X_2 (contact time) and X_3(temperature) as designed by design expert 7.0 and the results for the 17 experimental runs are summarized in Table 2. Multiple regression was applied on the experimental data and the following second-degree polynomial was found to represent the relationship between the oil sorption capacity (Y) and particle size (X_1), contact time (X_2) and temperature (X_3) in terms of the actual factors.

$$\begin{aligned} Y = \; & 8.61574 - 2.83694X_1 + 0.095443X_2 - 0.39160X_3 \\ & -3.78317E\text{-}003X_1X_2 + 0.028374X_1X_3 + 2.80000E\text{-}004X_2X_3 \\ & -0.86755X_1^2 - 6.48800E\text{-}004X_2^2 + 4.78000E\text{-}003X_3^2 \end{aligned} \qquad (5)$$

The second degree polynomial (equation 5) was used to determine the RSM predicted values presented in Table 2. A comparision of the RSM predicted values with the actual values obtained experimentally was carried out and a little deviation was noted meaning that they are both in reasonable agreeement. The significance of fit for the second degree polynomial of oil sorption capacity was investigated by carrying out analysis of variance (ANOVA) as shown in Table 3 and Table 4

Table 2: Experimental and predicted Response by RSM

Run no.	**Coded value of factors**			**Actual value of factors**			**Responses (g/g)**	
	X_1	**X_2**	**X_3**	**X_1**	**X_2**	**X_3**	**Actual value**	**RSM predicted value**
1	0	0	0	0.212	35	35	1.34	1.40
2	0	1	1	0.212	60	40	1.13	1.07
3	0	1	-1	0.212	60	30	4.41	4.46
4	0	0	0	0.212	35	35	4.14	4.08
5	0	0	0	0.212	35	35	3.59	3.62
6	1	0	-1	0.300	35	30	3.14	3.26
7	1	-1	0	0.300	10	35	3.44	3.29
8	-1	0	1	0.150	35	40	2.95	2.94
9	0	0	0	0.212	35	35	1.84	1.41
10	0	-1	-1	0.212	10	30	4.63	4.56
11	1	0	1	0.300	35	40	1.12	1.19
12	-1	-1	0	0.150	10	35	4.05	4.13
13	-1	1	0	0.150	60	35	3.15	3.20
14	0	0	0	0.212	35	35	3.15	3.20
15	1	1	0	0.300	60	35	3.12	3.20
16	0	-1	1	0.212	10	40	3.35	3.20
17	`-1	0	-1	0.150	35	30	3.21	3.20

Results obtained after carrying out ANOVA is presented in Table 3. The Model F-value of 143.44 and a very low probability value [(Prob > F) less than 0.0001] implies the model is significant. There is only a 0.01% chance that a "Model F-Value" this large could occur due to noise. Values of "Prob > F" less than 0.0500 indicate model terms are significant. In this case X_1, X_2, X_3, X_2^2 are significant model terms.

The "Lack of Fit F-value" of 2.88 implies the Lack of Fit is not significant relative to the pure error. There is a 16.63% chance that a "Lack of Fit F-value" this large could occur due to noise. Non-significant lack of fit is good as the intention is for the model to fit.

The coefficient of determination (R^2) of the model was 0.9946 as seen in Table 4, which gives the indication that the model gave an adequate representation of the real relationship between the considered variables. An R^2 value of 0.9946 means that 99.46% of the variability was explained by the model and only 0.54% was as a result of chance. The R^2 value indicates the degree to which the model was able to predict the response. The closer the R^2 value is to unity, the better the model can predict the response. The coefficient of variation (C.V.) obtained was 4.09%. The Coefficient of Variation indicates the degree of precision with which the runs were carried out. A low value of C.V suggest a high reliability of the experiment. Also, from Table 4, it can be seen that the "Pred R-Squared" of 0.9370 is in reasonable agreement with the "Adj R-Squared" of 0.9877 since their difference is less than 0.2. The "Adeq

Precision" measures the signal to noise ratio. A ratio greater than 4 is desirable. The value of 36.670 indicates an adequate signal and hence this model can be used to navigate the design space.

