



OPTIMIZATION OF GROUNDNUT OIL EXTRACTION USING CHEMICAL METHOD

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The chemical extraction technique is associated with suboptimal oil yield and quality, necessitating the development of optimized extraction technique. In this study, groundnut oil was extracted chemically with n-hexane using the Response Surface Methodology (RSM) and the extraction process was optimized considering the hexane concentration (30, 40 and 50), extraction duration (3, 5, 7 hrs) and the amount of groundnut (15, 20, and 25 g) as factors and the oil yield was evaluated. The Fourier Transform Infrared (FTIR) technique was used to check at the functional groups on the extracted oil. In the experimental results, the average optimum values of extraction time, solvent amount, groundnut amount, and oil yield were 5.05 hr, 40.5 mL, 20.25 g, and 48.18%, respectively. The profiled extracted oil by FTIR showed peaks at 3452 cm⁻¹ 2969 cm⁻¹ 2712 cm⁻¹ 1763 cm⁻¹ 1474 cm⁻¹ which indicates the existence of -CH and OH, CH, C-H, aldehydes, ketones, carboxylic acids, and methylene groups functional groups which are useful for determining the oil's quality. Furthermore, findings from this study showed that RSM-optimized process parameters result in significantly greater oil yields than conventional approaches.

1.0 Introduction

Groundnut (*Arachis hypogaea* L.) is produced worldwide, and it is widely consumed in many forms, including roasted grain, peanut butter, and peanut oil. The four main world producers are China, India, Nigeria, and the USA, which are distributed in Asia, Africa, and America, these countries are responsible for 63.5% of the world production of peanut grain, 75.3% of peanut oil and 75.8% of peanut meal/cake (Magalhães *et al.*, 2023). In addition to direct consumption, either with or without treatment, peanuts can be the subject of diverse applications focusing mainly on two distinct objectives: oil extraction and defatting processes (Mahfoud *et al.*, 2023) and more than 70% of the harvest is used for oil extraction (Kotadiya and Patel, 2022).

The nutritional value of groundnut proteins has been reported to be relatively high, with a biological value (BV) of 59 and net protein utilization of 51 (Cui *et al.*, 2023). It is a source of nutrients like niacin, which helps to promote healthy blood flow and brain function, folate, antioxidants, vitamin E, magnesium, phosphorus, and dietary fibers containing between 45.9% and 55.4% of lipids that are specifically high in essential polyunsaturated fatty acids (Kotadiya and Patel, 2022). Furthermore, Kotadiya and Patel, (2022) reported that high consumption of nuts is associated with a beneficial impact on the cardiovascular system, due to their antioxidant and anti-inflammatory properties.

Extraction of lipid and other valued substances is strongly reliant on the method employed for extraction therefore, an appropriate extraction technique is required to yield the desired components. Pressing, soxhlet system, and the combined use of pre-pressing and solvent extraction are the three most prevalent processes for producing oils (Idrissi *et al.*, 2022). Innovation in extraction techniques includes supercritical fluid extraction, pressurized liquid extraction, microwave assisted extraction, ultrasound assisted extraction, cold atmospheric plasma extraction, amongst others (Picot-allain *et al.*, 2021).

Mechanical techniques for oil extraction from peanuts are grouped into three main sections: extrusion and screw pressing, cold pressing, and hydraulic pressing (Mahfoud *et al.*, 2023), the oil content of oilseeds may be recovered using a mechanical screw press to the extent of 86% to 92% (Kotadiya and Patel, 2022). However, cold-pressing (CP) productivity is low, and it is difficult to obtain a consistent quality product (Idrissi *et al.*, 2022). Therefore, various novel methods have been developed to increase the yield, reduce denaturation, and improve the functional properties of plant proteins (Cui *et al.*, 2023).

When extracting oil from oilseeds, the organic solvent is used, the most frequently utilized organic solvent is hexane. Oilseeds that have been pre-treated and are in the form of a porous solid matrix are exposed to either pure solvent or a mixture of solvent and oil (known as miscella). According to Prasad *et al.* (2022), the solvent extraction is encountered during various processes in the food industry, viz oil extraction from oilseeds, flavoring and polyphenolic compounds extraction from herbs and spices, and the reasons for the preferred choice of solvent extraction include easy availability at lower price from petroleum-based solvents.

