JOURNAL OF ADVANCED INDUSTRIAL TECHNOLOGY AND APPLICATION
e-ISSN: 2716-7097

Vol. 6 No. 2 (2025) 102-109
https://publisher.uthm.edu.my/ojs/index.php/jaita

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Carbonised Agro-Waste Adsorbents for Automotive Lubricating Oil Removal

Nivethah Sokalinggam[1], Hasnida Harun[2]*, Nurdalila Saji[2], Nor Hazren Abdul Hamid[2], Nor Maizzaty Abdullah[2], Inawati Othman[3], Noor Hasyimah Rosman[4]

[1] *Ardence International School,*
Eco Ardence, Setia Alam, Shah Alam, Selangor, 40170, MALAYSIA
[2] *Department of Civil Engineering Technology, Faculty of Engineering Technology,*
Universiti Tun Hussein Onn Malaysia, Campus-(Pagoh Branch), Pagoh, 84600, MALAYSIA
[3] *Department of Civil Engineering, Faculty of Engineering,*
Universiti Malaysia Sarawak, 94300 Kota Samarhan, Sarawak, MALAYSIA
[4] *Department of Civil Engineering, Faculty of Engineering and Built Environment,*
Universiti Kebangsaan Malaysia, 43600 UKM Bangi, Selangor, MALAYSIA

*Corresponding Author: hasnidah@uthm.edu.my
DOI: https://doi.org/10.30880/jaita.2025.06.02.010

Article Info

Received: 27 June 2025
Accepted: 16 December 2025
Available online: 31 December 2025

Keywords

Automotive lubricating oil, carbonised adsorbents, agro-waste adsorbent, rice husk adsorbent, sugarcane bagasse adsorbent, CCD, SDG, circular economy

Abstract

The increasing generation of waste lubricating oil, driven by expanding industrial and automotive activity, poses a significant threat to aquatic ecosystems due to its persistence, hydrophobicity, and complex chemical composition. This study evaluates two abundant agro-waste residues: rice husk (RH) and sugarcane bagasse (SB) as potential adsorbents for lubricating oil removal. Both biomasses were pre-treated with 1M NaOH to enhance surface porosity and hydrophobicity, followed by carbonisation at 550 °C. Three adsorbent formulations were prepared based on RH:SB weight ratios of 24:6, 15:15, and 6:24 g. Batch experiments were conducted at oil concentrations of 25, 30, and 35 v/v% and contact times of 10, 20, and 30 min using a fixed 30g adsorbent dosage of RH and SB. The response behaviour was further examined using ANOVA and contour-plot analysis under Response Surface Methodology to interpret factor interactions. Optimum values for RH-SB adsorbent are 15:15 g of adsorbent dosage, 25 v/v% of oil concentration and 30 min of contact time. These resulted in 100% removal efficiency under controlled batch conditions. FTIR analysis confirmed the presence of lignocellulosic structures supporting oil–adsorbent interactions. Overall, the findings demonstrate that carbonised RH and SB are viable, low-cost, and environmentally sustainable adsorbents and are strongly recommended for removing used lubricating oil.

1. Introduction

Automotive lubricating oil is formulated from mineral or synthetic base oils that may contain high-molecular-weight hydrocarbons, trace metals, and by-products of thermo-oxidative degradation. It is crucial in ensuring engine reliability by minimizing friction, dispersing heat, and protecting against wear through the formation of a lubricating film [1]. However, once released into the environment, they may contribute to the oil's persistence. The lubricating oil components, such as hydrocarbons and heavy metals, can enter directly into aquatic systems through stormwater runoff, drainage pathways, or soil percolation due to leakage during transportation or servicing, improper handling, or both direct and indirect disposal. When lubricating oil is introduced into aquatic environments, it can persist as a contaminant, potentially harming marine life, disrupting ecological functions, and diminishing water quality [2]. Additionally, oil components that seep into the soil may eventually reach groundwater, posing a threat to communities that depend on these water sources. These processes altogether demonstrate that the lubricating oil can easily enter aquatic environments. Moreover, removing automotive lubricating oil from water bodies is a big challenge due to the chemical and harmful compositions, such as hydrophobic hydrocarbons, which have a high molecular weight that may create long-lasting and stable emulsions that are difficult to remove by using conventional techniques [3]. The struggle in eliminating these contaminants is driving conventional wastewater treatment systems to adopt more advanced methods such as adsorption, advanced oxidation, or bioremediation, which are resource-intensive friendly.