Table 1: Analysis of variance (ANOVA) for quadratic model of oil sorption capacity

Source	**Sum of Squares**	**Df**	**Mean Square**	**F Value**	**p-value Prob> F**	
Model	19.79	9	2.20	143.44	<0.0001	significant
X_1-particle size	0.25	1	0.25	16.44	0.0048	
X_2- contact time	18.15	1	18.15	1184.37	<0.0001	
X_3-temperature	0.20	1	0.20	13.33	0.0082	
X_1X_2	8.612E-004	1	8.612E-004	0.056	0.8194	
X_1X_3	4.078E-004	1	4.078E-004	2.660E-003	0.9603	
X_2X_3	0.011	1	0.011	0.72	0.4244	
X_1^2	6.31E-003	1	6.31E-003	0.41	0.5416	
X_2^2	0.85	1	0.85	55.44	0.0001	
X_3^2	0.024	1	0.024	1.58	0.2496	
Residual	0.11	7	0.015			
Lack of fit	0.073	3	0.024	2.88	0.1663	Not significant
Pure Error	0.034	4	8.480E-003			
Cor Total	19.89	16				

Table 2: Statistical information for ANOVA

Std. Dev.	**0.12**	**R-Squared**	**0.9946**
Mean	3.02	Adj R-Squared	0.9877
C.V.%	4.09	Pred R-Squared	0.9370
PRESS	1.25	Adeq Precision	36.670

OPTIMIZATION OF OIL SORPTION CAPACITY OF WTP

In order to carry out optimization of the variables that influence the oil sorption capacity of WTP, response surface plots were generated from the regression model to examine the effect of the interaction between the independent variables and evaluate their optimum variables. The effect of particle size and contact time on the oil sorption capacity is presented in Figure 1. It is seen that as the contact time increased from 10 minutes to 60 minutes and particle size decreased from 0.30 mm to 0.15mm, there was an increase in the oil sorption capacity with a maximum value of 4.5 g/g.

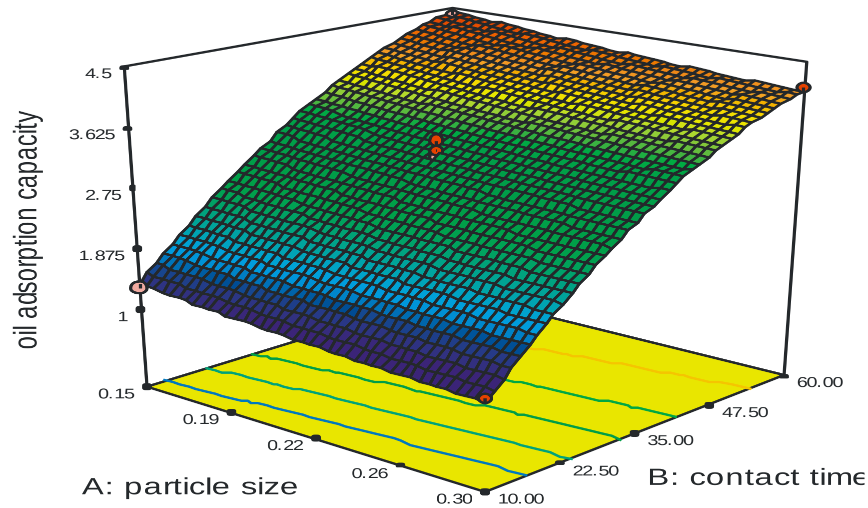

Figure 1: Response surface plot showing the effect of particle size and contact time on oil sorption capacity of WTP

Figure 2 shows the effect of particle size and temperature on the oil sorption capacity. It is shown that as the temperature is decreased from 40ºC to 30ºC and particle size decreased from 0.30 mm to 0.15mm, there was an increase in the oil sorption capacity with a maximum value of 3.63 g/g.

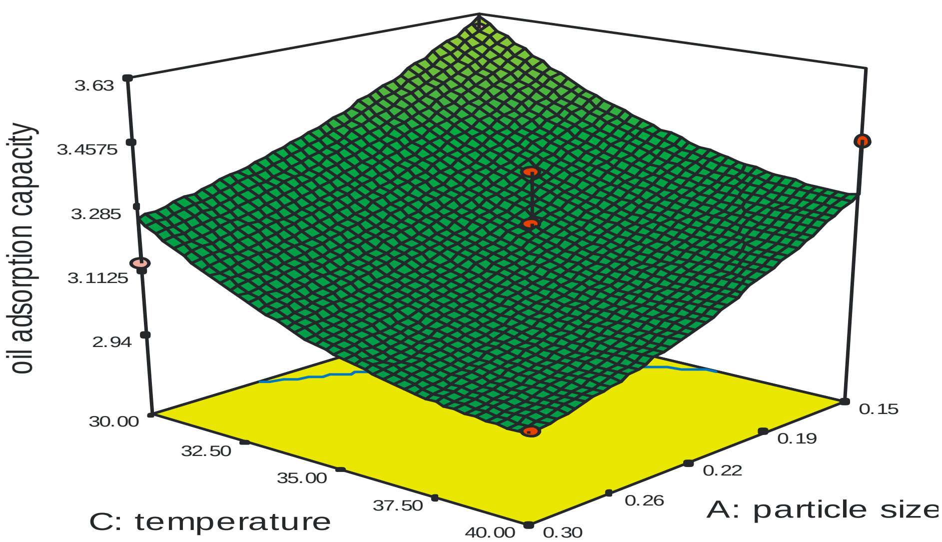

Figure 2: Response surface plot showing the effect of particle size and temperature on oil sorption capacity of WTP

Figure 3 shows the effect of contact time and temperature on the oil sorption capacity. It is shown that as the temperature is decreased from 40ºC to 30ºC and contact time increased from 10 minutes to 60 minutes, there was an increase in the oil sorption capacity with a maximum value of 4.6 g/g.

