Utilisation of Oil Palm’s Empty Fruit Bunch Spikelets for Oil-Spill Removal

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Abstract: Agricultural sorbents have received attention for their effectiveness in oil removal. In Malaysia, oil palm’s empty fruit bunch (EFB) spikelets are an abundant agricultural waste that provides a non-toxic, renewable resource of cellulosic materials. In this study, the effectiveness of EFB spikelets to remove oil spills from seawater pollution in a filter system was investigated and the best optimisation approach for filtering conditions was determined. Experiments for oil spill clean-up were performed using a filter-based oil sorption system with a series of conditions such as temperature, time, packing density, and oil concentration to evaluate sorption capacity, oil and water absorbed efficiency. Fourier transform infrared spectroscopy (FTIR) was used to characterise the physicochemical properties of untreated and treated EFB fibres. Based on one-factor-at-a-time (OFAT) analysis conducted at 160 °C for 30 min on 0.1 g/cm³ of packing density containing 25% diesel, 8.667 mL of oil and 5 mL of water was absorbed. In response surface methodology (RSM), the three parameters of temperature, packing density and diesel concentration were observed as significant. From RSM fitting model analysis, the predicted value obtained for both oil and water absorbed were 8.805 and 5.213 mL, respectively. The experimental RSM values of 9 and 5 mL of oil and water absorbed were obtained. The result demonstrated the validity of the model as the experimental RSM values were close to the RSM model’s prediction. As compared to OFAT, the RSM method is more efficient in oil removal. This research contributes to a better knowledge of the usage of a natural sorbent as a method of diesel pollution remediation.

Keywords: agriculture waste; oil spills; spikelets; absorbed; treated
1. Introduction

Diesel pollution poses a major risk as it can cause serious damage to the aquatic environment through exposure to anthropogenic pollutants. Numerous anthropogenic sources contribute to the diesel pollution in marine seawater, namely accidental oil spills from marine vessels and leakages from the transportation of supertankers [1]. The oil leakages have a huge impact on the local environment, including economic and human health effects. According to Nikkhah et al. [2], between 2010 and 2013, 22,000 tonnes of oil were estimated to have been spilt into marine environments, with petroleum fuels accounting for 48% of the total. When diesel fuel is transported and stored in bulk volumes, accidental spills and leaks can significantly raise the risk of contamination [1]. In 2010, three 200-litre drums of diesel fuel were dumped from a helicopter hauling supplies from Davis Station to preserve flight stability. As a result, the oil barrels burst, contaminating the sandy soil around Lake Dingle. It took nearly four years to clean up a total of 168 tonnes of toxic soil [3]. Other than that, a comprehensive assessment of oil spill episodes with detrimental effects on the marine environment was published by Lim et al. [4].

The long-term effects of oil spills on the environment and ecosystem demand an efficient clean-up in the polluted areas. To remove oil pollution contaminants from the polluted sites, physical, chemical and biological remediation methods can be implemented. Natural organic sorbents derived from agricultural biomass have recently attracted interest due to their efficacy in removing oil from water [1,5–7]. The freely available, inexpensive, effective, non-toxic and almost 100% biodegradable EFB waste makes it a preferable sorbent for oil removal [8]. Natural organic sorbents obtained from agricultural biomass as feasible spill clean-up solutions are known to be non-destructive to the aquatic environment compared to other methods. Agricultural waste as a biosorbent is promising as it produces no secondary pollution and has no negative effects on the ecosystem. The lignocellulosic sorbent materials that are proven to have good results in removing oil pollution include cotton [9], palm fibres [10], rice straw [11] and fruit peels [7]. The current procedures for cleaning up an oil spill are to use absorbent booms with a blend of synthetic and natural sorbent materials [12]. Natural sorbent materials have the potential to transform liquid oil contaminants into solid or semi-solid states, making them useful for oil spill cleanup [8,13].

Oil palm (*Elaeis guineensis*) is cultivated worldwide as a source of vegetable oil. Indonesia, Malaysia, Nigeria, and Thailand are among the top oil palm planting countries, with production exceeding 317.57 million tonnes [14,15]. Throughout the globe, Indonesia and Malaysia are the biggest producers and exporters of palm oil and palm oil products with the highest production of fresh fruit bunch (FFB) 49.86% and 32.04%, respectively [15,16]. The most abundant waste from the palm oil industry is the empty fruit bunch (EFB) [17]. Previously, the utilisation of EFB as a boiler fuel was banned as the burning soot can cause air pollution to the environment [15]. Since then, EFB is mainly disposed of in landfills, thus creating a significant disposal problem.