Chemical method is grouped under three major flags: organic solvent extraction (such as hexane), aqueous extraction processing (AEP), and supercritical fluid extraction (SFE) based on the use of CO₂ as solvent (SC-CO₂) (Mahfoud *et al.*, 2023). The solvent extraction is used to dissolve and extract the oil from the peanuts. This method is more efficient than mechanical pressing and can extract a higher percentage of oil. The oil is considered to be nutritious and healthful, owing to its fatty acid composition and the presence of natural bioactive components, such as squalene, phytosterols, tocopherols and tocotrienols (Zhu *et al.*, 2015). The most important quality requirements of groundnut as a source of oil are high protein and oil content in seed and high oleic acid (Sarvamangala *et al.*, 2011). For these reasons, many researchers focus on the increase in the oil yield and the higher recovery of these compounds and develop corresponding methods to extract them from oilseeds (Linn, 2022).

Response Surface Method (RSM) is a tool for function estimation that combines mathematical, statistics, and multiple quadratic regression to optimize influencing conditions from multiple experimental runs from numerous variables using polynomial equations and relate the response to the influential conditions with good practical value, precision, and optimization effects (Mukwevho *et al.*, 2023; Silas *et al.*, 2021). The application of RSM technique for optimizing the oil extraction process can improve the performance and economy of the process. Few studies reported such studies, for example, the effects of extraction time, extraction temperature, solid- to- solvent ratio and ultrasound power on the extraction yield and the oleic acid concentration of the thermosonically extracted groundnut oil were investigated, the findings indicated that extraction yield was primarily affected by the extraction temperature and solid- to- solvent ratio (Ketenoglu, 2020).

The one-at-a-time process optimization for understanding the influences of different operating parameters on the extraction of flavonoids from groundnut shells under ultrasound irradiation (Liao *et al.*, 2021) was reported. In another study, (Liu *et al.*, 2020) the effect of various enzymes on the molecular weight distribution of peanut protein, microscopy of oil bodies, and yield of peanut protein and oil bodies during the aqueous enzymatic extraction process were

investigated. However, these studies failed to report the functional groups on the extracted oil. This study, utilizes the RSM technique to design an experiment using Central Composite Design (CCD) to optimize the groundnut oil yield considering influential factors such as extraction time, amount of solvent and the amount of groundnut. Also, to study the functional groups on the oil using the FTIR technique.

2. Materials and Methods

2.1 Materials

Unroasted groundnuts were purchased from Gumboru local market in Maiduguri. The groundnuts were separated from all possible residues of leaves, hard shells and miscellaneous parts of the plant before analyzes, the sample was placed in an oven at 105 °C for 12 hrs, The dried groundnut seed was first fed to a jaw crusher to increase the surface area of the nut seed in other to aid good extraction. Analytical grade n-hexane used for oil extraction was purchased from Merck, Germany.

2.2 Experimental procedures solvent extraction using n-hexane

In the soxhlet procedure, oil and fat from solid material are extracted by repeated washing (percolation) with an organic solvent where hexane was used. The grounded groundnuts were placed in a porous cellulose thimble. The thimble is then placed in an extraction chamber which is suspended above a flask containing the solvent and below a condenser. Heat is applied to the flask, and the solvent evaporates and moves to the condenser, converted into a liquid that trickles into the extraction chamber containing the sample. The extraction chamber is made in such a way that when the solvent surrounding the sample exceeds a certain level it overflows and trickles back down into the boiling flask. The flask containing solvent and lipid is removed at the end of the extraction process. The solvent in the flask is separated via distillation, and the remaining lipid mass is measured. The percentage of the lipid in the initial sample is then calculated. The percentage oil yield (% by weight) was estimated using the Eq. 1 (Liu et al., 2020):

$$Oil\ Yield\ (\%) = \frac{MO}{MS} 100\% \quad |$$

Where, MO is the mass of oil in kg, MS is the mass of sample in kg.

2.3 Design of experiment (DOE)

The Central Composite Design of Design Expert 10.0 software was used to design the experiment. Extraction time (A), Amount of solvent (B), Amount of groundnut (C), worked as influencing factors, while the yield of peanut worked as response values. Encoding of factor levels is shown in Table I.

