

**Original Research Article**

**~~CHARACTERIZATION OF BLEACHED GROUNDNUT OIL USING  
ACTIVATED RICE HUSK~~**

**BLEACHING OF GROUNDNUT OIL USING ACTIVATED RICE HUSK:  
A SUITABLE ALTERNATIVE TO COMMERCIAL ADSORBENTS**

**ABSTRACT**

*Absorbent bleaching of crude groundnut oil was performed using activated rice husk as an adsorbent. The study focused on refining the oil by eliminating unwanted components. Response surface methodology was employed to optimize the bleaching process by analyzing the impact of time, adsorbent dosage, particle size, and temperature. Interactive effects on absorbance and peroxide value were also investigated. Bleaching efficiency was determined by measuring absorbance, with a 46.78% reduction observed (from 0.962 to 0.512). The peroxide value decreased from 13.333 to 3.571 mEq/kg of oil. Physiochemical properties were brought within the acceptable codex range. Fourier-transform infrared spectroscopy revealed decreased impurity concentrations, such as nitrile functional groups and unsaturated fatty acids. In conclusion, bleaching positively affected the oil's physiochemical properties, and rice husk proved to be a suitable alternative to commercial adsorbents.*

**Keywords:** Bleaching, Groundnut Oil, Rice Husk, Optimization, Peroxide Value, RSM.

**1. INTRODUCTION**

Vegetable oils, sometimes known as fats, are oils produced from seeds or other plant tissues. Flavor, shelf life, and product color are the aspects of the final product that need to be watched.

From an industrial standpoint, the primary goal of refining is to transform the crude oil into high-quality edible oil by efficiently reducing unwanted contaminants to the appropriate levels. The undesirable ingredient or impurities in groundnut oil may be biogenic, or produced by plants, or they may be pollutants that plants have absorbed from their surroundings. Because of how the peanut kernel is processed, peanut oil has a characteristic nutty flavor and aroma and is a light-yellow tint. However, refinement could produce odorless oil [1]. When paddy rice is husked in the first step of milling, the husk is created, and it is later stripped from other components of the rice grain. As a waste-utilization resource that adds value and lowers processing costs in both home and commercial settings, rice husks have gained popularity as a resource. The conventional method of identifying the best operating conditions while maintaining the others at a steady level involves parameter adjustment. This technique's primary drawback is that it does not account for interactions between the variables, which ultimately results in an incomplete representation of the parameters' full influence on the process. Response Surface Methodology (RSM) can be used to conduct optimization studies to resolve this issue. (RSM). So, this work shows the best way to bleach peanut oil with activated rice husk by adsorption.

## **2. MATERIALS AND METHODS**

### **2.1 RICE HUSK ACTIVATION**

Using the measuring balance, 400 g of Rice Husk (RH) was measured out and put into a 1000 ml conical flask. Then, 500 ml of 3M diluted Sulphuric acid was added to the flask to activate the RH. The mixture that was made was mixed and left at room temperature for two hours and then raised to 100°C and stirred every so often for 3 hours. The RH that had been handled with acid was then washed with enough purified water until the pH was neutral. The neutral RH was dried

in an oven at 80°C for four hours. The color of the RH was noted. It was then sieved and stored in air-tight cases.

## 2.2 DEGUMMING AND NEUTRALIZATION OF GROUNDNUT OIL

Crude groundnut oil in a 500 ml cylindrical jar was put in a thermostat water bath shaker set to 85 °C. Then, 2 ml of phosphoric acid was added to hydrate the phosphatides that couldn't be hydrated. The mixture was mixed for 10 minutes, and 8 ml of water was added to the hot acid oil mixture that had been split into layers. Alkaline refining was used to neutralize the degummed groundnut oil as follows. In a 250 ml beaker, 10 ml of 0.1M NaOH was added to 200 g of degummed oil, and the mixture was mixed for 20 minutes. The soap stock solution was then separated using a centrifuge at 2000 rpm for 10 minutes.

## 2.3 BLEACHING STUDIES

The oil was poured into the bleaching jar, which was set on a hotplate with a magnetic mixer. After bleaching, the samples were filtered and placed in airtight containers until further testing could be done. One milliliter of the oil sample was mixed with n-hexane and methanol, and the spectrophotometer at 450nm was used to measure the absorption. The bleaching efficiency was calculated thus:

$$\% \text{ Bleached} = \frac{A_0 - A_t}{A_0} \times 100 \quad (1)$$

Where  $A_0$  and  $A_t$  are the measured absorbance for crude and refined Groundnut oil at time,  $t$  respectively.

## 2.4 CHARACTERIZATION OF OIL

**Comment [AP1]:** How you do the bleaching, mention the qty of oil and qty of husk taken and stirred at what temperature for how much time

Determination of the physio-chemical characteristics of the bleached and unbleached oil (acid value, free fatty acid, saponification value, ester value, saponification value, iodine value, peroxide value, specific gravity, moisture, and volatile content) were done according to the methodology described in AOAC[2].