The main composition of elemental content in EFB is carbon [15] followed by oxygen [18], hydrogen [19], nitrogen [20], and sulphur [21]. EFB consists of 80% stalk and 20% spikelet, which is composed of lignin (10–34.37%), hemicellulose (19.5–38.8%), and cellulose (22.2–65%) [22]. Previous research has demonstrated that the chemical composition and other fibre features of the stalk and spikelet are identical [23–25]. Oil palm EFB has a rough, jagged shape and is rich in carbon. The abundant, hard and multicellular solid fibres make it suitable for biosorption. In the past, it has been reported that EFB is utilised as a natural biosorbent to remove heavy metal and dye contaminants from wastewater. Due to the wide use of cellulose as adsorbent materials, the research into the use of EFB for oil spill removal is timely.

The goal of this study was to investigate the ability of EFB spikelets to remove oil spills from seawater in a filter system as well as to determine the best optimisation approach for filtering conditions. Experiments for oil spill clean-up were carried out using a filter-based oil sorption system with a variety of factors such as temperature, time, packing density, and oil concentration to determine sorption capacity, oil, and water absorption efficiency. The
statistical optimisation of numerous aspects was achieved via one-factor-at-a-time (OFAT) analysis and response surface methodology (RSM).

2. Materials and Methods

2.1. Materials

Empty fruit bunch was collected from the oil palm sector in Manjung, Perak, Malaysia and stored at room temperature until future use. The samples were rinsed with distilled water to remove the debris. The samples were sun-dried until they reached a constant weight. Diesel fuel (Dynamic diesel fuel Euro 5) was bought from PETRONAS UPM Serdang, Selangor, Malaysia. Seawater with a salinity of 15.19 ppt and pH 7.5–8.1 was obtained from Port Klang (2.9999° N, 101.3928° E).

2.2. Laboratory Scale Set Up and Sorbents Selection

A filter column made from a plastic bottle (250 mm × 50 mm) was used. Each sample (5 g) was placed inside a PVC mesh wire cylinder spacer (h = 10 cm, d = 5 cm) and put in the column. Diesel (40 mL) was vigorously mixed in seawater (400 mL) and poured into the column. After each run, it was left to drip for 10 min. The final weight of samples together with the seawater and oil effluents were observed and measured. The sorption capacity (Equation (1)) together with the oil and seawater absorption efficiency (Equation (2)) were calculated using the following formula:

\[
\text{Diesel sorption capacity} \ (\text{g/g}) = \frac{M_a - M_b}{M_b} \tag{1}
\]

where \(M_a\) is the mass of the sample after it has absorbed diesel, and \(M_b\) is the mass of the sample before it has absorbed diesel [26].

\[
\text{Efficiency of diesel/seawater removal} \ (\%) = \frac{D_f - D_i}{D_i} \times 100\% \tag{2}
\]

where \(D_f\) is the diesel’s final volume (mL) after sorption, and \(D_i\) is the diesel’s starting volume (mL) before sorption.

2.3. Fourier Transform Infrared Spectroscopy (FTIR) Analysis

The differences in samples composition of untreated and treated samples before and after the filtration system based on the presence of functional groups was determined by using FTIR (ALPHA, Bruker Optik GmbH, Ettlingen, Germany) [27].

2.4. Statistical Experimental Design

2.4.1. One-Factor-at-a-Time (OFAT)

Conventional OFAT analysis based on the four selected parameters of temperature (140, 150, 160, 170, 180, and 190 °C), time (10, 15, 20, 25, 30, and 35 min), packing density (0.06, 0.07, 0.08, 0.09, 0.10, and 0.11 g/cm³) and oil concentration (5, 10, 15, 20, 25, and 30% (v/v)) was carried out to evaluate the optimum effects on the efficiency of diesel-seawater sorption. All experiments were done in triplicates. One-way analysis of variance (ANOVA) by GraphPad Prism 8.0.2 software (GraphPad Inc, San Diego, CA, USA) was conducted to analyse the significant factors. Through One-way ANOVA, the significant influence of each parameter was tested by pairwise post hoc comparisons using Tukey’s test [28].