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Study Type Response Surface Subtype Randomized

Design Type Central Composite Runs 20

Table I: Design of experiment

Factor	Name	Units		Levels
A	Extraction time	hr	3	7
B	Amount of solvent	mL	30	50
C	Amount of G/nut	g	15	25

2.4 Fourier Transform Infrared Spectroscopy (FTIR) procedure

The Fourier Transform Infrared Spectroscopy (FTIR) analysis was carried out using Perkin Elmer RX. The FTIR spectra were collected in the range of 4000-400 cm^{-1} . Potassium bromide (KBr) pellet was first prepared by mixing about 0.01 g of powdered catalyzed adsorbent with about 0.3 g of KBr (Merck; for spectroscopy) in an agate mortar. The mixture was compressed under a certain pressure in a special die to form a small disk.

3. Results and Discussion

3.1 RSM optimization result

The independent variables (extraction time, solvent quantity, and groundnut amount) as designed by the DOE with the final oil yield data is presented in Table 2. There are six center points with similar independent variables values as designed by the software but their oil yield as calculated differs however, their oil yields are insignificant.

Table 2: Experimental factors and response data

Run	Factor 1 A: Extraction time hr	Factor 2 B: Amount of solvent mL	Factor 3 C: Amount of G/nut g	Response 1 Oil yield (%)
1	7	50	15	50.12
2	3	30	25	48.63
3	5	40	20	47.55
4	3	50	25	48.72
5	5	40	20	46.71
6	5	40	20	46.38
7	7	30	25	58.87
8	3	40	20	45.20
9	5	30	20	48.55
10	5	40	20	48.42
11	7	30	15	50.03
12	3	30	15	44.97
13	7	40	20	54.00
14	7	50	25	59.79
15	5	40	20	48.66
16	5	40	20	48.86
17	5	40	25	50.32
18	5	40	15	46.73
19	5	50	20	47.45
20	3	50	15	45.06

Table 3 shows the ANOVA table with the statistical analysis. The Model F-value of 33.28 implies the model is significant. There is only a 0.01% chance that an F-value this large could occur due to noise. Values of "Prob > F" less than 0.0500 indicate model terms are significant. In this case A, C, AC, A² are significant model terms.

Table 3: ANOVA for Response Surface Quadratic model

Source	Sum of Squares	df	Mean Square	F Value	p-value Prob > F	
Model	300.47	9	33.39	33.28	< 0.0001	significant
A-Extraction time	161.85	1	161.85	161.34	< 0.0001	
B-Amount of solvent	8.100E-004	1	8.100E-004	8.074E-004	0.9779	
C-Amount of G/nut	86.55	1	86.55	86.28	< 0.0001	
AB	0.086	1	0.086	0.086	0.7755	
AC	15.65	1	15.65	15.60	0.0027	
BC	0.086	1	0.086	0.086	0.7755	
A ²	9.93	1	9.93	9.90	0.0104	
B ²	0.25	1	0.25	0.25	0.6296	
C ²	1.87	1	1.87	1.87	0.2017	
Residual	10.03	10	1.00			
Lack of Fit	4.53	5	0.91	0.82	0.5826	not significant
Pure Error	5.51	5	1.10			
Cor Total	310.50	19				

Values greater than 0.1000 indicate the model terms are not significant. If there are many insignificant model terms (not counting those required to support hierarchy), model reduction may improve your model. The "Lack of Fit F-value" of 0.82 implies the Lack of Fit is not significant relative to the pure error. There is a 58.26% chance that a "Lack of Fit F-value" this large could occur due to noise. Non-significant lack of fit is good.

The ANOVA goodness-of-fit, provided by the coefficient of determination (R²), was determined. R² measures the percentage of changes in the response variable that can be attributed to independent variables and their interactions. It also evaluates how well a statistical model fits a given set of data. The closer the R² value is to 1, the better the model matches the data. The R² value of 0.9677 is showing that the model was adequate. To further confirm the model's adequacy, the lack-of-fit error test was carried out. The lack-of-fit error test quantifies inaccuracies due to any flaw(s) in a model.

The lack of fit is not significant if the error probability p, of the lack-of-fit F-statistic is larger than the confidence interval. In contrast, if the F-statistic of the lack-of-fit error is greater than the associated error probability, the lack-of-fit test is said to be significant, and the regression model is inadequate to explain the data. In the present study, as shown in Table 3, the lack of fit was found to be non-significant, indicating that the regression model for oil extraction, was sufficient in explaining the experimental data, similar findings are reported (Bello et al., 2023; Chen et al., 2022). The final equation in terms of coded factor is given in Eq. 2.

$$\text{Oil yield\%} = 47.74 + 4.02*A + 9.00E-003*B + 2.94*C + 0.10*AB + 1.40*AC + 0.10*BC + 1.90*A^2 + 0.3*B^2 + 0.83*C^2 \quad 2$$

The equation in terms of coded factors can be used to make predictions about the response for given levels of each factor. By default, the high levels of the factors are coded as +1 and the low levels of the factors are coded as -1. The coded equation is useful for identifying the relative impact of the factors by comparing the factor coefficients.