## 2.5 FOURIER TRANSFORM INFRARED SPECTROSCOPY ANALYSIS

A few crystals were mixed with KBr (Merck for spectroscopy) and then ground up in an agate mill to make a uniform powder. This powder was then used to make the right pellet under 7 tons of pressure. The Nicolet Model iS10 FT-IR Spectrometer was used to record all the samples' spectra from 4000 to 400  $\text{cm}^{-1}$ . The sample of peanut oil was scanned with a precision of 4  $\text{cm}^{-1}$  and 32 scans per spectrum. With a high sensitivity of 2  $\text{cm}^{-1}$ , the optical program Win-IR Pro Version 3.0 was used to look at the IR spectra.

**Comment [AP2]:** Which crystals? You have to mix the oil with KBr and make a pellet out of it

## 2.6 EMPIRICAL OPTIMIZATION AND STATISTICAL ANALYSIS DESIGN

Using Design Expert 13.0.5.0 (Stat-Ease Inc., Minneapolis, MN, USA), the response surface analysis method was used to set up the experiments and do the statistical analysis. Based on the central composite design (CCD), RSM was used with four factors: temperature (45-105°C), time (15-55 min), adsorbent dose (2.5-8.5 g), and particle size (0.2-1.8 mm) at five levels (-, -1, 0, 1, and) and four replications at the center point. The statistical design table was made by considering these factors and their amounts (28 runs of the process). The absorption and peroxide value were the two responses.

Table 1: Independent factors and chosen levels of the experimental design.

|                                      | <b>TEMPERATURE</b><br>(°C) | <b>ADSORBENT</b><br><b>DOSAGE (g)</b> | <b>BLEACHING</b><br><b>TIME (min)</b> | <b>PARTICLE</b><br><b>SIZE (mm)</b> |
|--------------------------------------|----------------------------|---------------------------------------|---------------------------------------|-------------------------------------|
| <b>LOW (-1)</b>                      | 60                         | 4.0                                   | 25                                    | 0.6                                 |
| <b>HIGH (+1)</b>                     | 90                         | 7.0                                   | 45                                    | 1.4                                 |
| <b>-ALPHA (-<math>\alpha</math>)</b> | 45                         | 2.5                                   | 15                                    | 0.2                                 |
| <b>+ALPHA (+<math>\alpha</math>)</b> | 105                        | 8.5                                   | 55                                    | 1.8                                 |
| <b>CENTER</b><br><b>(MID)</b>        | 75                         | 5.5                                   | 35                                    | 1.0                                 |

Analysis of Variance (ANOVA), regression analysis, and response surface plots of the relationship effects of the factors were used in the statistical analysis to find the best conditions for bleaching. The effects of the process factors on the efficiency were figured out, and the ANOVA test was used to determine how important each effect was. The crude Groundnut oil is then bleached at these optimized variables with a newly prepared adsorbent at the optimal conditions.

### 3. RESULTS AND DISCUSSION

#### 3.1 PHYSIOCHEMICAL CHARACTERIZATION OF GROUNDNUT OIL

Table 2: Physiochemical characterization of groundnut oil before and after bleaching

| PROPERTIES                            | A0            | A1           | A2             |
|---------------------------------------|---------------|--------------|----------------|
| Acid Value (mgKOH/g Oil)              | ≤ 0.6         | 6.73         | 1.4            |
| Saponification Value (mgKOH/g Oil)    | ≤ 2           | 3.388        | 0.70           |
| Free Fatty Acid (%)                   | Light Yellow  | Light Yellow | Pale Yellow    |
| Colour                                | 187 - 196     | 201.96       | 192            |
| Odour                                 | Odourless     | Nutty        | Slightly Nutty |
| Moisture and Volatile Content (% m/m) | ≤ 0.2         | 1.98         | 0.3            |
| Ester Value                           | 186.4 - 195.4 | 198.57       | 191.3          |
| Peroxide Value (mEq/kg of oil)        | ≤ 10          | 13.333       | 3.571          |
| Specific Gravity                      | 0.909 - 0.920 | 0.9095       | 0.9105         |
| Lead, Pb Concentration (mg/kg)        | ≤ 0.1         | 0.75         | 0.12           |
| Copper, Cu Concentration (mg/kg)      | ≤ 0.1         | 0.032        | 0.031          |
| Mercury, Hg (mg/kg)                   | ≤ 0.1         | 0.6          | 0.11           |
| Iodine Value (mg I <sub>2</sub> /g)   | 77 – 107      | 53.086       | 51.624         |
| Refractive Index (ND 40 °C)           | 1.460 – 1.465 | 1.5216       | 1.464          |
| Viscosity (mPas at 40 °C)             | 38.2 ± 5.0    | 77.7         | 43.2           |
| Absorbance at 450nm (A450)            | -             | 0.962        | 0.512          |

\*A0 = Codex Alimentarius Standard for Groundnut Oil [3], A1 = Crude Groundnut Oil, A2 = Groundnut Oil Bleached at Optimum Values.

