

Article

Biosorption of Engine Oil Using Rice Husk in a Filtration System

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Abstract: Owing to its excellent qualities as a natural sorbent, rice husk (RH), a significant agricultural waste product obtained from the milling process, is employed as a biosorbent for engine oil. Engine oil spillages in rivers will flow to the ocean, exposing marine life to deadly contaminants. To date, there are very few natural sorbent studies specifically targeting engine oil removal. The purpose of this study was to optimise the significant factors in the efficiency of engine oil sorption by RH. Spectroscopic analyses using Fourier transform infrared spectroscopy (FTIR) and scanning electron microscopy (SEM) were performed to characterise the chemical composition and surface morphology of RH sorbent after pre- and post-treatment. A conventional optimisation approach, one-factor-at-a-time (OFAT), was used to assess the range of factors affecting the efficiency of engine oil sorption through heat treatment, heating time, packing density, and concentration of engine oil. The efficiency of engine oil removal obtained from this method was 74.5%. All the factors were assessed using a Plackett–Burman design (PBD) to eliminate non-significant factors. Furthermore, a central composite design (CCD) was employed to explore significant interactions among the factors. The quadratic model generated ($R^2 = 0.9723$) fitted the data well. The optimised conditions from the CCD were 160 °C, 20 min, 0.16 g/cm³, and 12.5% (*v/v*), with improved oil sorption from 74.5% (OFAT) to 78.89% (RSM).

Keywords: biosorption; agriculture waste; hydrocarbon



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1. Introduction

The purpose of engine oil, often known as motor oil, is to lubricate, protect, and cool internal combustion engines while they are operating. Modern engine oils are largely based on polyalphaolefin, which has poor lubricating qualities. Hence, a combination of additives is required for it to accomplish its necessary functions [1–3]. Engine oil is subjected to various factors that can lead to its deterioration, including high temperatures and chemically active particles that affect its quality and lead to it being discarded [4,5]. When disposed of improperly, the range of pollutants present in engine oil, including lead,

copper, arsenic, magnesium, chromium, zinc, cadmium, and polychlorinated biphenyls, can damage streams and groundwater [6–9]. Oil spills are usually caused by accidents that occur during the production and transportation of oil [10]. This phenomenon causes serious and long-term harm to marine ecosystems, the depletion of natural resources, and the disruption of the fragile ecological equilibrium [11–13]. A famous example is the oil spill catastrophe in Brazil in August 2019, which impacted almost 3000 km of the country's coastline and was the worst oil leak in Brazilian history as well as the worst environmental disaster ever recorded in the tropical coastal region [14].

In recent years, biosorption has emerged as a promising approach for the removal of oil contamination [15–17]. Adsorption is the process in which certain components of a fluid phase, known as solutes, are transferred selectively to the surface of an insoluble solid [18,19]. When an adsorbent is placed in contact with a fluid phase, molecules from the fluid phase diffuse to the surface of the adsorbent (including the pores if the adsorbent is porous), where they either form chemical bonds with the solid surface or are physically held there by weak van der Waals forces [20,21]. This method is preferred due to its high removal effectiveness, low starting costs, design simplicity, and lack of toxicity selectivity [22–24].

Biosorption relies on the utilisation of natural materials, including agricultural wastes, which possess intrinsic properties enabling them to selectively absorb and eliminate oil from water [25]. Sorbents are used for materials with a mechanism that is absorbent or adsorbent or both, existing in natural or synthetic form [26,27]. These liquid-absorbing materials have great potential to be employed as the only means of cleaning small spills and for the removal of oil vestiges in areas such as wetlands and beaches [28,29]. The ability of sorbent material to float and have a low density is one of its benefits, making it an excellent option for use in situations involving oil and derivative spills in aquatic environments [26]. Another advantage of sorbents is their high regeneration capacity, which allows for their in-place application and sorption, the subsequent discharge of recovered oil, and their reuse in contaminated environments [30,31].

Natural organic sorbents have received more attention than their synthetic and inorganic counterparts, although all three types of sorbents have been studied in depth during the past few decades [32–34]. Kenaf fibre, cotton fibre, sawdust, wood fibre, corn cob, rice straw, kapok fibre, wool fibre, milkweed, coconut husk, rice husk (RH), oil palm empty fruit bunch (EFB), cattail fibre, hay, feathers, Guinea grass, and bagasse are examples of organic sorbents [29,35–37]. Among the diverse range of biosorbents available, rice husk (RH) has garnered significant attention owing to its abundance, renewability, and low cost [38]. RH is the protective covering on rice grains that is typically removed during the dehulling process, generating substantial quantities of waste material [39]. RH exhibits several characteristics that render it a promising sorbent material for engine oil remediation. RH is composed of organic substances (75% cellulose, hemicellulose, and lignin), as well as silica (15%) [40]. Several variables influence the composition of RH and rice husk ash (RHA), including agricultural practices (the type and amount of fertiliser applied) and geographical or meteorological conditions. These factors are the reason for the disposal of RH [41,42].

Materials derived from natural sources are found to be less costly, more abundant, and not as harmful to the environment, which makes them a more attractive option for effective waste disposal [36,42,43]. However, their primary drawback is that they have weak oleophilic/hydrophobic characteristics [42]. One way to enhance the oil sorption capability of natural sorbents is by using thermal treatment methods. Low-temperature thermal treatment, such as drying, has little effect on the sorbent's oleophilic and hydrophobic characteristics. Its major action is the eliminating of certain surface contaminants, allowing for faster oil absorption [44]. A high-temperature thermal treatment, such as pyrolysis, leads to the carbonisation of the sorbent [45]. As a result, oil sorption capacity and oil-to-water selectivity can be greatly improved.

Natural sorbent materials that have been ground have a high oil sorption capacity per unit mass as the contact surface of the material is more accessible and the binding sites on smaller particles are more available [42]. Some natural sorbents have equivalent

or even higher oil sorption capacities than synthetic sorbents, but they also absorb more water, which results in lower oil sorption capacities [35]. As a result, it is critical to ensure that the natural sorbent is extremely hydrophobic or lipophilic to retain high selectivity of oil over water. Moreover, natural organic sorbent-based devices have been proposed for use in small-scale oil spills on calm water surfaces [38]. A unique natural oil sorbent, non-woven cotton wadding has been claimed to have a significantly superior oil sorption capacity than synthetic products and is ecologically safe and straightforward to deploy in practical applications [29].