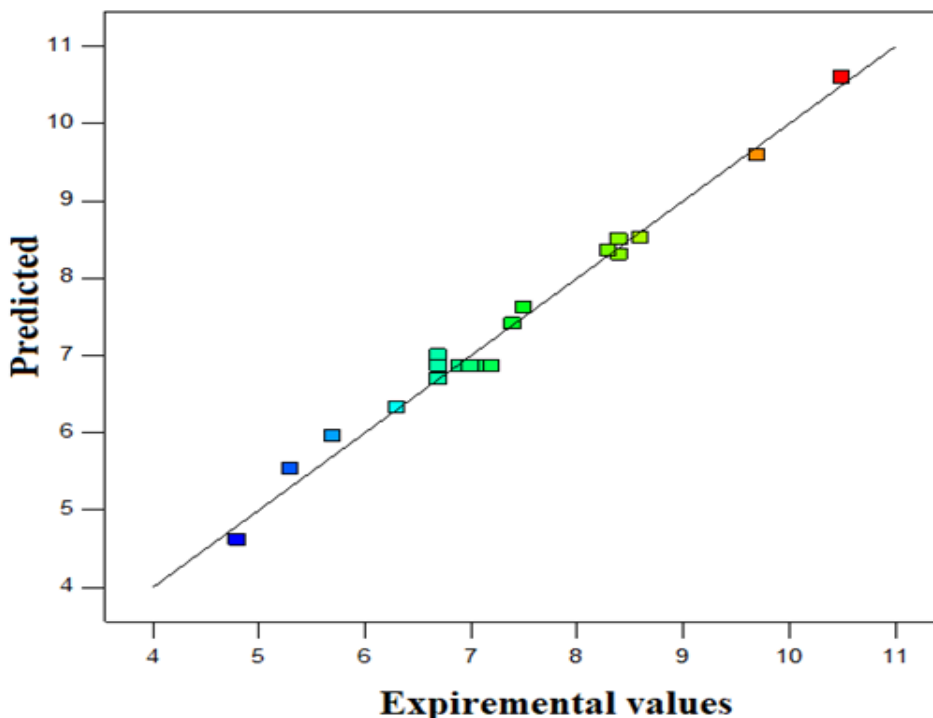


Figure 1: Predicted values versus the actual values for the oil yield.

This plot depicts that the developed model can correlate independent variables (extraction time, amount of groundnut and amount of solvent) with the response (oil yield). The implication of this pattern is that the model proposed is suitable for the present study.

3.2 Response surfaces 3D analysis

These 3D graphics provide an in-depth look at the correlations between oil yield and the various parameters. They enable a better knowledge of how different extraction times, solvent amounts, and groundnut amounts affect oil yield.