### **3.1.1 ACID VALUE**

The acid value of the crude groundnut oil was experimentally found to be 6.73 mgKOH/g Oil. After bleaching with the acid activated rice husk at optimum condition, the acid value is determined to have reduced to a value of 1.4 mgKOH/g Oil which is a 79.2% drop from its original value.

### **3.1.2 SAPONIFICATION AND ESTER VALUE**

The saponification value of an oil is a measure of its suitability for use in soap production. The saponification value of the bleached oil decreased by 5% (from 201.96 to 192 mgKOH/g), but this is still a very high value, indicating that the oil that was treated is a typical triglyceride and will be very useful in the production of liquid soap and shampoo. The existence of many ester linkages, as indicated by high saponification levels, is evidence that the fat molecules survived the refining process mostly intact [2].

### **3.1.3 FREE FATTY ACID (FFA) VALUE**

The FFA content seems to be the most utilized criteria for assessing palm oil quality. As predicted, adsorbent addition resulted in an 80% drop in FFA concentration, from 3.388% to 0.7%. A reduction in acidity is largely the consequence of the elimination of phosphatides, phospholipids, and acid generated by the neutralization reactions; nonetheless, it was thought that degumming and neutralization processes contributed significantly to FFA loss.

### **3.1.4 COLOUR AND ODOUR**

Chlorophyll and carotenoids are the two main natural colours in vegetable oils that are not eliminated during alkali refining owing to their inherent properties [4]. The results showed that

the bleached groundnut oil was noticeably paler than the unbleached oil. This indicates that some of the pigment was lost during the bleaching process. The strong nutty smell of the unrefined oil became less intoxicating after refining. This suggests that the odour-inducing chemicals were eliminated by bleaching.

### **3.1.5 MOISTURE AND VOLATILE CONTENT**

It was determined that the unbleached oil had 1.98% moisture, whereas the bleached oil contained just 0.3%. This means that the oil's moisture and volatile content have been cut down by 85% throughout the refining process. Possible causes for this decline include bleaching-induced losses of more water molecules and oil components.

### **3.1.6 PEROXIDE VALUE**

The bleaching process decreases the oil's peroxide value from an experimentally observed 13.333 mEq/kg of oil to an acceptable range of 3.571 mEq/kg of oil, a drop of 73.22%. Since oxidation is slowed in the bleaching process, bleached oil has a longer shelf life than its unrefined counterpart. Factors such as the number of metals bonded in the phospholipids present, the percentage by which the fatty acids content in the degummed oil was reduced, and the addition of a suitable adsorbent all contributed to the observed decrease in **peroxide value (PV)**.

### **3.1.7 SPECIFIC GRAVITY**

The specific gravity of the crude oil was measured at 0.9095 whereas that of the refined oil was measured at 0.9105. This demonstrates a density increase of 0.11 percent following bleaching. It can be concluded that the bleaching process has little to no effect on the specific gravity of the oil sample.

### 3.1.8 METAL CONTAMINANTS (Pb, Hg, Cu)

According to the measured values of metal concentration using the Flame Atomic Absorption Spectroscopy (FAAS) technique, the results show an 84% and 81.7% reduction in the concentration of Lead (0.75 ppm to 0.12 ppm) and Mercury (0.6 ppm to 0.11 ppm) respectively, putting both values of metals within an acceptable codex range for oil [3]. The Copper concentration showed little change and was already within the acceptable range. Bleaching stage metal reduction may be attributable to adsorbents' adsorption characteristics and ion exchange rates, notably for lead and mercury [5]. The activation of adsorbents using acid seems to have had little effect on metal reduction either [6].

### 3.1.9 IODINE VALUE

The results revealed that the iodine content of both the untreated and bleached oil decreased by about 3 percentage points, from 53.086 to 51.624 mgI<sub>2</sub>/g. Both the pre- and post-refining iodine values of groundnut oil are unusually low, indicating that the oil is not as saturated and contains more double bonds than is typical for groundnut oil and hence a higher level of heat treatment was applied to the oils during refining [7] (Beki et al., 1989). Also, the presence of mainly saturated triglyceride molecules indicates that they are a non-drying oil. According to the criteria established by G. Birch [8], an oil is considered drying if its iodine value is more than 100, whereas a non-drying oil has an iodine value of less than 100. This is a good sign of this oil sample's oxidative stability. This may be because of the source of the groundnut oil itself being one with uncharacteristically low double bond saturation than the norm.

### 3.1.10 REFRACTIVE INDEX

After bleaching, the oil sample's apparent refractive index was almost the same (a change in value of 3% was found), suggesting that the bleaching technique had little influence on the oils' refractive index. It has also been observed that the refractive index remains constant during the bleaching process [9]. According to [10], while determining an oil's refractive index, the quantity of reflection created by a beam of light depends on the concentration of impurities in the oil. The reported value is also consistent with the efforts of P. C. Verma [11].