However, in order to harness the biosorption capabilities of RH and optimise its performance as a sorbent material for engine oil, it is crucial to gain a deeper understanding of the underlying mechanism and key factors influencing the biosorption process. Factors such as the composition and structure of RH, contact time, pH, temperature, and oil concentration influence the efficiency of the biosorption process. An in-depth investigation of these parameters and their interplay is essential for developing effective strategies to maximise the biosorption capacity of RH and enhance its suitability for practical applications.

Therefore, the aim of this study is to explore the biosorption of rice husk as a sustainable and efficient method for mitigating engine oil spills. Through the examination of the underlying mechanism and optimisation of the key parameters (temperature, time of heating, packing density, and engine oil concentrations) involved in the biosorption process, valuable insights can be obtained to guide the development of innovative and environmentally friendly approaches to oil spill clean-up. The outcomes of this study are anticipated to make significant contributions to the broader field of environmental remediation and offer practical solutions for addressing the challenges associated with engine oil spills.

2. Materials and Methods

2.1. Materials

The RH and biocharcoal RH used in this study were acquired from Kampung Agong, Penaga, Pulau Pinang, Malaysia. Meanwhile, engine oil was acquired at a workshop in Serdang, Selangor, while 70 L of distilled water (dH₂O) was obtained from the laboratory and held at room temperature (25 °C ± 1 °C) until usage.

2.2. Filtration Setup

The combined components were placed in a filter system modified by Taufik et al. [29]. A 400 mL plastic bottle, a cylinder holder, and a 500 mL measuring cylinder were acquired. The latter was inverted onto the retort stands, and a wire mesh net was formed into a cylinder holder. The 500 mL measuring cylinder was used to collect the effluents by being placed beneath the bottle.

2.3. Sorption Capacity and Efficiency Evaluations

RH (15.71 g) was put into the cylinder holder, and 40 mL engine oil was mixed with 400 mL of dH₂O. Then, the engine oil–water mixture was poured into the cylindrical container filled with RH and left for 10 min. The final sample weight and effluent were recorded.

Sorption capacity was evaluated in the laboratory (25 °C ± 1 °C) using the F 726-99 technique. The oil sorption capacity was used to determine the sorption capacity values (Equation (1)).

$$\text{Oil sorption capacity (g/g)} = \frac{X_f - X_i}{X_i} \quad (1)$$

where X_f denotes the total mass (g) of the wet sample after immersion and X_i denotes the mass (g) of the sample prior to immersion. The efficiency of the engine oil absorption was determined using Equation (2).

$$\text{Efficiency of engine absorption (\%)} = \frac{E_i - E}{E_i} \quad (2)$$

where E_i is the initial (v/v) concentration of engine oil and E is the concentration of engine oil after attaining equilibrium. The efficiency of the water absorption was determined using Equation (3).

$$\text{Efficiency of water absorption(\%)} = \frac{W_i - W}{W_i} \quad (3)$$

where W_i is the dH_2O at the beginning of the experiment, while W is the dH_2O after attaining equilibrium.

2.4. Screening of the Different RH

The untreated, treated (heated at 160 °C for 20 min in a laboratory forced convection oven (Taisite, New York, NY, USA) with a temperature precision of ± 1 °C), and biocharcoal RH samples were packaged (15.71 g) and placed in the cylinder holder to be examined.

2.5. Identification of RH

2.5.1. Fourier Transform Reflectance Infrared Spectroscopy (FTIR)

The chemical structures of the untreated, treated, and biocharcoal RH (before and after soaking with the oil–water combination) were studied using FTIR (Bruker Optik GmbH, Ettlingen, Germany). The FTIR spectra of each sample were analysed and obtained in the range from 4000 to 400 cm^{-1} .

2.5.2. Scanning Electron Microscopy (SEM)

In order to investigate the components that make up RH, a variable pressure scanning electron microscope (VPSEM) (Carl Zeiss AG, Jena, Germany) was utilised. The RH samples had a gold sputter coating applied to them, and then they were secured to circular sample holders made of stainless steel using double-sided conductive adhesive tape. An accelerating voltage of 15 kV was utilised in order to analyse the SEM pictures.

2.6. Optimisation of Engine Oil Biosorption

2.6.1. One-Factor-at-a-Time (OFAT) Approach

The optimisation and assessment of the biosorption of engine oil into RH were carried out via the one-factor-at-a-time (OFAT) approach based on four selected parameters, namely, heat treatment (130, 140, 150, 160, 170, and 180 °C), time of heating (10, 20, 30, 40, 50, and 60 min), packing density (0.12, 0.14, 0.16, 0.18, and 0.20 g/cm^3) and engine oil concentration (5, 10, 15, 20, and 25% (v/v)). The effects of these parameters were investigated by maintaining all the other factors at constant conditions except for the one factor of interest.

2.6.2. Response Surface Methodology (RSM)

The RSM-based optimisation employed two statistical mathematics designs: Plackett–Burman design (PBD) and central composite design (CCD). With minor modifications, the factors matched from OFAT were further analysed using Design-Expert software version 13.0 under the Factorial Miscellaneous Design (standard design). The significant parameters identified using the PBD were then further optimised using a CCD [46]. Experiments were conducted to further validate the statistical models, and the optimal values predicted by the models were used in the experiments. $p < 0.05$ was considered significant in all of the analyses. In the CCD, Equation (4) of the quadratic mathematical model of CCD design was employed:

$$Y = \beta_0 + \sum_{i=1}^k \beta_i x_i + \sum_{i=1}^k \beta_{ii} x_i^2 + \sum_{1=i < j}^k \beta_{ij} x_i x_j \quad (4)$$

where Y is the response variable, x is the independent factors that influence y , β_0 is the intercept, β_i is the i th linear coefficient, β_{ii} is the quadratic coefficient, β_{ij} is the coefficient of interaction effect, and k is the number of involved factors.