Adsorption is recognized as one of the most effective and practical methods for removing oil from contaminated water. Adsorbents such as activated carbon [4], bentonite [5], and chemically modified natural adsorbents [6] consistently exhibit high removal efficiency, particularly when utilized under optimal conditions, where their performance can achieve nearly complete removal. Agricultural by-products like rice husk [7] and sawdust [8], which are abundant and low-cost, also demonstrate strong adsorption capabilities, making them a favorable adsorbent in removing contaminants from water bodies. The advantage of the adsorption process is that it does not leave additional pollutants in the treatment system, but it transfers the contaminants onto a solid phase that is suitable for controlled handling and disposal.

Rice husk (RH) and sugarcane bagasse (SB) are promising adsorbent materials due to their abundance as agricultural byproducts that have demonstrated effective performance in removing contaminants, including heavy metals, dyes and volatile compounds. Their adsorption performance can be further enhanced through carbonisation and chemical activation. Carbonisation develops carbon-rich porous structures, while NaOH activation generates additional porosity and functional groups that strengthen interactions with hydrophobic molecules [9–10]. These modifications are expected to improve the hydrophobicity and adsorption capacity of RH and SB toward lubricating oil. Despite extensive research on agro-waste adsorbents, limited studies have examined carbonised RH–SB composites specifically for lubricating-oil remediation, as most work focuses on dye or metal removal. This study hypothesized that the NaOH-activated carbonized RH and SB adsorbent would exhibit enhanced adsorption efficiency for automotive lubricating oil, with performance expected to be significantly influenced by oil concentrations, adsorbent dosage, and contact time.

To optimize adsorption processes, the mechanism typically adheres to pseudo-second-order kinetics, indicating dependable chemisorption behavior. In adsorption studies, statistical tools like analysis of variance (ANOVA) and contour plots within the framework of Response Surface Methodology (RSM) are frequently used to assess factor effects and interaction behavior, ensuring a comprehensive evaluation of adsorption performance [11]. Furthermore, optimisation studies using central composite design (CCD) within the RSM framework for oil adsorption using RH–SB blends remain scarce. The combined effects of oil concentration, contact time, and dosage on lubricating-oil adsorption are also not well established.

This study therefore aims to evaluate the adsorption performance of carbonized and chemically modified RH and SB for the removal of used automotive lubricating oil. The effects of oil concentration, contact time, and adsorbent dosage are examined by utilizing statistical tools such as ANOVA and contour-plot analysis within the RSM framework to interpret factor interactions. Transforming RH and SB into carbonised adsorbents supports sustainable waste valorisation while providing an environmentally responsible approach to managing lubricating-oil contamination.

2. Methodology

2.1 Materials

Rice husk, a major by-product of rice milling that constitutes about 20% of the rice grain's weight, was sourced from a rice mill in Selangor, Malaysia. Meanwhile, sugarcane bagasse was obtained from local stalls, served as an additional lignocellulosic material for carbonised adsorbent production. The used automotive lubricating oil, collected from a local workshop, was used as the target contaminant due to its complex chemical composition and classified as hazardous waste.