**Comment [AP3]:** Put the authors name et al here

### 3.1.11 VISCOSITY

The unbleached oil had a very high measured value of viscosity at 77.7 mPas, 103.4% higher than the accepted codex standard. After bleaching there was a 44% decrease in the viscosity of the oil to 43.2 mPas which was within the Codex range, ensuring longer shelf life and sustainability of use when undergoing the most common applications of groundnut oil which are deep frying processes.

### 3.1.12 ABSORBANCE

A high absorbance value when bleaching oil can indicate that the oil contains a high level of impurities that need to be removed. The absorbance value of 0.512 of the oil after bleaching shows a reduction from the unbleached oil's value of 0.962. The bleaching efficiency was estimated to be 46.78% which shows the prepared adsorbent was a moderate success at bleaching the oil.

### 3.2 FOURIER TRANSFORM INFRARED (FT-IR) SPECTROSCOPY ANALYSIS OF GROUNDNUT OIL

Figure 1 shows the plotted spectra of the crude oil and the refined oil. Indicators of the bleaching-induced alterations may be found in the composition of the functional groups. From the FT-IR analysis of the oil as presented in the figures below, it can be noticed that they all shared common peaks and values which is to justify the fact that they are the same sources. Some of the major peaks are thus explained below. In the functional group region of both spectra, there are two major broad intense peaks noticed. The first major bands at  $3458\text{cm}^{-1}$  and  $3452\text{cm}^{-1}$  may be due to the stretching vibrations of O-H groups present in the oil and may indicate the presence of water or hydroxyl containing compounds like phospholipids present in the oil. The peak at  $3458\text{cm}^{-1}$  may indicate the presence of amino acids which are an impurity in oil as the band may be caused by the stretching vibrations of N-H bonds in primary amines. The transmittance value of the bleached oil is higher in comparison to the unbleached oil, and this indicated a lower concentration of the compounds present which show the bleaching process has helped reduce the presence of water and amino acid impurities in the oil. The second major bands at  $1636.62\text{cm}^{-1}$  and  $1615\text{cm}^{-1}$  may be caused by the stretching vibrations of carbonyl C=O or C=C bond in esters, carboxylic acids, and ketones in an unsaturated fatty acid. This peak is a characteristic absorption band of triglyceride oils and is often used to assess the degree of unsaturation of the fatty acids in the oil. It indicates the presence of triglycerides in the oil. The presence of unsaturated fatty acids can affect the properties of the oil, such as its melting point and oxidative stability. The degree of unsaturation can also be an indicator of the quality and freshness of the oil, as unsaturated fatty acids are more susceptible to oxidation and rancidity. The % transmittance of this peak is higher in the bleached oil when compared to the unbleached

oil which indicates a lower concentration of these constituents. The reference spectra used for this analysis were obtained from the following: MERCK Sigma-Aldrich<sup>®</sup> IR Spectrum Table & Chart, NIST/EPA/NIH Mass Spectral Library (NIST 17): Standard Reference Database 1A (NIST Standard Reference Database 1A | NIST, 2020) and the KnowItAll<sup>®</sup> Informatics System available through Bio-Rad Laboratories, Philadelphia, PA.

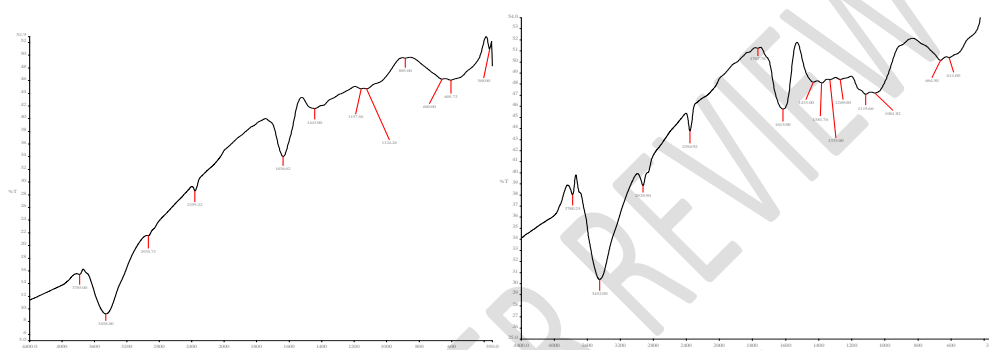


Figure 1: FT-IR spectra of the (L-R) unbleached and bleached groundnut oil

### 3.3 DESIGN OF EXPERIMENT

Design Expert 13.0.5.0 (Stat-Ease Inc., Minneapolis, MN, USA) was used to conduct the response surface analysis-based statistical analysis and experimentation. The statistical design table was generated by consideration of the factors and the levels at which they appeared. The four variables in this setup were (A) temperature, (B) adsorbent dose, (C) particle time, and (D) contact time. The two dependent variables were the peroxide value and absorbance. ANOVA was used to determine the statistical significance of the determined bleaching efficiency impacts of the process variables. The statistical importance of the regression coefficients was determined using the p-value, where a p-value of less than 0.05 indicated significance. The accuracy of the

model was evaluated by comparing the coefficient of determination ( $R^2$ ) value before and after adjusting for outliers in the experimental data.