2.7. Statistical Analysis

All experiments were performed in triplicate, and the mean value and standard error of the mean were computed. The experiments that were conducted were analysed using GraphPad Prism. Statistical analysis was performed using ANOVA and Tukey's test.

3. Results and Discussion

3.1. Screening of Rice Husk Samples

The screening procedure included three groups of untreated, treated (160 °C), and biocharcoal RH samples (Figure 1). Values are means \pm the standard error of the mean ($n = 3$). The treated RH samples displayed the highest level of sorption capacity ($2.7 \text{ g/g} \pm 0.07$) and the most efficient oil absorption ($48.95\% \pm 2.69$). Therefore, the treated RH was selected for further experiments. Meanwhile, water absorption by the treated RH was lower ($4.28\% \pm 0.40$) compared to the other two, which were untreated ($8.68\% \pm 0.57$) and biocharcoal ($9.71\% \pm 0.71$). Due to the preferred lesser water absorption, the selection of treated RH was further proven [29].

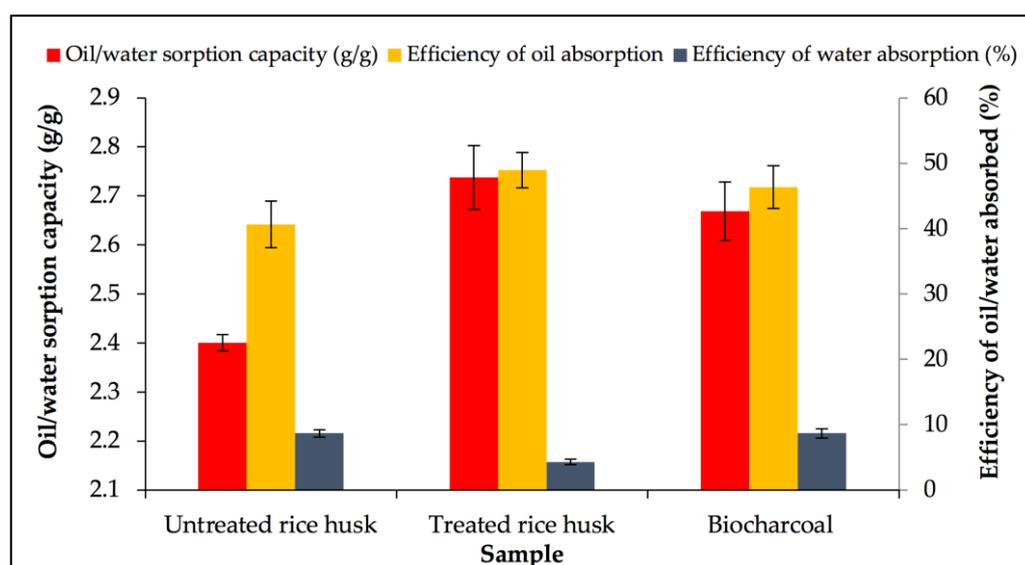


Figure 1. Screening of untreated and treated RH samples and biocharcoal sample.

Thermal treatment is by far the most common kind of electrical discharge and is widely used as it can alter the material's physical qualities while still preserving the chemical components of the fibre [21]. Drying out the lignocellulose fibre bundles causes the bundles to split into single filaments when the plant fibre is heated to temperatures between 100 and 200 °C for varying amounts of time. Other non cellulose or chemical compounds with lower glass transition temperatures or constituents that are comparable to lignin will either be released from the fibre bundles or depolymerised from them [47].

3.2. Characterisation of RH

3.2.1. Spectroscopic Analysis Using Fourier Transform Infrared Spectroscopy (FTIR)

The use of FTIR in this experiment is to gain a better understanding of the mobility profile of functional groups within the macromolecules that are present in RH. A spectroscopy investigation of RH revealed that ionisable functional groups such as hydroxyl, amino, and carboxyl groups can combine and chelate with engine oil [48].

Figures 2 and 3 display the infrared spectra of the untreated, treated, and biocharcoal RH both before and after the engine oil was absorbed into them. Prior to the adsorption process, the groups Si-O-Si, Si-H, and Si-O corresponded to some of the peaks in the spectrum. The peak with the highest intensity can be seen in the band at 1026.78 cm^{-1} , which corresponded to the stretching vibrations of the siloxane group (Si-O-Si), whereas

the peak that can be seen at 788.62 cm^{-1} corresponded to the stretching vibration of Si-H from the silane group. Both peaks can be seen in the spectrum. The presence of a significant amount of SiO_2 in the RH was responsible for the observation of a peak at a frequency of 553.32 cm^{-1} that corresponded to Si-O.

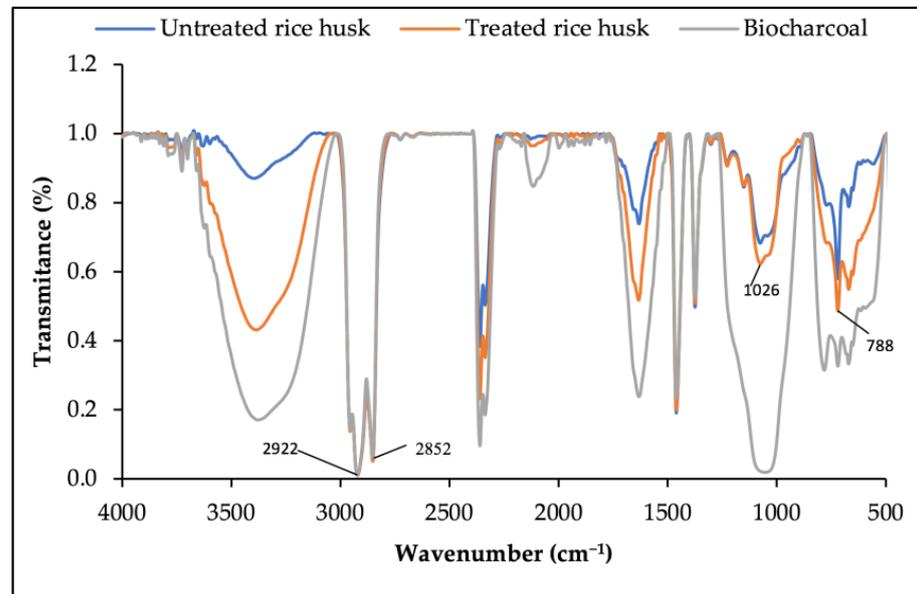


Figure 2. FTIR spectroscopy of untreated, treated, and biocharcoal RH before wetting with engine oil–water mixture.