2.4.2. Response Surface Methodology (RSM)

Response surface methodology is a technique for creating tests with fewer runs. Two experimental designs, the Plackett–Burman Design (PBD) and the central composite design (CCD), were used in RSM. The appropriateness of the model terms was determined using analysis of variance (ANOVA) in both configurations. Fisher’s F test and ANOVA were used to examine the significance of each model term. To indicate that the model terms
are accepted as significant, the p-values must not exceed 0.05. The R² values determine the goodness of fit and adequate precision where values greater than 4 are desirable for measuring the signal to noise ratio. The efficiency of oil and water absorbed were examined using the statistical approach of RSM [28].

Plackett–Burman Design

Four factors were picked using OFAT, analysed using the Plackett–Burman Design and tested at high (+1) and low (−1) levels (Table 1). Eighteen experimental runs together with six centre points were obtained. Using the Design Expert 13.0.5.0 software (Stat-Ease Inc., Minneapolis, MN, USA), the experimental design was developed and analysed [28]. PB factorial design at two levels was listed in Equation (3):

$$y = \beta_0 + \sum_{i=1}^{k} \beta_i X_i$$  \hspace{1cm} (3)

where $y$ is diesel and seawater absorbed efficiency, $\beta_0$ is the intercepted model and $\beta_i$ is the linearity coefficient, $X_i$ is the independent variable’s coded level, and $k$ is the number of variables.

Table 1. Experimental values and levels of variables tested in Plackett–Burman design with EFB spikelet as biosorbent.

| Variables         | Code | Unit   | Experimental Range |
|-------------------|------|--------|--------------------|
| Temperature       | A    | °C     | Low (−1) 140       |
|                   |      |        | High (+1) 170      |
| Time              | B    | min    | 20                 |
| Packing density   | C    | g/cm³  | 0.08               |
| Oil concentration | D    | % (v/v)| 15                 |

Central Composite Design (CCD)

To construct the response surface of the identified significant parameters with p-values of less than 0.05, CCD was conducted [28]. In Table 2, the combination of two factorial points (−1, +1), two axial points (−2, +2) and a sole central point (0) were applied to three significant variables. The experimental response was fitted to a second-order polynomial regression model to predict the optimal conditions. Equation (4)’s quadratic mathematical model was used:

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

where $y$ is the response variable, $x$ is the independent factors that influence $y$, $\beta_0$ is the intercept, $\beta_i$ is the coefficient of ith linear, $\beta_{ii}$ is the coefficient of quadratic, $\beta_{ij}$ is the coefficient of interaction effect and $k$ is the number of involved factors. From three significant variables, the total of 20 experiment runs with 6 centre points was determined. All experiments were done in triplicates.

Table 2. CCD optimisation values for chosen independent factors.

| Symbol          | Unit | Experimental Values |
|-----------------|------|---------------------|
| Temperature     | A    | −2 129.773, 140 155, 170 180.227 |
| Packing density | B    | −2 0.069773, 0.08, 0.095, 0.11, 0.120227 |
| Oil concentration| C    | −2 9.88655, 15, 22.5, 30, 35.1134 |

Note: Factorial points (−1, +1); Axial points (−2, +2); Sole central point (0).
3. Results and Discussion

3.1. EFB Spikelets Profile

FTIR spectra for EFB spikelets untreated before and after wetting with oil are presented in Figure 1. EFB spikelets in FTIR spectra untreated before wetting with oil showed a broad peak at 3297.41 cm\(^{-1}\), providing evidence for high amounts of stretching of O-H groups, which indicated the presence of cellulose, hemicellulose, and lignin from the plant [29,30]. The O-H stretching (3291.72 cm\(^{-1}\)) in untreated EFB spikelets was decreased after the oil treatment, implying that hydroxyl groups from lignin, hemicellulose, and cellulose were reduced [31,32]. FTIR spectra showing the elevated stretching band of the C-H (2852.19 cm\(^{-1}\) and 2920.08 cm\(^{-1}\)) before wetting with oil is believed to be from lignin, hemicellulose, and cellulose. The existence of a long chain alkyl group from the diesel hydrocarbon was revealed by FTIR spectra showing the stretching band of the C-H (2855.11 cm\(^{-1}\) and 2924.21 cm\(^{-1}\)) following wetting with oil.