2.2 Synthesis of Carbonised Adsorbents

In this study, rice husk (RH) and sugarcane bagasse (SB) were used as materials for adsorbents. Initially, both RH and SB were washed with distilled water to remove any impurities and then proceeded with the drying process in an oven for 24 hours at 105°C before undergoing chemical treatment. Each adsorbent was immersed in 1M sodium hydroxide (NaOH) for 1 hour to enhance pore development that may result in an increase in surface area and thereby improve adsorption performance. After the chemical treatment, the adsorbents were rinsed to eliminate any residual NaOH until the pH was stable. The treated adsorbents were then ground and sieved to a particle size of 400 μm before carbonisation. Carbonisation was conducted in a muffle furnace at 550 °C, with RH pyrolyzed for 20 minutes and SB for 45 minutes. Both carbonised adsorbents were subsequently characterised using Fourier transform infrared (FTIR) spectroscopy to identify the functional groups present.

2.3 Batch Experiments

A Central Composite Design (CCD) was applied to structure the batch adsorption experiments and evaluate the effects of operating variables on the removal of used automotive lubricating oil. The design considered three independent factors: lubricating oil concentration (25, 30, and 35 v/v%), contact time (10, 20, and 30 min), and adsorbent dosage ratio. The total weight of adsorbent dosage consisting of carbonised rice husk (RH) and carbonised sugarcane bagasse (SB) was prepared for 30g with three different mass ratios, 24:6 g, 15:15 g, and 6:24 g, to assess the influence of precursor proportion on adsorption performance. For each CCD run, the specified oil volume was mixed with 300 mL of water for 10 minutes at room temperature. With the oil floating on the surface water, 30 g of adsorbents (at specified RH to SB ratio) were added and mixed at specified contact time, before removal of the adsorbent using a sieving mesh. The oil-loaded adsorbents were air-dried and measured [12,13]. Adsorbent dosage ratio, oil concentration, and contact time were varied to determine their effects. The amount of oil removed was obtained from the weight of the adsorbents before and after the adsorption process, and the corresponding removal efficiency was calculated using the following equation:

$$\text{Oil Removal (\%)} = \frac{Co-Ce}{Co} \text{ x } 100 \qquad (1)$$

Where C_o (mL) is the initial volume of oil, and C_e (mL) is the final volume of oil.

2.4 Experimental Design

Response Surface Methodology (RSM) integrated with CCD was employed to optimise the oil removal process within the experimental ranges of 25-35 v/v% oil concentration, 6–24 g/L adsorbent dosage of RH ratio, and 10–30 min contact time. Initial oil concentration significantly affected removal performance, with lower concentrations favouring higher adsorption due to reduced competition for active sites. Adsorbent dosage also played a major role by controlling the number of available adsorption sites; however, diminishing returns were observed at higher dosages, indicating the presence of an optimum. Contact time influenced the progression toward adsorption equilibrium, with removal efficiency increasing rapidly at early stages before stabilising within the 10–30 min window. The experimental data were fitted using a second-order polynomial model of the form:

$$Y = \beta_0 + \sum\beta_i X_i + \sum\beta_{ii} X_i^2 + \sum\beta_{ij} X_i X_j \qquad (2)$$

where Y is the predicted removal efficiency, and X_i and X_j are the coded values of oil concentration, adsorbent dosage, and contact time. This model effectively captured both the linear and nonlinear behaviour of the system and identified optimal operating conditions within the studied range. Overall, the CCD framework provided a statistically reliable and experimentally efficient method for optimising oil removal, enabling accurate prediction of process performance and identification of conditions that maximise removal efficiency. The experimental variables considered in this study were: (A) oil concentration, (B) adsorbent dosage of RH ratio, and (C) contact time. Each factor was evaluated at three coded levels—low (−1), medium (0), and high (+1) as commonly applied in response surface optimisation. The actual values with the corresponding coded levels and experiment matrix are summarised in Table 1 and Table 2, respectively. Based on these factor settings and the structure of the CCD, a total of 20 experimental runs were generated to allow estimation of pure error and assessment of model adequacy.