### 3.4 ANALYSIS OF VARIANCE (ANOVA)

#### 3.4.1 RESPONSE 1: ABSORBANCE

The Model F-value of 875.74 indicates that the model is statistically significant. An F-value this high could only happen by accident 0.01% of the time. If the p-value for a model term is less than 0.0500, then it is significant. B, C, D, A<sup>2</sup>, B<sup>2</sup>, C<sup>2</sup>, and D<sup>2</sup> all play significant parts in this model. If the value of a model term is larger than 0.1000, it is not important in the model. From the results of the analysis, it showed that the model exhibited no evidence of poor fit and had strong determination coefficients ( $R^2 = 0.9989$ ), suggesting that 99.89% of the variance could be accounted for by the model. A signal-to-noise ratio of 133.188 signifies that this model may be utilized for navigating the design space.

Table 3: Analysis of variance for absorbance

| Source             | Sum of Squares | df | Mean Square | F-value | p-value  |             |
|--------------------|----------------|----|-------------|---------|----------|-------------|
| <b>Model</b>       | 0.0451         | 14 | 0.0032      | 875.74  | < 0.0001 | significant |
| A-Temperature      | 0.0000         | 1  | 0.0000      | 4.35    | 0.0574   |             |
| B-Adsorbent Dosage | 0.0024         | 1  | 0.0024      | 646.33  | < 0.0001 |             |
| C-Particle Size    | 0.0017         | 1  | 0.0017      | 463.53  | < 0.0001 |             |
| D-Contact Time     | 0.0030         | 1  | 0.0030      | 803.03  | < 0.0001 |             |
| AB                 | 0.0000         | 1  | 0.0000      | 0.0000  | 1.0000   |             |
| AC                 | 0.0000         | 1  | 0.0000      | 0.0000  | 1.0000   |             |
| AD                 | 0.0000         | 1  | 0.0000      | 0.0000  | 1.0000   |             |
| BC                 | 0.0000         | 1  | 0.0000      | 0.0000  | 1.0000   |             |

|                  |        |    |           |         |          |
|------------------|--------|----|-----------|---------|----------|
| BD               | 0.0000 | 1  | 0.0000    | 0.0000  | 1.0000   |
| CD               | 0.0000 | 1  | 0.0000    | 0.0000  | 1.0000   |
| A <sup>2</sup>   | 0.0067 | 1  | 0.0067    | 1823.58 | < 0.0001 |
| B <sup>2</sup>   | 0.0086 | 1  | 0.0086    | 2324.54 | < 0.0001 |
| C <sup>2</sup>   | 0.0061 | 1  | 0.0061    | 1647.27 | < 0.0001 |
| D <sup>2</sup>   | 0.0072 | 1  | 0.0072    | 1962.40 | < 0.0001 |
| <b>Residual</b>  | 0.0000 | 13 | 3.682E-06 |         |          |
| Lack of Fit      | 0.0000 | 10 | 4.787E-06 |         |          |
| Pure Error       | 0.0000 | 3  | 0.0000    |         |          |
| <b>Cor Total</b> | 0.0452 | 27 |           |         |          |

Table 4: Model summary/ fit statistics for absorbance

|                       |        |                                |        |
|-----------------------|--------|--------------------------------|--------|
| <b>Std. Dev.</b>      | 0.0019 | <b>R<sup>2</sup></b>           | 0.9989 |
| <b>Mean</b>           | 0.5656 | <b>Adjusted R<sup>2</sup></b>  | 0.9978 |
| <b>C.V. %</b>         | 0.3393 | <b>Predicted R<sup>2</sup></b> | 0.9939 |
| <b>Adeq Precision</b> |        | 133.1883                       |        |

### 3.4.2 RESPONSE 2: PEROXIDE VALUE

The Model F-value of 29.21 indicates that the model is statistically significant. An F-value this high could only happen by accident 0.01% of the time. If the p-value for a model term is less than 0.0500, then it is significant. Here, the model terms A, B, C, D, A<sup>2</sup>, B<sup>2</sup>, C<sup>2</sup>, and D<sup>2</sup> are very important. If the value of a model term is larger than 0.1000, it is not important in the model. From the results of the analysis, it showed that the model offered no evidence of poor fit and displayed strong determination coefficients ( $R^2 = 0.9692$ ), suggesting that 96.92% of the variance could be accounted for by the model. The signal-to-noise ratio of 24.303 is sufficient, therefore this model may be utilized for navigating the design space.