![Figure 1. Comparison of IR spectra between untreated EFB spikelets before and after wetting with oil.](image1)

FTIR spectra for EFB spikelets treated at 160 °C before and after wetting with oil are presented in Figure 2. After the heat treatment, the O-H stretching in EFB spikelets was weakened, which concluded the loss of hydroxyl groups from lignin, hemicellulose, cellulose and other impurities [31–33]. From FTIR spectra, the enhanced stretching band of the C-H (2853.47 cm\(^{-1}\) and 2921.59 cm\(^{-1}\)) after wetting with oil showed the presence of a long chain alkyl group from the diesel hydrocarbon. FTIR spectra of treated samples after wetting with oil showed a broad peak at a range from 1460 to 1375 cm\(^{-1}\) that gives evidence for the presence of a diesel aromatic C=C group. The presence of the C-H functional group confirmed the sorption of diesel oil at the hydrophobic sites of the EFB [34–36]. Thus, it is confirmed that EFB spikelets can adsorb diesel oil.

![Figure 2. Comparison of IR spectra between EFB spikelets treated with 160 °C before and after wetting with oil.](image2)
3.2. Optimisation of Oil Absorbed Using OFAT

3.2.1. Effects of Temperature and Time on Treated EFB Spikelets

Temperature and time can affect the efficiency of oil absorption, water absorption, and sorption capacity of EFB spikelets. Untreated EFB spikelets have lower oil absorption than treated EFB spikelets, as seen in Figure 3. The temperatures of 140, 150, 160, 170, 180 and 190 °C were applied to the samples based on previous studies [37]. The highest amount of oil and a considerable amount of water were absorbed at 160 °C (Figure 4). The results also showed that among different heating times, the efficiency of oil absorbed was the highest at 160 °C at 30 min. The amount of high oil and low water absorbed at various heating temperatures and times indicated a high selectivity of the sorbent for oil.

![Figure 3. Temperature effects on treated EFB spikelets. The average effectiveness of oil absorption (%), the efficiency of water absorption (%), and the sorption capacity (g/g) on temperature were all found. SEM of three replicates illustrated by vertical bars.](image1)

![Figure 4. Time effects on treated EFB spikelets. The average efficiency of oil absorption (%), the efficiency of water absorption (%), and the sorption capacity (g/g) on time were obtained. SEM of three replicates illustrated by vertical bars.](image2)
To reinforce the weak surface properties and improve the interface of the fibre matrix, a thermal treatment modification that reduces the polar components was used [38]. Previous research has proven that the absorbent efficiency increases in heat-treated samples [7,11,39–42]. Thermal treatment, which leads to the modification of the fibre surface, supports a more significant fibre interaction by successfully decreasing the water absorption [43,44]. In addition, thermal treatment eases oil adsorption by removing the surface impurities [45] and helps to develop surface roughness to improve the oil ability adhesiveness [46]. However, thermal treatment for spikelets requires lower temperatures than stalks due to their higher dry weight, which leads to reduced heat requirements for sterilisation [47].

3.2.2. Effects of Packing Density on Treated EFB Spikelets

Oil absorption, water absorption and sorption capacity of sorbent were studied using a range of packing densities based on previous studies [37]. The efficiency of oil absorption increased gradually from a packing density of 0.08 to 0.1 g/cm\(^3\); after a further increase to 0.11 g/cm\(^3\) packing density, the oil absorption decreased (Figure 5). Less availability of space compound at high packing density (0.11 g/cm\(^3\)) to adsorb oil is the main reason for the decrease in oil absorbed efficiency [48]. Similar observations have been reported previously by Verasoundarapandian et al. [7], Taufik et al. [11], Khalid et al. [39] and Abel et al. [49]. In addition, the development of porosity between the fibre was increased in a lower fibre packing density (0.06 and 0.07 g/cm\(^3\)) due to non-compacted samples having no sufficient capillary pressure to absorb oil [39,50].

![Figure 5. Packing density effects on treated EFB spikelets. The average effectiveness of oil absorption (%), the efficiency of water absorption (%), and the sorption capacity (g/g) on packing density were all found. SEM of three replicates illustrated by vertical bars.](image)

3.2.3. Effects of Oil Concentration on Treated EFB Spikelets

The heat-treated spikelets were exposed to various concentrations of diesel oil ranging from 5% to 30% (v/v) (Figure 6) based on previous studies [37]. At 25% concentration, the oil absorbed was the highest and decreased at a higher concentration of 30%. This proved that different ranges of oil concentrations significantly affect the efficiency of oil absorption in treated samples. Oil molecules attached to the sample’s hydrophobic reactive sites and the sorbents’ hollow lumen increases the amount of oil absorbed [46]. The sorbents’ micropores and macropores were able to absorb oil until equilibrium was reached [41]. When the reactive sites of sorbents were saturated with oil molecules, equilibrium was reached, and desorption occurred [51]. These findings revealed that the saturation of
sorbent occurred at a 25% oil concentration, which coincides with the findings by Vera-
soundarapandian et al. [7].