Table 1 *Experimental factors and levels in the central composite design*

Factors	Name	Units	Low (-1)	High (+1)
A	Oil concentration	v/v %	25	35
B	Adsorbent Dosage	g	6	24
C	Contact time	min	10	30

Table 2 *CCD matrix for the removal of lubricating oil by RH and SB adsorbent*

Run	Factors			Response
	A	B	C	%Removal
1	30	15	20	90
2	25	6	30	100
3	30	15	20	90
4	30	15	10	80
5	30	6	20	84
6	30	15	20	90
7	35	24	10	69
8	30	15	20	90
9	25	6	10	90
10	25	15	20	100
11	30	15	20	90
12	30	15	30	100
13	35	24	30	93
14	35	6	30	95
15	25	24	10	98
16	25	24	30	100
17	30	15	20	90
18	35	6	10	72
19	35	15	20	85
20	30	24	20	80

2.5 Results and Discussion

2.5.1 FTIR Analysis

The FTIR spectra of carbonised RH and SB is shown in Fig.1 (a) and (b), respectively, after NaOH activation and pyrolysis process. Each peak corresponds to a functional group that reflects the chemical composition and surface functionalities responsible for oil adsorption. For carbonised RH (Fig. 1a), the broad band at 2922 cm^{-1} corresponds to asymmetric and symmetric C–H stretching of $-CH_2$ groups [14]. A weaker band at 2337 cm^{-1} is commonly attributed to O=C=O stretching, which may arise from atmospheric CO_2 adsorption. The peak 1371 cm^{-1} represent C–H bending associated with cellulose and hemicellulose structures. This functional group is common in the organic compounds present in biomass from agricultural residues [15]. A strong absorption at 1051 cm^{-1} is associated with C–O stretching vibration in polysaccharides, especially cellulose and hemicellulose. This is supported by studies indicating similar peaks in FTIR spectra of various agro-wastes [16]. The band at 794 cm^{-1} corresponds to C–H deformation, indicating aromatic structures formed during carbonization. Meanwhile for carbonised SB a shown in Fig.1 (b), the broad band between 3659–3197 cm^{-1} is due to O–H stretching reflecting hydroxyl groups in cellulose, hemicellulose and lignin, and indicating extensive hydrogen bonding. The peak at 2922 cm^{-1} again corresponds to aliphatic $-CH_2-$ groups, which are major components of plant biomass [17]. The band at 1735 cm^{-1} corresponds to C=O stretching from hemicellulose acetyl groups and ester linkages in lignin. The peak at 1594 cm^{-1} represents aromatic C=C stretching, indicating structural components derived from lignin. The absorption at 1170 cm^{-1} is attributed to C-O and C-C stretching vibrations in polysaccharides. Both spectra confirm the presence of hydroxyl, carbonyl and aromatic functional groups typical of agro-waste biomass. The NaOh modification and carbonization process have created active sites that facilitate the adsorption of lubricating oil. These functional groups—particularly O–H, C–H, C=O and C–O—play an essential role in the interaction between the adsorbent surfaces and hydrophobic oil molecules.