Table 5: Analysis of variance for peroxide value

| Source             | Sum of Squares | df | Mean Square | F-value | p-value  |             |
|--------------------|----------------|----|-------------|---------|----------|-------------|
| <b>Model</b>       | 75.84          | 14 | 5.42        | 29.21   | < 0.0001 | significant |
| A-Temperature      | 6.18           | 1  | 6.18        | 33.31   | < 0.0001 |             |
| B-Adsorbent Dosage | 1.95           | 1  | 1.95        | 10.51   | 0.0064   | +           |
| C-Particle Size    | 8.64           | 1  | 8.64        | 46.60   | < 0.0001 | + -         |
| D-Contact Time     | 0.9095         | 1  | 0.9095      | 4.90    | 0.0453   | + -         |
| AB                 | 0.0000         | 1  | 0.0000      | 0.0000  | 1.0000   | -           |
| AC                 | 0.0000         | 1  | 0.0000      | 0.0000  | 1.0000   | -           |
| AD                 | 0.0000         | 1  | 0.0000      | 0.0000  | 1.0000   | -           |
| BC                 | 0.0000         | 1  | 0.0000      | 0.0000  | 1.0000   | -           |
| BD                 | 0.0000         | 1  | 0.0000      | 0.0000  | 1.0000   | -           |
| CD                 | 0.0000         | 1  | 0.0000      | 0.0000  | 1.0000   | -           |
| A <sup>2</sup>     | 17.93          | 1  | 17.93       | 96.66   | < 0.0001 |             |
| B <sup>2</sup>     | 1.15           | 1  | 1.15        | 6.19    | 0.0272   |             |
| C <sup>2</sup>     | 5.93           | 1  | 5.93        | 31.99   | < 0.0001 |             |
| D <sup>2</sup>     | 22.97          | 1  | 22.97       | 123.84  | < 0.0001 |             |
| <b>Residual</b>    | 2.41           | 13 | 0.1855      |         |          |             |
| Lack of Fit        | 2.41           | 10 | 0.2411      |         |          |             |
| Pure Error         | 0.0000         | 3  | 0.0000      |         |          |             |
| <b>Cor Total</b>   | 78.25          | 27 |             |         |          |             |

Table 6: Model summary/ fit statistics for peroxide value

|                  |        |                               |        |
|------------------|--------|-------------------------------|--------|
| <b>Std. Dev.</b> | 0.4306 | <b>R<sup>2</sup></b>          | 0.9692 |
| <b>Mean</b>      | 5.30   | <b>Adjusted R<sup>2</sup></b> | 0.9360 |

**C.V. %** 8.13    **Predicted R<sup>2</sup>** 0.8225

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**Adeq Precision** 24.3034

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### **3.5 3D MODEL PLOTS**

#### **3.5.1 ABSORBANCE**

From the response surface plot of the interactive effect of temperature and adsorbent dosage, it can be noticed that the highest absorbance values occur at moderate temperature values at the minimum and maximum adsorbent dosage and the lowest absorbance values occur at moderate adsorbate dosage values at the maximum and minimum temperature. This means that the factors have a hybrid positive/negative interactive effect on the absorbance response. This is the same relationship between temperature and particle size but with a noticeable upward skew in absorbance when the particle size is at its maximum of 1.8 mm indicating that that particle size is not suitable for bleaching. The same effect is noticed in the interactive plots of adsorbent dosage and contact time and particle size and contact time. For the plot of the interaction between temperature and contact time, an increase in both factors leads to an increase in absorbance until relatively moderate values where further increase leads to a decrease in the absorbance value. An opposite effect is noticed in the plot of adsorbent dosage and particle size where an increase in both values leads to a reduction in absorbance values until a certain moderate value where further increase in their values leads to an increase in absorbance indicating that a moderate value of both factors is necessary for optimum bleaching.

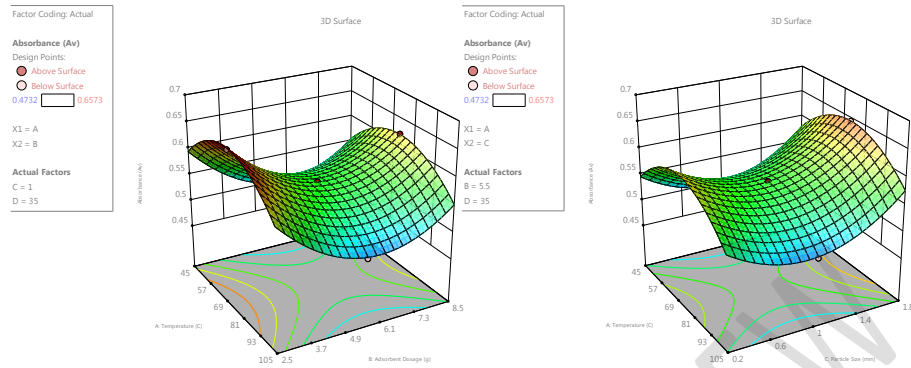


Figure 2: Effect of temperature and adsorbent dosage (L) and temperature and particle size (R) on absorbance