![Figure 6. Oil concentration effects on treated EFB spikelets. The average efficiency of oil absorbed (%), the efficiency of water absorbed (%) and sorption capacity (g/g) on oil concentration were obtained. SEM of three replicates illustrated by vertical bars.](image)

3.3. Optimisation of Oil Absorbed Using Response Surface Methodology (RSM)

3.3.1. Plackett–Burman Design

After analysing all factors using OFAT, 18 runs from PB design for diesel and seawater sorption efficiency were generated (Table 3). At run 11, the lowest value of oil-absorbed efficiency was found under the conditions of 140 °C, 20 min, and 0.08 g/cm³ with 15% diesel and the highest value of oil-absorbed efficiency was obtained at run 8 with the conditions of 170 °C, 20 min, and 0.11 g/cm³ with 30% diesel.

Table 3. Screening of significant parameters affecting oil and water effluent using Plackett–Burman design.

| Run | A   | B   | C   | D   | Oil Absorbed | Water Absorbed |
|-----|-----|-----|-----|-----|--------------|----------------|
| 1   | 140 | 20  | 0.11| 15  | 8            | 8              |
| 2   | 155 | 27.5| 0.095| 22.5| 9.06667     | 5              |
| 3   | 140 | 35  | 0.11| 30  | 9.33333     | 5.33333        |
| 4   | 155 | 27.5| 0.095| 22.5| 9.16667     | 5.33333        |
| 5   | 155 | 27.5| 0.095| 22.5| 9             | 5              |
| 6   | 140 | 35  | 0.11| 15  | 8.3333      | 9.3333         |
| 7   | 155 | 27.5| 0.095| 22.5| 9.33333     | 5              |
| 8   | 170 | 20  | 0.11| 30  | 10.6667     | 5.33333        |
| 9   | 140 | 35  | 0.08| 30  | 8            | 6              |
| 10  | 155 | 27.5| 0.095| 22.5| 9.06667     | 5.33333        |
| 11  | 140 | 20  | 0.08| 15  | 7.66667     | 6.3333         |
| 12  | 140 | 20  | 0.08| 30  | 7.66667     | 5.33333        |
| 13  | 170 | 35  | 0.08| 30  | 9.83333     | 4.33333        |
| 14  | 155 | 27.5| 0.095| 22.5| 10           | 5              |
| 15  | 170 | 35  | 0.08| 15  | 8            | 6              |
| 16  | 170 | 20  | 0.11| 30  | 10.3333     | 5.16667        |
| 17  | 170 | 35  | 0.11| 15  | 9            | 9.66667        |
| 18  | 170 | 20  | 0.08| 15  | 9            | 7              |

A: Temperature (°C); B: Time (min); C: Packing density (g/cm³); D: Oil concentration (%).
Table 4 shows the results of the ANOVA, which illustrated that three components, A (Temperature), C (Packing density), and D (Oil concentration), were significant with p-values less than 0.05. In PBD analysis, the non-significant factors were eliminated and the significant factors were carried forward in CCD to generate the response surface of the identified significant parameters (p < 0.05). Three factors, A (Temperature), C (Packing density), and D (Oil concentration) were therefore carried forward to CCD analysis and the parameter of B (Time) was eliminated.

Table 4. The PBD model’s oil absorbed ANOVA was utilised to discover the element that had a significant impact on diesel sorption.

| Source       | Sum of Squares | DF | Mean Square | F Value | p Value |
|--------------|----------------|----|-------------|---------|---------|
| Model        | 10.53          | 4  | 2.63        | 14.72   | 0.0001  |
| A            | 5.11           | 1  | 5.11        | 28.60   | 0.0002  |
| B            | 0.0579         | 1  | 0.0579      | 0.3237  | 0.5799  |
| C            | 2.52           | 1  | 2.52        | 14.10   | 0.0027  |
| D            | 2.84           | 1  | 2.84        | 15.86   | 0.0018  |
| Residual     | 2.15           | 12 | 0.1788      |         |         |
| Cor Total    | 13.49          | 17 |             |         |         |
| Std. Dev.    | 0.4228         |    |             |         |         |
| Mean         | 8.97           |    | R²          | 0.8307  |         |
| C.V.         | 4.71           |    | Adjusted R² | 0.7743  |         |
|              |                |    | Predicted R² | 0.5857  |         |
|              |                |    | Adequate Precision | 13.0860 |         |