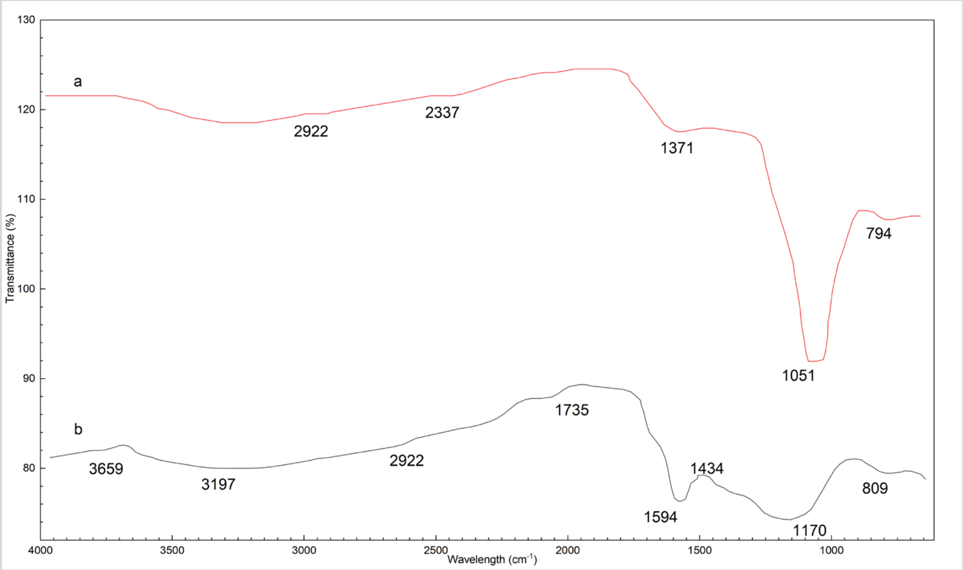

Fig. 1 *FTIR spectra of carbonised adsorbent. (a) RH; and (b) SB*

2.5.2 Statistical studies and optimization

In this study, the analysis of variance (ANOVA) (Table 2) indicates that the quadratic response surface model is highly significant, as evidenced by the large model F-value (39.27) and its associated p-value ($p < 0.0001$). This confirms that the model explains a substantial proportion of the lubricating-oil removal and is highly significant at the 95% confidence level. Significant linear effects were observed for initial oil concentration (A) and contact time (C), whereas adsorbent dosage (B) did not exhibit a meaningful linear contribution ($p=0.880$). This is because the adsorption process approaches site saturation, causing further increases in adsorbent dosage to not produce a proportional increase in removal efficiency and lead to a weak linear response [18]. The significant quadratic terms A^2 and B^2 reflect curvature in the system, and the AC interaction term further demonstrates that removal efficiency is jointly influenced by oil concentration and contact time. The lack-of-fit test was not significant ($p > 0.05$), indicating that the model adequately represents the experimental data. Strong goodness-of-fit was achieved, with $R^2 = 0.9725$, adjusted $R^2 = 0.9477$, and predicted $R^2 = 0.7092$, confirming good explanatory capacity and acceptable predictive performance. The close correspondence between predicted and experimental values supports the reliability of the quadratic model for optimisation and process prediction.

Table 2 *ANOVA for the response surface quadratic model*

Source	DF	Adj SS	Adj MS	F-Value	p-Value
Model	9	1472.54	163.615	39.27	<0.0001
A	1	547.60	547.600	131.44	<0.0001
B	1	0.10	0.100	0.02	0.880
C	1	624.10	624.100	149.80	<0.0001
A^2	1	52.36	52.364	12.57	0.005
B^2	1	103.55	103.551	24.86	0.001
C^2	1	9.55	9.551	2.29	0.161
AB	1	21.13	21.125	5.07	0.048
AC	1	153.13	153.125	36.75	<0.0001
BC	1	6.12	6.125	1.47	0.253
Error	10	41.66	4.166		
Lack-of-Fit	5	41.66	8.332		
Pure Error	5	0.00	0.000		
Total	19	1514.20			

R^2=0.9725; Adj R^2=0.9477; Pred R^2=0.7092

*DF is degrees of freedom; SS is the sum of squares, and MS is the mean of squares.

The following second-order polynomial equation (eq. 3) was developed to quantify the relationship between the removal percentage and the independent parameters influencing the oil adsorption process. The second-order model relates oil removal efficiency to three key factors: initial oil concentration (A), adsorbent dosage (B), and contact time (C). The equation includes linear terms describing the direct effect of each factor, quadratic terms capturing curvature and potential optima, and interaction terms showing how pairs of factors jointly influence the response. Together, these components provide a comprehensive predictive model for optimising removal performance within the studied range.

$$\begin{aligned} & Y\ (_{\%Removal}) = +\ 2 \\ & 98.9 - 13.16A + 3.539B - 2.435C + 0.1745A^2 - 0.0758B^2 + 0.0186C^2 \\ & \qquad - 0.0361AB + 0.0875\ AC - 0.00972\ BC \end{aligned} \tag{3}$$