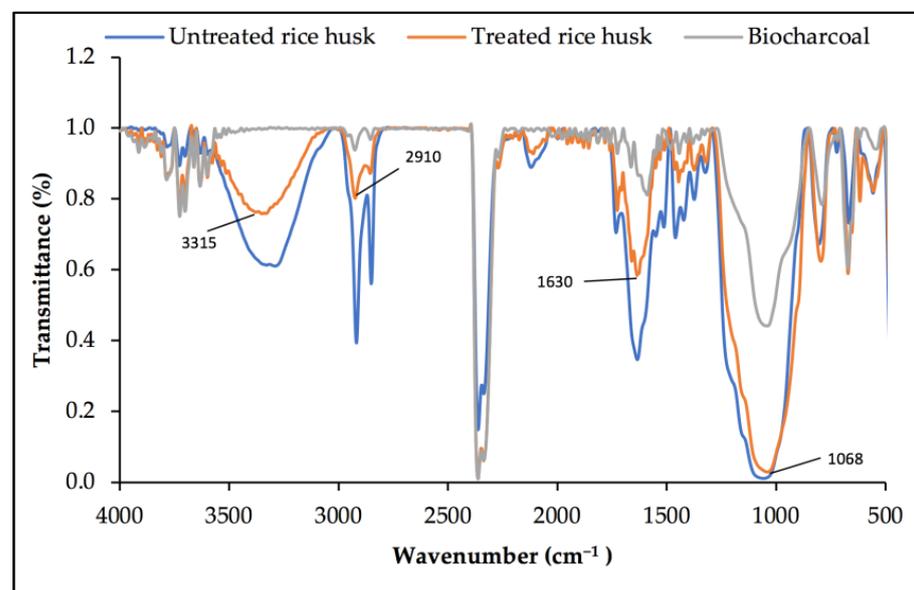


Figure 3. FTIR spectroscopy of untreated, treated, and biocharcoal RH after wetting with engine oil–water mixture.

The presence of silicon groups on the surface of the RHA is consistent with the findings reported in the literature, which suggested that silicon makes up 95% of the composition of RHA [21]. However, the removal of the silica bond vibration around 1068.13 cm^{-1} (Figure 3) showed that it may be possible to partially break down silica by the use of heat treatment at $160\text{ }^\circ\text{C}$ [21].

It was determined that the O-H stretching of the silanol group (Si-OH) was responsible for the FTIR band seen in RH at $3000\text{--}3600\text{ cm}^{-1}$ [49]. The hydroxyl groups (OH) found

on the surface of cellulose fibres give hydrophilicity, which might limit the application of cellulose fibres for cleaning oil from water. Cellulose fibres, however, can successfully absorb oil [29]. It may be possible to modify the surface of the sorbent to make it less hydrophilic by exchanging its OH groups [50]. Several different chemical processes may be used to attach functional groups of various substances to hydroxyl groups located on the cellulose chain (in the β -D glucopyranose).

The CO stretching vibration, which is typical of unconjugated ketones, aldehydes, and carboxyl, was determined to be the cause of the absorption seen for RH at a wavelength of 1639.99 cm^{-1} [49,51]. The peaks at 2922 cm^{-1} and 2852 cm^{-1} (Figure 2) vanished when the heating temperature was raised. High-temperature inputs can lead to the degradation of polysaccharides as well as the inclination of energy inputs. This implies the development of CO_2 at increasing temperatures and leftover methylene group at peaks between 2935 and 2915 that may disintegrate [49].

3.2.2. Surface Morphology Characterisation by Scanning Electron Microscopy (SEM)

The rough surface of RH could be seen in all of its intricate texture in the SEM micrographs. The SEM shows what kinds of molecules were on the surface of the RH. Figure 4a,b show that untreated RH has a greater number of silica grooves than treated and biocharcoal RH (Figure 4a). In the treated and biocharcoal RH, there was a significant increase in the number of pores (Figure 5b,c). Under low magnification ($500\times$), the traits that were seen were still evident.

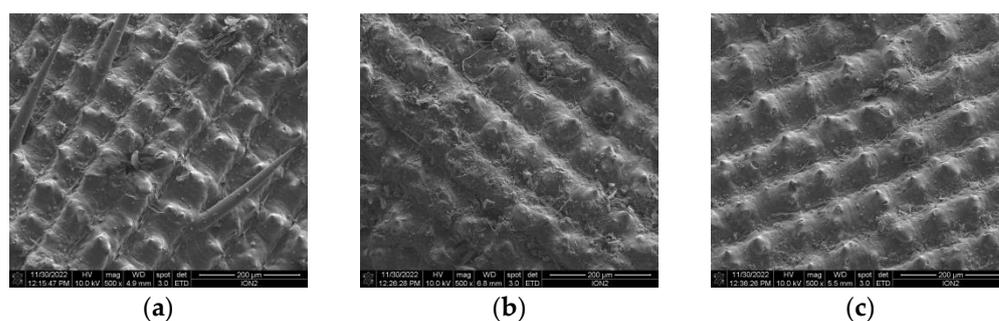


Figure 4. Images from SEM: (a–c) untreated, treated, and biocharcoal RH before wetting with engine oil–water mixture at $500\times$ magnification.