3.3.2. Central Composite Design (CCD)

The CCD design resulted in 20 runs as a consequence of the outcome analysis (Table 5). The maximum amount of oil was absorbed in run 16 at 155 °C, 0.095 g/cm³ and 22.5% diesel. At 129.773 °C, 0.095 g/cm³ with 22.5% diesel, the lowest value of oil-absorbed efficiency was recorded in run 2.

Table 5. Optimised parameters for oil removal by EFB fibre using central composite design (CCD).

| Run Order | A  | B     | C     | Oil Absorbed (mL) |
|-----------|----|-------|-------|-------------------|
|           |    |       |       | Experimental Value | Predicted Value |
| 1         | 155| 0.095 | 22.5  | 8.5               | 5                |
| 2         | 129.773| 0.095 | 22.5  | 5                 | 6                |
| 3         | 140| 0.11  | 30    | 6.33333           | 6.66667          |
| 4         | 140| 0.08  | 15    | 5.33333           | 3.33333          |
| 5         | 140| 0.11  | 15    | 7.33333           | 6                 |
| 6         | 155| 0.095 | 22.5  | 8.33333           | 4                 |
| 7         | 155| 0.0697731| 22.5  | 6.33333           | 2.66667          |
| 8         | 155| 0.120227| 22.5  | 8                 | 9                 |
| 9         | 170| 0.08  | 15    | 7                 | 7.33333          |
| 10        | 155| 0.095 | 22.5  | 8.33333           | 5.16667          |
| 11        | 170| 0.08  | 30    | 7.16667           | 5                 |
| 12        | 155| 0.095 | 22.5  | 8.66667           | 4.33333          |
| 13        | 155| 0.095 | 9.88655| 7.22667           | 7.77333          |
| 14        | 155| 0.095 | 22.5  | 8.16667           | 4.5               |
| 15        | 170| 0.11  | 30    | 7.66667           | 5.33333          |
| 16        | 155| 0.095 | 22.5  | 9                 | 5                 |
| 17        | 140| 0.08  | 30    | 6                 | 4.83333          |
| 18        | 170| 0.11  | 15    | 7.33333           | 7.66667          |
| 19        | 155| 0.095 | 35.1134| 8.66667           | 8.33333          |
| 20        | 180.227| 0.095| 22.5  | 7                 | 5.33333          |

A: Temperature (°C); B: Packing density (g/cm³); C: Oil concentration (%).
In CCD, the significant factors from PBD were used: A (Temperature), B (Packing density), and C (Oil concentration). An ANOVA quadratic model was applied and conducted to detect the significance of each model term. The outcome analysis displayed in Table 6 suggested that the model was highly significant ($p < 0.0001$), with $R^2 = 0.9358$ and a non-significant lack of fit (0.1662) to show that the model used was acceptable. The oil absorption ($Y$) model equation efficiency is listed in Equation (5).

$$Y = +19.44 - 1.34A + 3.87B - 8.82C + 1.34A^2 - 1.30C^2 \quad (5)$$

Table 6. The ANOVA results for the CCD model identified components and pairwise interactions that have a substantial impact on diesel absorption.

| Source     | Sum of Squares | df | Mean Square | F-value | $p$ Value |
|------------|----------------|----|-------------|---------|-----------|
| Model      | 22.83          | 9  | 2.54        | 16.19   | <0.0001   |
| A          | 4.15           | 1  | 4.15        | 26.50   | 0.0004    |
| B          | 2.61           | 1  | 2.61        | 16.66   | 0.0022    |
| C          | 0.4906         | 1  | 0.4906      | 3.13    | 0.1072    |
| AB         | 0.2813         | 1  | 0.2813      | 1.80    | 0.2099    |
| AC         | 0.0868         | 1  | 0.0868      | 0.3541  | 0.4738    |
| BC         | 0.2813         | 1  | 0.2813      | 1.80    | 0.2099    |
| A$^2$      | 12.24          | 1  | 12.24       | 78.11   | <0.0001   |
| B$^2$      | 3.73           | 1  | 3.73        | 23.83   | 0.0006    |
| C$^2$      | 0.7840         | 1  | 0.7840      | 5.00    | 0.0492    |
| Residual   | 1.57           | 10 | 0.1567      |         |           |
| Lack of Fit| 1.12           | 5  | 0.2244      | 2.53    | 0.1662    |
| Pure Error | 0.4444         | 5  | 0.0889      |         |           |
| Cor Total  | 24.40          | 19 |             |         |           |