The Pareto chart of standardized effects (α = 0.05) as shown in Fig. 1 demonstrates that contact time (C) and initial oil concentration (A) are the most influential variables governing oil removal efficiency, with both effects substantially exceeding the significance threshold (t = 2.23). A notable interaction between A and C (AC) further indicates that the influence of contact time is strongly dependent on the initial concentration of the solution. Significant curvature associated with the quadratic terms A^2 and B^2 confirms the nonlinear behaviour of these factors within the studied range. Conversely, adsorbent dosage (B), BC, and C^2 exhibit effects below the critical value, suggesting comparatively limited contributions to the overall model. Collectively, these results validate that removal efficiency is predominantly controlled by contact time and initial oil concentration, supported by their interaction and quadratic responses across the operating domain (25- 35 v/v%; 6–24 g/L; 10–30 min).

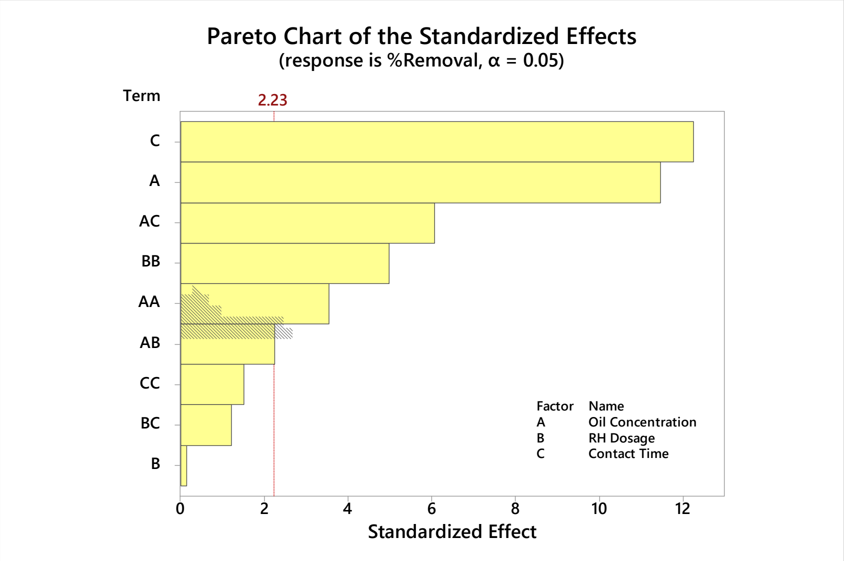

Fig. 2 *Pareto chart of standardized effects following response transformation*

Fig. 3 displays the two-dimensional contour plots illustrating the combined influence of oil concentration, RH dosage, and contact time on removal efficiency. The colour scale reflects the % removal, with darker blue regions indicating low performance (<80%) and darker green regions representing the highest efficiencies (>105%). In the RH dosage versus oil concentration plot, removal efficiency declines noticeably as oil concentration increases from 25 to 35 v/v%, particularly at low dosages. Higher RH dosages shift the contours toward the green region, confirming their strong positive effect on %Removal. A comparable pattern appears in the contact time–oil concentration plot, where higher oil concentration suppresses removal, while longer contact times enhance performance by promoting adsorption equilibrium. Meanwhile, the contact time versus RH dosage plot shows that moderate dosages (12–18 g/L) combined with extended contact times (25–30 min) yield the highest removal efficiencies (>100%), whereas low dosage–short time combinations correspond to the poorest outcomes (<85%). Collectively, the contour plots indicate that high RH dosage and adequate contact time, together with low oil concentration, provide the most favourable conditions for effective removal. These visual trends are consistent with the RSM model predictions and the statistically significant factor effects identified in the ANOVA.