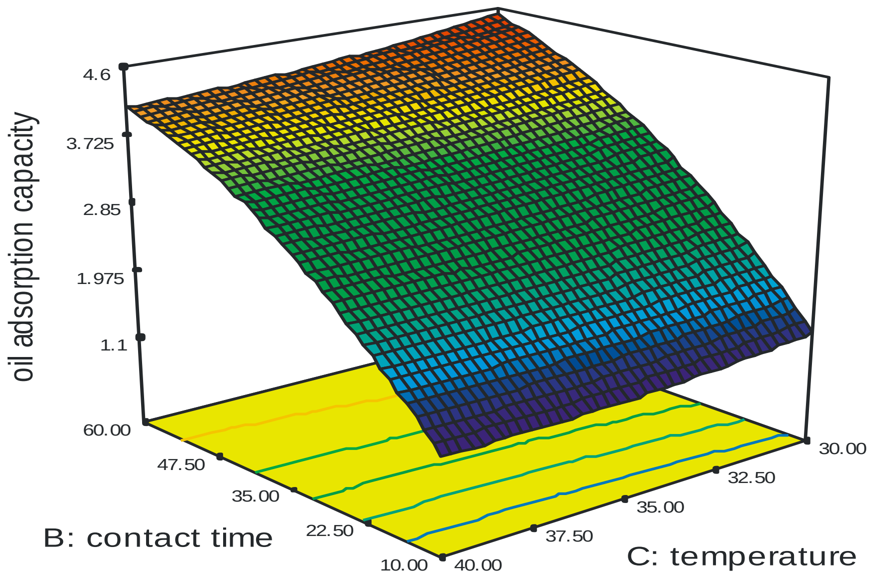

Figure 3: Response surface plot showing the effect of temperature and contact time on oil sorption capacity of WTP

Oil sorption capacity decreases as temperature increases, this is because as temperature increases, the kinetic energy of the crude oil molecules increases making retention or sorption of crude oil by the tyre powder more difficult due to the reduction of the attractive forces. These results are common for all sorption processes (i.e. absorption and adsorption) and corroborates results obtained and reported by Aisien *et al*.; 2003. Higher temperatures result to a low viscosity in oil, making it difficult for the oil to adhere (stick) to the WTP. When the temperature falls, there is a corresponding increase in the viscosity of the crude oil, thereby increasing the rate of sorption of oil as possible. This suggests that the WTP would be more effective and recover more oil in cold regions than in tropical regions (Lin *et al*,; 2010)

Oil sorption capacity increases linearly as contact time increases, this is followed by a slower increase in sorption rate, then the line gradually becomes horizontal i.e. the level of absorption gradually becomes constant, which means no sorption occurs.

This profile of sorption of crude oil is mainly accounted for by the oil concentration difference which acts as the driving force. The difference between the concentration of crude oil in the rubber particle initially and that at equilibrium is high and there is rapid uptake of crude oil by the rubber, this difference in crude oil concentration eventually decreases to zero at equilibrium sorption of oil and there is no more uptake of crude oil (Aisien *et al*.; 2003).

The uptake of the crude oil by the WTP is a sorption process, which depends on the surface area of contact between the crude oil and the WTP, the higher the surface area of contact, the higher the oil sorption process. The smaller rubber particles have larger surface areas per unit mass and therefore higher oil sorption.

To determine the optimum values of particle size, contact time and temperature that resulted in the maximum oil sorption capacity, the statistical model of Equation 5 was optimized. The result of optimization by response surface methodology indicated a maximum oil sorption capacity of 4.71 g/g. The optimum conditions of the sorption process that resulted in this value are as follows: temperature 30.19^{o}C, contact time 59.04 minutes and particle size 0.15mm.