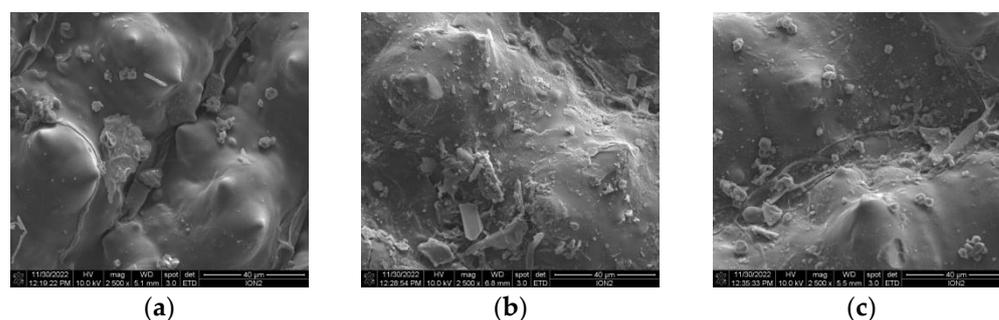


Figure 5. Images from SEM: (a–c) untreated, treated, and biocharcoal RH before wetting with engine oil–water mixture at $2500\times$ magnification.

The porous structure of adsorbents, in addition to the physical and chemical interactions of their functional groups with the components of engine oil, is what determines an adsorbent's capacity for high petroleum sorption [52]. It is clear from the SEM pictures that subjecting the RH samples to heat treatment results in the production of a structure that is vastly different and has a larger porosity than before (Figure 4b).

The untreated and biocharcoal RH surfaces differ from the treated RH surface area. On the surface of the treated RH's outer wall can be seen a great number of button-like

structures with tiny pores (Figure 5b). These structures did not exist in the untreated RH particles. The particles suffered significant transformations during this high-temperature treatment. Pores became larger in quantity as well as in size, new types of pores emerged, smaller pores merged into larger ones, and changes occurred in the surface of the pores as well as in the volume of the pores [53].

3.3. Optimisation of Factors for Oil/Water Sorption Capacity Using OFAT

Figure 6 depicts the performance of RH following treatment at temperatures ranging from 130 to 180 °C. Based on the findings, 160 °C was the best treatment temperature, with a maximum sorption capacity of (3.30 g/g \pm 0.067) and an engine oil absorption efficiency of 65.61% \pm 0.71. ANOVA was significant ($F_{6,14} = 15.12, p < 0.0001$), while Tukey's post hoc pairwise comparisons indicated that all points in the range were substantially different from the untreated RH. In general, the features of RH are dependent on several parameters. These elements include the geological location, rice variety, climatic variation, cultivation methods, and fertilisers used in paddy growth [54].

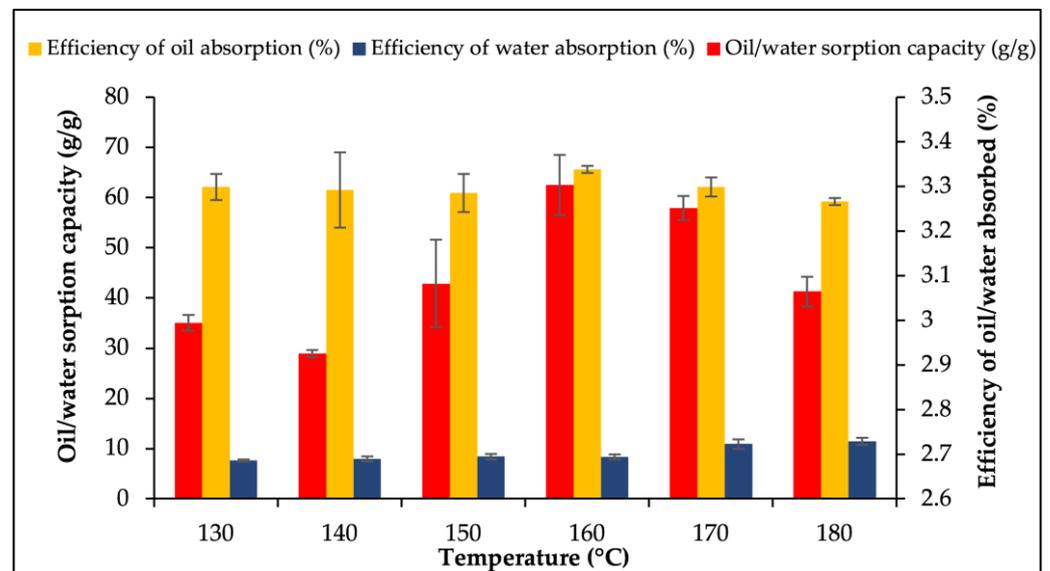


Figure 6. The effects of temperature on sorption and efficiency of engine oil–water absorbed by RH.

Figure 7 displays the oil/water sorption capacity and absorption efficiency of the engine oil–water mixture for a time of heating for RH ranging from 10 to 60 min. When the heating time was extended, the efficiency of oil/water was improved due change in its fibre properties [41,55]. However, there was a significant drop when the temperature reached 50 min. The greatest amount of water that could be absorbed was demonstrated after 60 min of heating. The optimal amount of time for heating was found to be 40 min, which resulted in the highest oil/water sorption capacity (3.33 g/g \pm 0.03) and efficiency of engine oil absorbed (72.03% \pm 1.24) and yielded the lowest amount of water absorbed (8.17% \pm 0.44). There was a statistically significant difference between RH ($p = 0.0011$) and engine oil sorption effectiveness ($p = 0.0002$) when compared with a heating time of 10 min. The results of the overall ANOVA revealed that there were significant variations in the amount of time it took to heat the RH ($F_{5,12} = 13.15, p = 0.0041$), the amount of engine oil efficiency ($F_{5,12} = 10.30, p = 0.0002$), and the amount of water sorption ($F_{5,12} = 6.313, p = 0.0213$).