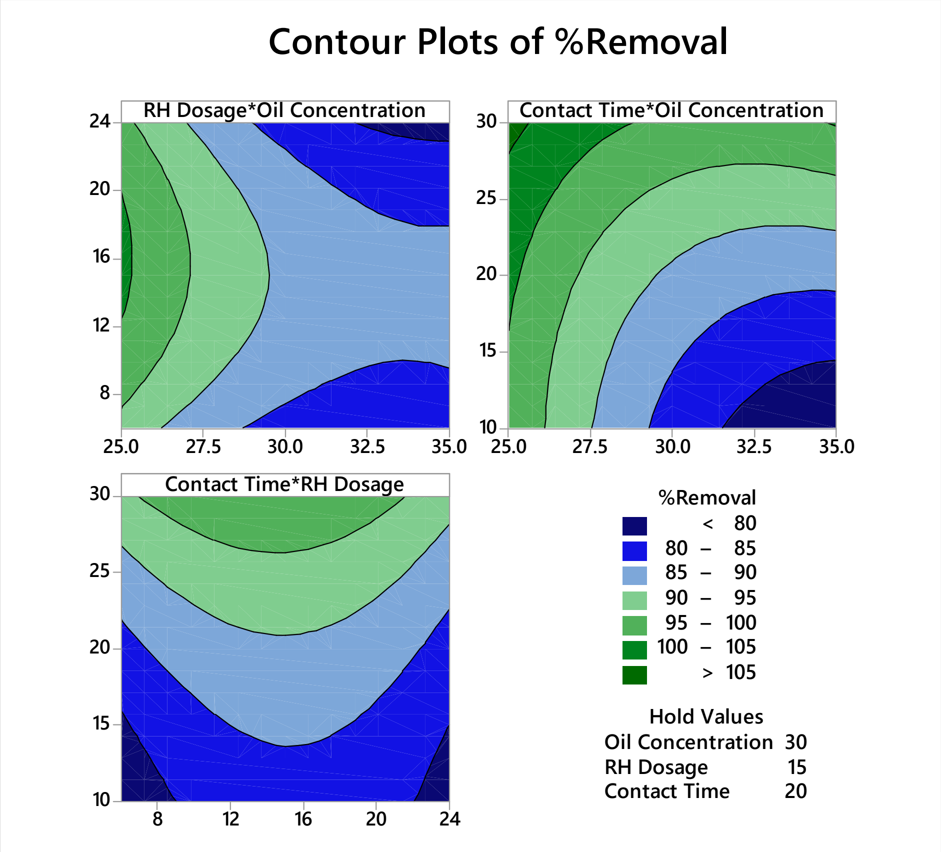

Fig. 3 *Response contour plots for the % removal of oil*

2.6 Conclusion

In this study, an abundant agro-waste of carbon-based adsorbents derived from rice husk (RH) and sugarcane bagasse (SB) were prepared as efficient adsorbents for the removal of used lubricating oil. The adsorbents were chemically treated with NaOH and carbonised to enhance surface properties favourable for oil adsorption. Characterisation using FTIR analysis confirmed the presence of lignocellulosic functional groups that support oil–adsorbent interactions. A Central Composite Design (CCD) integrated with Response Surface Methodology (RSM) was employed to evaluate the effects of key operating parameters, namely oil concentration, adsorbent composition, and contact time, and to determine the optimum removal conditions. The statistical analysis demonstrated that the RSM model adequately described the adsorption behaviour and effectively identified optimal operational settings. The highest removal efficiency was achieved using an RH:SB ratio of 15:15 g, an oil concentration of 25 v/v%, and a contact time of 30 min, resulting in complete oil removal under batch conditions. These findings confirm that carbonised RH–SB adsorbents are promising, sustainable, and efficient adsorbents, and further demonstrate the effectiveness of RSM as a powerful optimisation tool for lubricating-oil remal efficiency using RH-SB adsorbents.

Acknowledgement

Communication of this research is made possible through the facilities assistance by Universiti Tun Hussein Onn Malaysia.

Conflict of Interest

The authors declare that there are no conflicts of interest regarding the publication of this paper.

Author Contribution

The authors confirm their contribution to the paper: ***study conception and design:*** *Hasnida Harun, Nivethah Sokalinggam, Nurdalila Saji;* ***analysis and interpretation of results:*** *Inawati Othman, Noor Hasyimah Rosman, Nor Hazren Abdul Hamid, Nor Maizzaty Abdullah. All authors reviewed the results and approved the final version of the manuscript.*

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