COMPARISON OF THE SORPTION CAPACITY OF FRESH AND REGENERATED TIRE POWDER

Comparison of the oil sorption capacity of fresh rubber and regenerated tyre powder by subjecting them to the same conditions was undertaken, i.e. their particle sizes were both kept constant at 0.300mm and the temperatures of the crude oil at 32^{o}C and contact time was varied for 10-50 minutes. The results obtained are summarized in Table 5

Table 3: Comparison of the sorption capacity of fresh and regenerated tyre powder at same temperature and particle size but varying contact time

			Oil sorption capacity (g/g)		
Time (mins)	**Particle size (mm)**	**Temperature (ºc)**	**Fresh rubber**	**Regenerated rubber**	**Deviation**
10	0.30	32	4.41	3.064	1.346
20	0.30	32	4.79	3.55	1.240
30	0.30	32	4.84	3.826	1..014
40	0.30	32	4.85	3.95	0.900
50	0.30	32	4.88	4.034	0.846

A graphical representation of Table 5 is given in Figure 4, from the plot it can be seen that shape of the curve (oil sorption pattern) for both regenerated and fresh rubber are similar, because contact time have the same effect for both tyre powders, that is. there is an initial rapid increase in the crude oil absorption. This is followed by a slower increase in sorption rate, then the line gradually becomes horizontal, that is,.the level of absorption gradually becomes constant which means no sorption occurred.

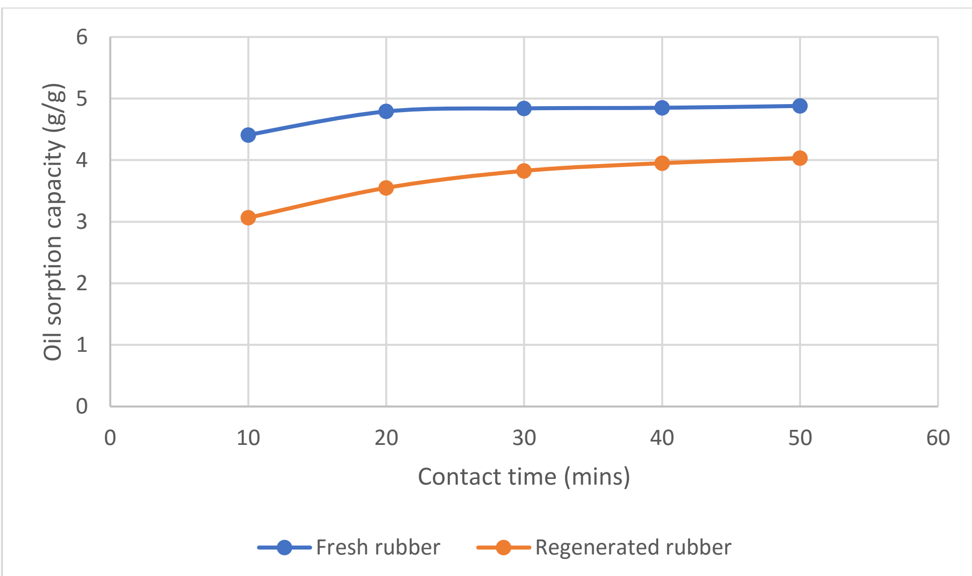

Figure 4: Comparison of the sorption capacity of fresh and regenerated tyre powder at same temperature and particle size but varying contact time

From Table 5, it can be seen that the oil sorption capacity of fresh tyre powder is higher than those of regenerated rubber. This is because of incomplete removal of the crude oil in the regenerated powder and hence lower oil concentration difference than in fresh rubber powder which is the driving force for the oil uptake and results to lower oil sorption (Aisien *et al*., 2003).

Though the oil sorption capacity of fresh rubber is greater than that of regenerated rubber, it can be seen that this decreases with contact time i.e. from 1.346 g/g at 10 mins to 0.846 g/g for 50 minutes. The regenerated rubber powder can still absorb as much as 3-4 times of oil as its initial weight and hence the regenerated rubber can be re-used as sorbent for oil spill clean-up.

CONCLUSION

Waste tyres powder has been demonstrated to have potential for use as sorbents for oil spill clean-up, because of its intrinsic ability to absorb crude oil. Rubber powder from waste tyres can absorb as high as four to five times its weight of oil. RSM with Box-Behnken design has proved to be a useful tool in analyzing the important parameters that influence the oil sorption capacity of WTP (temperature, contact time and particle size) as well as their optimized levels (conditions). The optimum conditions of the sorption process were determined to be 30.19^{o}C for the temperature, 59.04 minutes for the contact time for particle size of 0.15mm. A maximum oil sorption capacity of 4.71 g/g was obtained at these optimized conditions. The oil sorption capacity (efficiency) of fresh rubber is greater than that of regenerated rubber and it decreases with contact time at constant temperature and particle size. We consider the application of RSM in exploring the absorption and desorption capability of WTP for oil spill clean-up as novel. Detailed characterization of the findings employing advanced analytical methods are not included in this report as emphasis was only on the sorption capability of WTP.

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