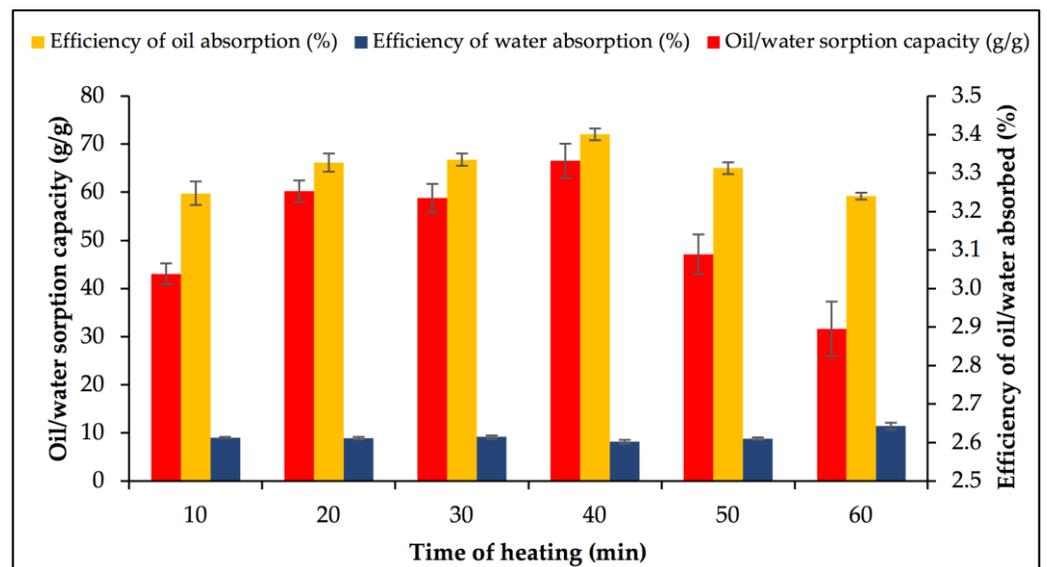


Figure 7. The effects of time of heating on the sorption capacity and efficiency of oil/water absorption by RH treated at 160 °C.

After that, a variety of various packing densities ranging from 0.12 to 0.2 g/cm³ were tested, while the RH treatment temperature and length of heating remained unchanged (Figure 8). At a low packing density, the surface area was increased because it correlated with the increased porosity that gave the rapid oil sorption results [33,56]. The results collected indicated that the packing density of 0.16 g/cm³ was the most effective one. This packing density offered the largest sorption capacity (3.33 g/g ± 0.04) and efficiency of engine oil absorption (66.78% ± 1.23) while also offering the lowest water absorption (8.5% ± 0.35). In this particular instance, however, an analysis of variance found no statistically significant differences between the treatments ($F_{4, 10} = 1.112, p = 0.2143$).

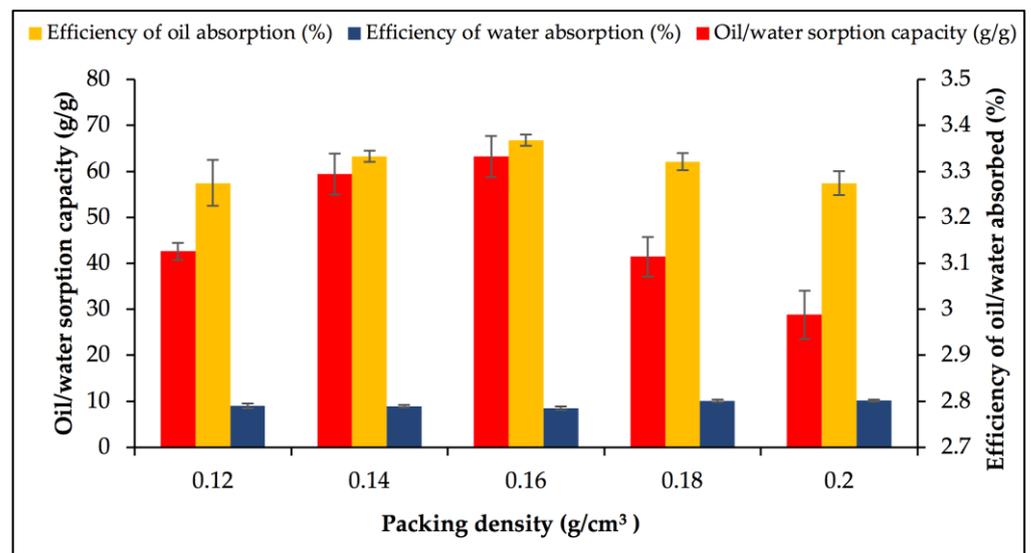


Figure 8. The effects of packing density on the sorption capacity and efficiency of oil/water absorption by RH treated at 160 °C and for 60 min.

The last factor examined using OFAT was engine oil concentration. As shown in Figure 9, engine oil concentration ranging from 5% to 25% was carried out, while the other factors were fixed. The oil/water sorption capacity continued to increase, despite the fact that the efficiency of engine oil absorption (%) was decreasing from 10%. Puasa et al. [34]

state that the equilibrium established when the reactive sites of sorbents are saturated with oil molecules, which encourage desorption, causes the efficiency of engine oil adsorption by RH to decline once the engine oil concentration approaches 10%. This is the reason that 10% was chosen as the best engine oil concentration, giving the highest efficiency of engine oil (71.67 ± 1.02) and the lowest water absorption (8.17 ± 0.44) compared to other concentrations. An analysis of variance (ANOVA) showed significant variations between the treatments that were shown to be impacted by the starting engine oil concentration in terms of the sorption capacity ($F_{5,12} = 16.88, p < 0.0001$) and the efficiency of engine oil absorbed (%) ($F_{5,12} = 29.77, p < 0.0001$). On the other hand, there was not a discernible change in the efficiency with which water was absorbed ($F_{5,12} = 1.512, p = 0.4375$).