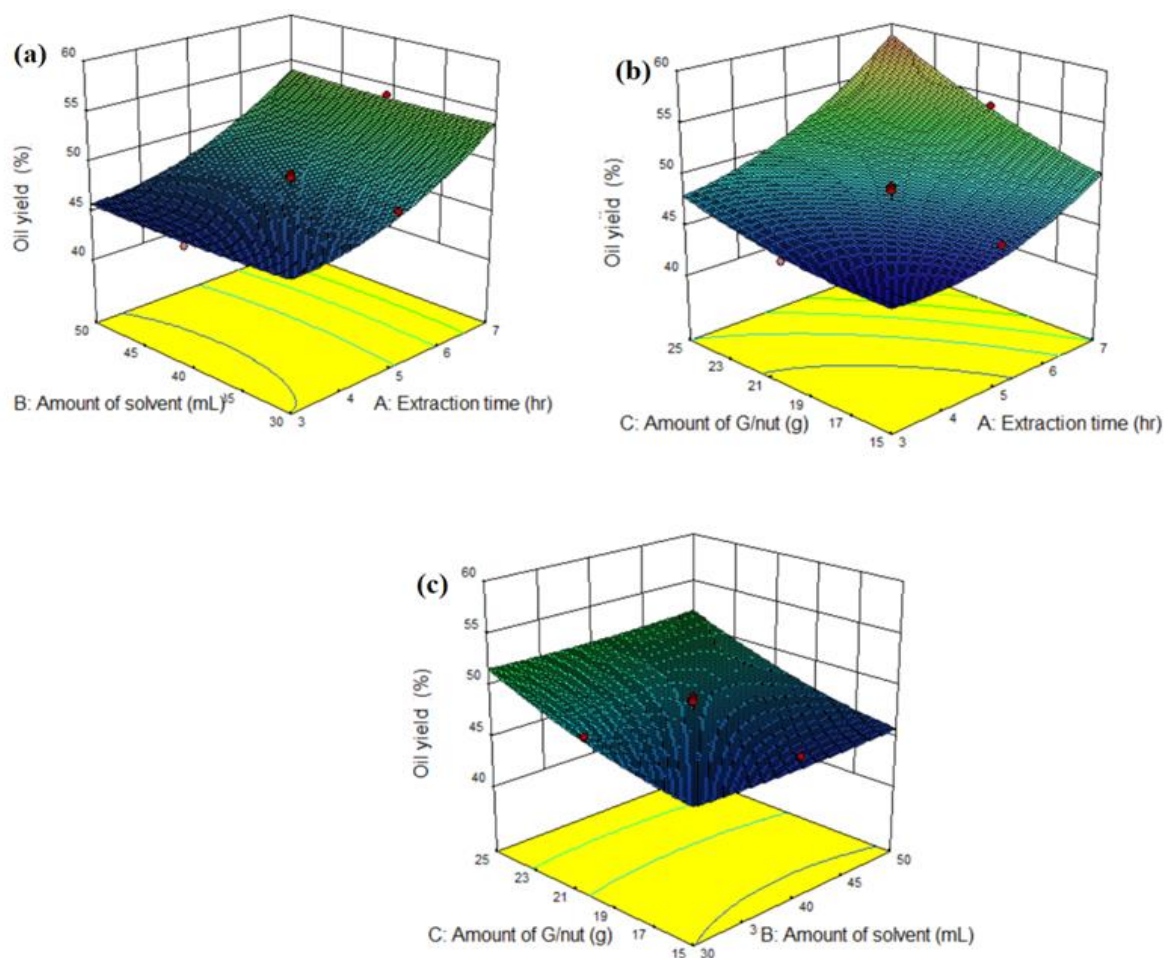


Figure 2: Response surfaces of (a) Amount of solvent against extraction time (b) Amount of g/nut against extraction time (c) Amount of g/nut against Amount of solvent.

Oil yield variation with extraction time and solvent amount depicts the link between oil yield and two major parameters. It displays how variations in extraction time and solvent concentration affect the final oil yield. Through the contour shape, we can see the strong and weak relationship of interaction effect. The closer the shape is to the circle, the less is the significant of the interactive effects of the factors also, parallel line indicates that the interaction between the two factors is insignificant (Chen et al., 2022). It can be seen from contour lines in the Figures 2a-d that the graph is not curved much close to a circle, indicating that the interaction between the two factors, amounts solvent and extraction time (Fig. 2a), amount of g/nut and extraction time (Fig. 2b) and, amounts solvent and amount of g/nut (Fig. 2c) shown on the response surfaces are significant. According to the experiments and the predicted values, Table 4 shows the oil yield obtained.

Table 4: Oil yield obtained by the predicted and the experimental values.

	Extraction time (hr)	Amount of groundnut (g)	Amount of solvent	Oil yield
Predicted	5.0	40.5	20.25	48.2
Experimental	5.0	40.5	20.25	46.2

3.4 FTIR analysis

FTIR technique shows the functional groups on the extracted oil from groundnut providing valuable insights into the chemical composition of the extracted oil. The FTIR spectrum of the extracted oil considering the optimum oil yield is presented in Fig. 3.