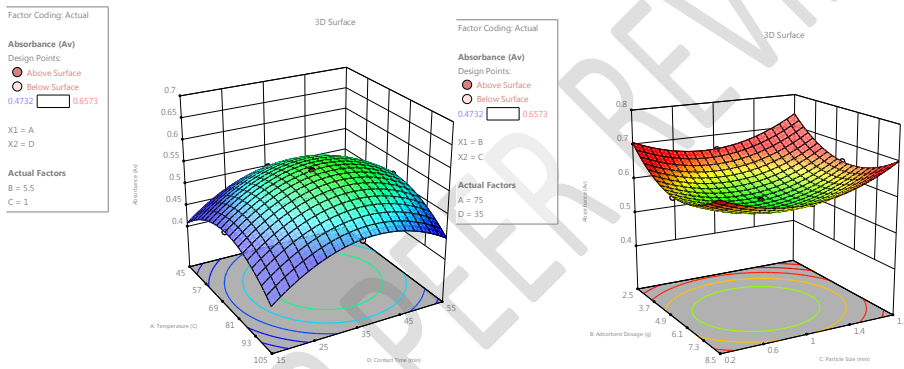


Figure 3: Effect of temperature and contact time (L) and adsorbent dosage and particle size (R) on absorbance.

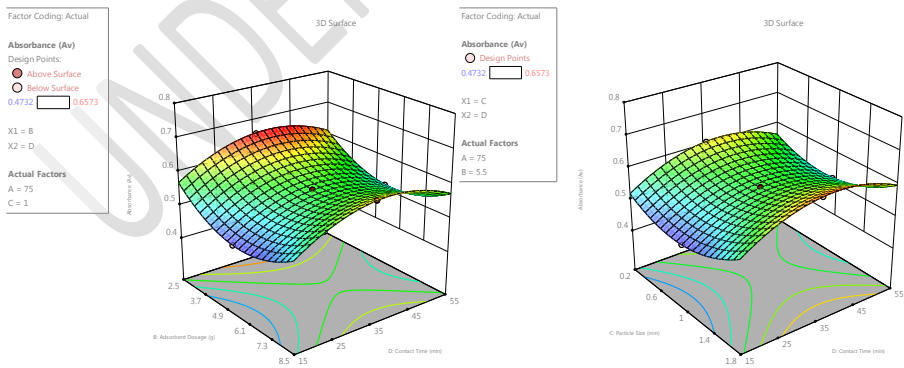


Figure 4: Effect of adsorbent dosage and contact time (L) and particle size and contact time (R) on absorbance.

### 3.5.2 PEROXIDE VALUE

From the response surface plot of the interactive effect of temperature and adsorbent dosage, it can be noticed that the highest peroxide values occur at moderate adsorbent dosage at the minimum and maximum temperature and the lowest peroxide values occur at moderate temperature values at the maximum and minimum adsorbent dosage. This means that the factors have a hybrid positive/negative interactive effect on the absorbance response. In general, it looks like a moderate temperature is the most constraining factor in obtaining lower peroxide values. This is the same relationship between temperature and particle size but with a noticeable upward skew in peroxide value when the temperature is at its maximum of 105°C indicating that higher temperatures are not suitable for bleaching. The same effect is noticed in the interactive plots of adsorbent dosage and contact time and particle size and contact time. For the plot of the interaction between temperature and contact time, an increase in both factors leads to a decrease in peroxide value until relatively moderate values where further increase leads to an increase in the peroxide value. The interactive plot of adsorbent dosage and particle size is relatively flat, indicating that there is little to no interactive effect between both factors on the peroxide value response.

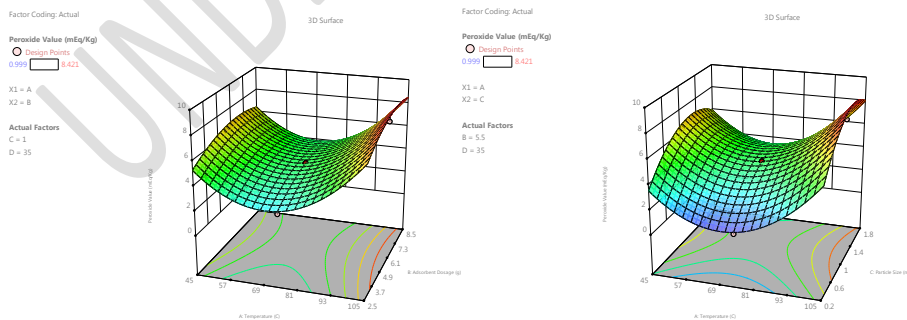


Figure 5: Effect of temperature and adsorbent dosage (L) and temperature and particle size (R) on peroxide value