Table 6 shows that all parameters were independent, indicating no interaction between the pairings of variables A-B, A-C, and B-C. The data clearly reveal that temperature and packing density had a greater impact on the outcome than oil temperature. Three RSM contour plots were generated even though ANOVA analysis resulted in no significant pairwise interaction between any parameters. The model graph of temperature and packing density created by CCD is shown in Figure 7a. The results show that increasing the temperatures and packing density to 158–164 °C and 0.1 g/cm$^3$, respectively, affected the efficiency of oil absorption. The contour plot suggests that temperature has a higher impact on oil absorption compared to packing density. The result, as shown in Figure 7b, demonstrates that the combination of optimum oil contents (21–25%) and temperatures (155–162 °C) produced the major oil absorption at 8.667 mL. Oil absorption was lower at high and low oil concentrations and temperatures. From the 3D contour plot generated by CCD, the oil concentration is suggested to have a lower impact than temperature. Figure 7c depicts that oil concentrations in the range of 21–24% resulted in an increased oil absorption at packing densities of 0.098–0.104 g/cm$^3$. As the graph stayed constant, raising and reducing packing density and oil concentration from the optimal conditions did not affect the amount of oil absorbed. According to the 3D contour plot, the impact of packing density on oil absorption is larger compared to that of oil concentration.
Figure 7. Design Expert (Stat Ease, Inc.) generates 3D Contour plots of the significantly interacting model terms (a) A: temperature and B: packing density, (b) A: temperature and C: oil concentration, and (c) B: packing density and C: oil concentration.
3.3.3. Model Validation Experiment

The model was validated by conducting an experimental trial using the predicted traditional OFAT and statistical RSM as shown in Table 7. According to the predicted value of OFAT, 8.667 mL of oil and 5 mL of water were absorbed. From the RSM fitting model analysis, the predicted values obtained were 8.805 mL and 5.213 mL of oil and water absorbed, respectively. The experimental RSM values of 9 mL and 5 mL of oil and water absorbed were obtained. The result shows the validity of the model as the experimental RSM value was close to the RSM model’s prediction. As compared to OFAT, the data revealed that the RSM design gave a recommended approach to the effectiveness of oil absorption.

Table 7. Validation model using the predicted optimum value in OFAT and RSM.

| Optimised Parameters | Predicted OFAT | Predicted RSM | Experimental RSM |
|----------------------|----------------|---------------|------------------|
| Temperature (°C)     | 160            | 159.660       | 159.660          |
| Packing density (g/cm³) | 0.1           | 0.098         | 0.098            |
| Oil concentration (%) | 25            | 26.998        | 26.998           |
| Oil absorbed (mL)    | 8.667          | 8.805         | 9                |
| Water absorbed (mL)  | 5              | 5.213         | 5                |

4. Conclusions

This is the first study to examine natural sorbents, EFB spikelets sampled from Manjung, Malaysia, as a tool for oil spill removal using the statistical experimental design. According to the experimental results, all parameters were independent, showing that the pairings of variables temperature-packing density, temperature-oil concentration, and packing density-oil concentration, had no interaction. Temperature and packing density had a larger impact on the outcome than oil temperature, according to the findings. Overall, the optimisation of the efficiency of oil absorbed through conventional OFAT and the statistical approach of RSM aided the improvement in the EFB spikelets’ performance, which resulted in 8.667 and 9 mL of oil absorbed, respectively. According to the findings, the statistical RSM model beat the traditional OFAT technique in terms of optimising parameters to optimise oil absorption. The natural biodegradable sorbent material should be recycled and properly disposed of according to the Environmental Protection Agency (EPA). The use of EFB as a sorbent material for oil spill cleanup has proven to be an environmentally benign solution that also maximises the utilisation of agricultural waste. Because of their low cost, environmentally benign nature, ease of deployment, and high efficiency, sorbents made from natural materials are gaining a lot of attention.

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