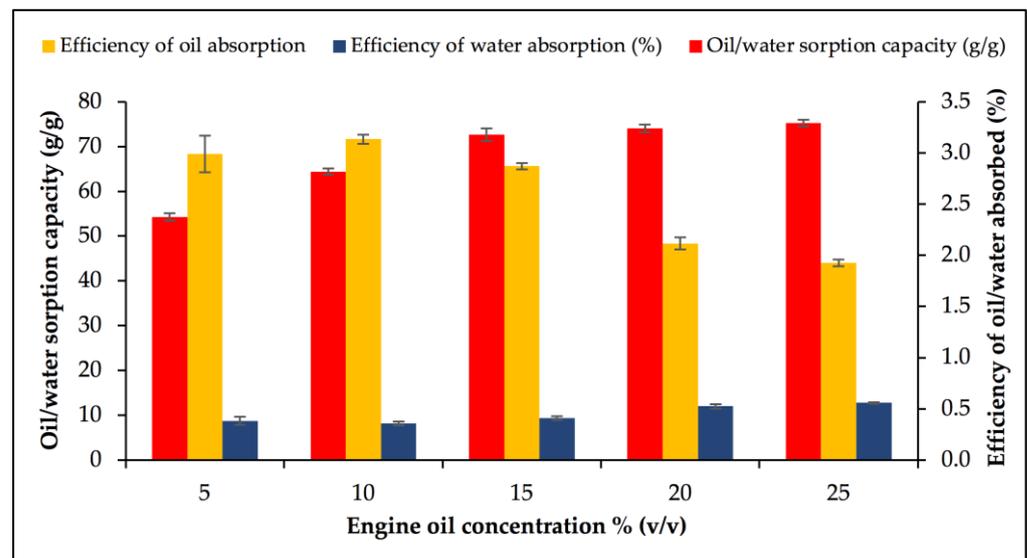


Figure 9. The effects of engine oil concentration and efficiency of oil/water absorption by RH treated at $160\text{ }^{\circ}\text{C}$ for 60 min and packed at 0.16 g/cm^3 .

3.4. Response Surface Methodology (RSM) Optimisation

3.4.1. Optimisation of Factors by PBD

The PBD was used in the secondary analysis to exclude non-significant components following the OFAT analyses. With all factors aligned, 18 runs were generated (Table 1). The lowest experimental values found for engine oil–water sorption was achieved during run number 9, and they were 50 mL (45%) and 37 mL (8.67%), respectively.

Table 1. Secondary screening of significant parameters affecting engine oil sorption using Plackett–Burman design (PBD).

Run	A	B	C	D	Response 1 Oil Efficiency (%)	Response 2 Water Efficiency (%)
1	180	30	0.12	5.0	81.67	9.17
2	165	45	0.16	12.5	76.67	9.33
3	165	45	0.16	12.5	76.00	8.83
4	180	60	0.20	5.0	97.33	10.08
5	150	60	0.20	20.0	62.50	9.25
6	150	30	0.20	5.0	98.50	9.75
7	165	45	0.16	12.5	74.67	8.42
8	180	60	0.12	5.0	96.50	9.33
9	150	30	0.12	20.0	45.00	8.67
10	165	45	0.16	12.5	76.67	9.41

Table 1. *Cont.*

Run	A	B	C	D	Response 1 Oil Efficiency (%)	Response 2 Water Efficiency (%)
11	150	60	0.12	20.0	49.58	9.25
12	180	30	0.20	20.0	62.50	9.17
13	150	30	0.12	5.0	81.67	7.58
14	165	45	0.16	12.5	77.33	9.47
15	180	60	0.12	20.0	54.58	10.92
16	165	45	0.16	12.5	80.00	9.58
17	150	60	0.20	5.0	98.67	9.25
18	180	30	0.20	20.0	66.67	9.50

A: temperature (°C); B: time of heating (min); C: packing density (g/cm³); D: oil concentration % (v/v).

The assessments for PBD go beyond the assessment that OFAT offers and also consider the likelihood of interactions going in both directions between the various factors [29]. The results of the ANOVA showed that the constructed model was very significant in general, with an $R^2 = 0.9699$, and the packing density and the engine oil concentration were all relevant components (Table 2). These aspects were thus taken into consideration in the CCD analysis.

Table 2. ANOVA analysis for the PBD model to evaluate the significant factors affecting engine oil sorption by RH.

Source	Sum of Squares	DF	Mean Square	F-Value	p-Value	
Model	4384.84	4	1096.21	96.73	<0.0001	significant
A: Temperature	45.37	1	45.37	4.00	0.0685	
B: Time	44.72	1	44.72	3.95	0.0703	
C: Packing density	496.22	1	496.22	43.79	<0.0001	
D: Engine oil concentration	3798.52	1	3798.52	335.20	<0.0001	
Curvature	21.01	1	21.01	1.85	0.1984	
Residual	135.99	12	11.33			
Lack of Fit	111.60	6	18.60	4.58	0.0433	significant
Pure Error	24.38	6	4.06			
Cor Total	4541.83	17				
Std. Dev.	3.37			R^2	0.9699	
Mean	75.36			Adjusted R^2	0.9599	
C.V.%	4.47			Predicted R^2	0.9168	
				Adeq precision	20.125	

3.4.2. Interactions of Significant Factors Analysed Using CCD

The significant parameters determined using PBD were then subjected to additional analysis with the CCD to determine the optimal and interactive conditions that existed between the factors. After generating a total of 13 experimental runs based on two significant factors, an optimisation process using a five-level CCD approach was performed on the data. The experimental variables, along with the experimental and predicted values of engine oil–water mixture for the oil/water efficiency of RH, are presented in Table 3.

A quadratic model was adopted, and ANOVA was performed to examine the importance of each model term (Table 4). The significance of the quadratic model was determined to be extremely high ($p < 0.0001$), and its R^2 value was found to be 0.9723.

Table 3. Secondary screening of significant parameters affecting engine oil sorption using a CCD.

Run	A	B	Response 1 Oil Efficiency (mL)	Response 2 Water Efficiency (mL)
1	0.12	5.00	90.00	7.92
2	0.16	12.50	75.33	9.00
3	0.16	12.50	78.00	8.92
4	0.16	12.50	78.00	8.92
5	0.12	20.00	46.25	7.67
6	0.20	20.00	58.33	8.83
7	0.20	5.00	99.83	9.50
8	0.16	1.89	100.00	8.25
9	0.16	12.50	78.00	8.67
10	0.16	12.50	73.33	8.83
11	0.22	12.50	91.33	9.42
12	0.10	12.50	66.67	8.67
13	0.16	23.11	50.96	9.42

A: Packing density (g/cm^3); B: engine oil concentration % (v/v).