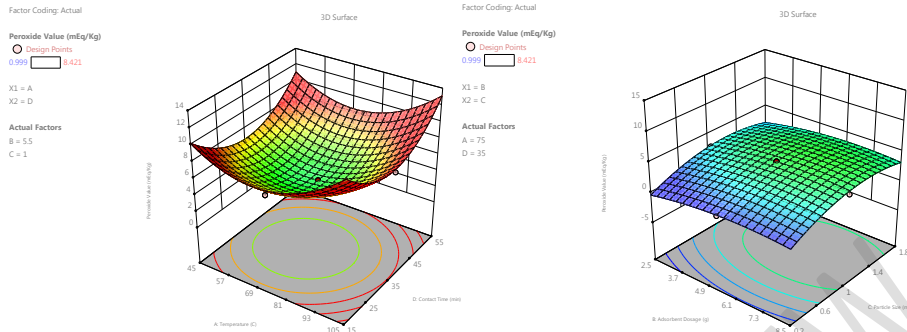


Figure 6: Effect of temperature and contact time (L) and adsorbent dosage and particle size (R) on peroxide value.

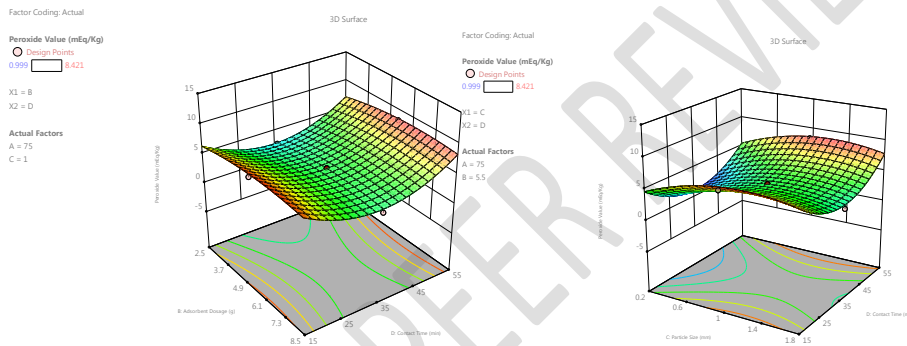


Figure 7: Effect of adsorbent dosage and contact time (L) and particle size and contact time (R) on peroxide value.

### 3.6 NUMERICAL OPTIMIZATION

Numerical optimization involves using statistical and mathematical techniques to find the factor settings that maximize or minimize the response variable, subject to some constraints. These constraints are presented in table 7 below.

Table 7: Factor and response constraints on the numerical optimization

| Name                       | Goal        | Lower Limit | Upper Limit | Importance |
|----------------------------|-------------|-------------|-------------|------------|
| <b>A: Temperature</b>      | is in range | 45          | 105         | 3          |
| <b>B: Adsorbent Dosage</b> | is in range | 2.5         | 8.5         | 3          |
| <b>C: Particle Size</b>    | is in range | 0.2         | 1.8         | 3          |
| <b>D: Contact Time</b>     | is in range | 15          | 55          | 3          |
| <b>Absorbance</b>          | minimize    | 0.4732      | 0.6573      | 5          |
| <b>Peroxide Value</b>      | minimize    | 0.999       | 8.421       | 5          |

The solution given when applying these constraints on the design space selected the optimum conditions with a desirability index of 0.659 as thus:

Temperature: 54.123°C, Adsorbent Dosage: 5.672 g, Particle Size: 0.2mm, Contact Time: 22.34 min

The predictive responses to these optimum conditions were given by the software as:

Absorbance: 0.538 Av, Peroxide Value: 3.463 mEq/kg of oil

Confirmatory experiments were carried out at these conditions to validate the predicted optimum values of the responses. The experimental values of the responses were obtained as follows:

Absorbance: 0.512 Av, Peroxide Value: 3.571 mEq/kg of oil

Which closely agreed with that obtained from the regression model showing a percentage error of 4.83% and 3.11% for absorbance and peroxide value respectively. All physiochemical

characterization and external analysis on the oil were carried out using these optimum conditions.

#### 4. CONCLUSION

In this work, the use of an absorbent made from active rice husk to bleach peanut oil has been investigated. The absorbent was made by making it react to acid. To find the best working settings for the process, the trial setup used CCD in response surface technique. The best bleaching rate of 46.78% was found with a reaction time of 22.34 minutes, a dose of 5.672 g of adsorbent, a particle size of 0.2 mm, and a reaction temperature of 54.123°C. The difference between what was expected and what was found was less than 5% for both numbers. The untreated oil and the ideally bleached oil were looked at and compared. The bleached oil had values that were the same as the Codex vegetable oil standards, which shows that the oil was refined well. An FT-IR test was done on both samples of oil, and it showed that there were less impurities from nitrogen-containing functional groups and that the oil contained unsaturated fatty acid chains. This work has shown that rice husk can be used to make an effective absorbent if the process conditions are just right.

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**Comment [AP4]:** Correct the reference

**Comment [AP5]:** Where is this reference mentioned in the text?