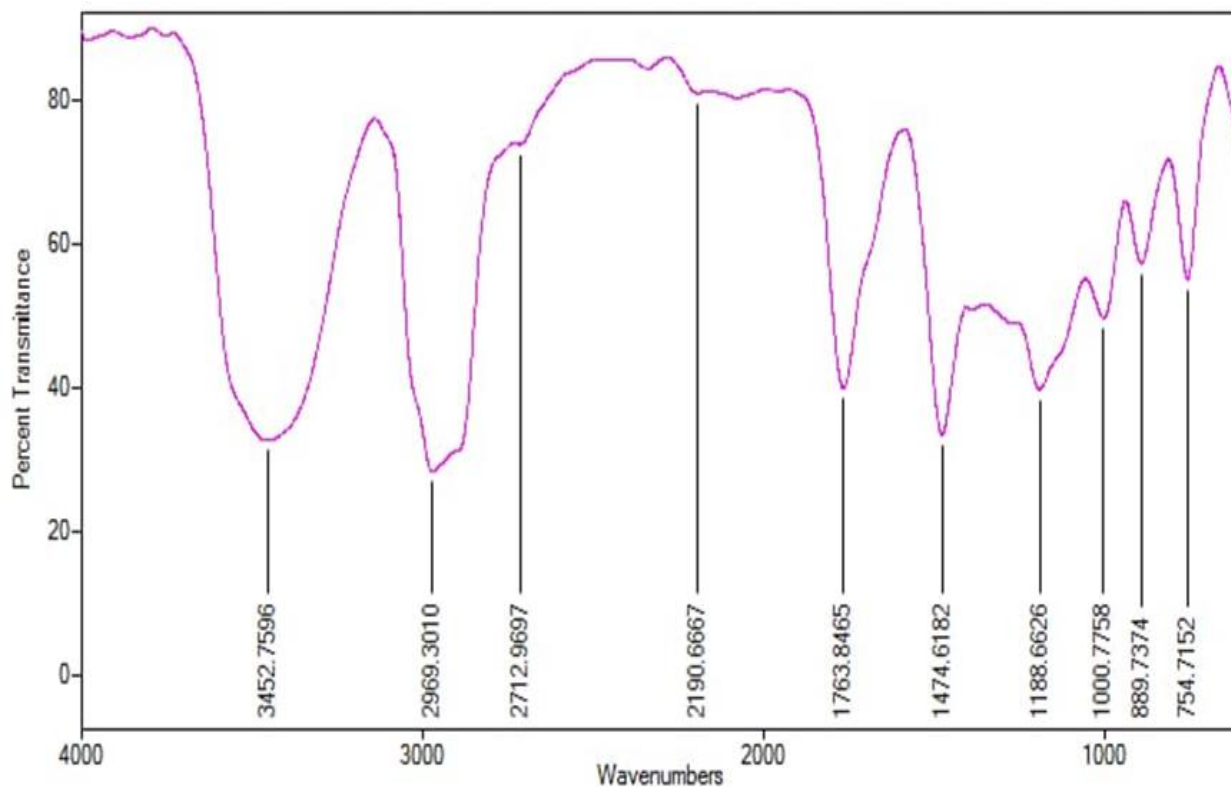


Figure 3: FTIR spectra of the extracted groundnut oil.

The presence of -CH and -OH functional groups with stretching vibrations is shown by the peak at 3452 cm^{-1} (Sumesh et al., 2020) which suggests the existence of organic molecules or components similar to C-H bonds at 2712 cm^{-1} (Wazir et al., 2020). The CH functional group with aliphatic stretching vibrations is shown by the peak at 2969 cm^{-1} (Thavasi et al., 2011) which suggests the presence of organic molecules with carbon-carbon single bonds. The peak at 2190 cm^{-1} corresponds to stretching vibrations in O=P-OH functional groups indicating the presence of phosphate-containing chemicals, which are common in biomolecules such as nucleotides and phospholipids (Erfani and Javanbakht, 2018). The presence of phosphate groups implies that biological or biochemical components may be present in the sample. The C=O functional group with stretching vibrations is shown by the peak at 1763 cm^{-1} . The existence of carbonyl compounds, such as aldehydes, ketones, and carboxylic acids, indicates the presence of carbonyl compounds as shown in the 1474 cm^{-1} peak associated with -CH₂ functional groups with symmetric bending vibrations.

4. Conclusion

In this study, the optimal oil yield extracted from groundnut was reported using the RSM Design of Experiment and gained significant information resulting in the efficient groundnut extraction oil yield technique. In addition, there is an established predictive models that accurately forecast oil yield based on varying extraction parameters. The FTIR study shows the various functional groups that are found on the oil which allows a better understanding of its organic composition. Further researchers should consider oil profiling techniques for comprehensive profiling of the extracted oil, use advanced analytical techniques such as Gas Chromatography-Mass Spectrometry (GC-MS) and High-Performance Liquid Chromatography (HPLC).

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