Table 4. ANOVA analysis for the CCD model to evaluate the significant factors affecting engine oil sorption by RH.

Source	Sum of Squares	DF	Mean Square	F-Value	p-Value	
Model	3408.87	5	681.77	49.14	<0.0001	significant
A-Packing density	403.29	1	403.29	29.07	0.0010	
B-Engine oil concentration	2988.10	1	2988.10	215.36	<0.0001	not significant
AB	1.27	1	1.27	0.0912	0.7714	
A ²	0.7337	1	0.7337	0.0529	0.8247	
B ²	14.36	1	14.36	1.04	0.3429	
Residual	97.12	7	13.87			
Lack of Fit	78.99	3	26.33	5.81	0.0611	
Pure Error	18.13	4	4.53			
Cor Total	3505.99	12				
Std. Dev.	3.72			R ²		0.9723
Mean	75.85			Adjusted R ²		0.9525
C.V.%	4.91			Predicted R ²		0.8317
				Adeq precision		21.7175

Although, the ANOVA did not find any significant pairwise interactions between any of the components (Table 4), two response surface contour plots were created. The packing density and the engine oil concentration were the relevant considerations, which were denoted by the letters A and B, respectively. The interaction between packing density (g/cm^3) and engine oil concentration is depicted in Figure 10. At 0.16 g/cm^3 and 12.5% (v/v) engine oil it was expected that there would be the most oil absorption (76.09%). However, the surfaces of the two plots are relatively flat rather than domed, which is consistent with the fact that substantial interactions were not found and that these two factors worked independently.

3.4.3. RSM Model Validation

Employing OFAT, the best conditions for sorption capacity and oil/water absorption were attained by using treated RH at 160 °C for 40 min, packed at 0.16 g/cm^3 packing density, and exposed to 10% (v/v) engine oil. From these circumstances, 49 mL of engine oil and 30 mL of water were successfully captured (Table 5).

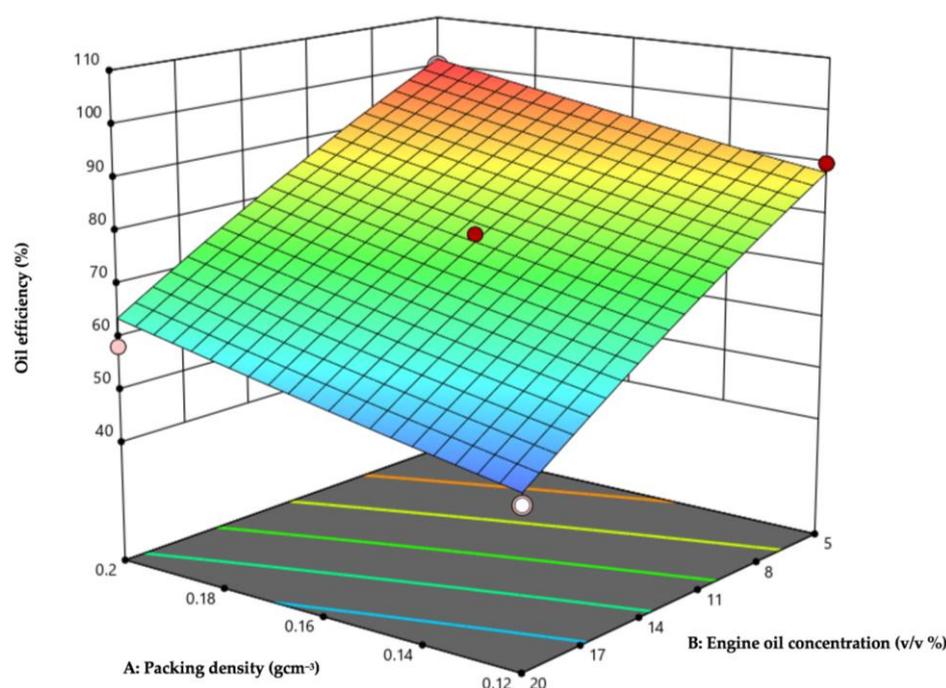


Figure 10. Three-dimensional contour plot made through Design-Expert 13.0 (Stat-Ease, Inc., Minneapolis, MN, USA). A: packing density and B: engine oil concentration.

Table 5. Model validation for using the optimum condition.

Optimised Factors	Value	Predicted Value	Experimental Value
Packing density	0.16 g/cm ³	49 mL of engine oil with 30 mL of water *	49 mL of engine oil with 30 mL of water ±1 mL *
Engine oil concentration	12.5% (v/v)		

* The *p*-value that is statistically significant between predicted and experimental values (*p* = 0.9999).

The effectiveness of oil absorption was 74.5% with a margin of error of 0.2. Using the PBD and the CCD techniques, the conditions advised were to treat RH at a temperature of 160 °C for 20 min, pack it at 0.148 g/cm³, and utilise engine oil at 12.5% (v/v). Under these conditions, the efficiency of oil absorption was 79.89% with a margin error of 0.5, which represented a marginal improvement over the use of OFAT on its own. In other words, both studies relied on the pure components working separately, and as a consequence, the near similarity in the results that were produced should not come as a surprise, as it is consistent with the fact that the interaction terms were found to be non-significant.

4. Conclusions

The biosorption of engine oil using RH offers several advantages over conventional oil spill clean-up methods, including cost-effectiveness, eco-friendliness, and the utilisation of a renewable and abundant resource. By utilising RH as a biosorbent, the negative impacts of oil spills on aquatic ecosystems and human health can be mitigated while minimising the depletion of natural resources and maintaining ecological balance. The findings of this study emphasise the potential of RH as a valuable tool in the field of oil spill remediation. Screening results indicated that treated RH outperformed the untreated and biocharcoal RH with a sorption capacity of 2.7 g/g ± 0.07 and oil absorption of 48.95% ± 2.69. The identification and molecular morphology before and after the RH was wetted with oil were also assessed, which further suggested that treated RH is better for absorption. The application of a statistical approach (RSM) for optimisation led to a significantly higher oil sorption efficiency of 78.89%, surpassing the conventional approach (OFAT) with an oil sorption efficiency of 74.5%.

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