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PHYSICOCHEMICAL PROPERTIES OF REFINED SOYBEAN OIL AND DEODORIZER DISTILLATE AS BIODIESEL FEEDSTOCKS

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ABSTRACT
Biodiesel is an eco-friendly fuel that possesses the right qualities as a renewable source of energy including excellent lubricity, high flash point and biodegradability and this mainly depends on the raw material used. Hence, the physicochemical properties of refined soybean oil and soybean oil deodorizer distillate were determined to compare the most suitable for biodiesel production. The physicochemical parameters of the two oils which include: acid, iodine, saponification, free fatty acid and peroxide value, were determined by the official standard method of the Association of Official Analytical Chemists. The results revealed free fatty acid, peroxide, acid, iodine, and saponification content of soybean oil deodorizer distillate to be: 47.70±1.00 mg KOH/g, 248.55±0.89 meq O2/kg, 115.69±0.85 mg KOH/g, 5.58±0.51 g I2/100 g, and 64.95±0.65 mg KOH/g. While that of refined soybean oil showed the content to be 0.63±0.11 mg KOH/g, 4.08±1.02 meq O2/kg, 10.33±1.53 mg KOH/g, 11.58±0.38 g I2/100 g, and 34.70±1.00 mg KOH/g. These results indicate the free fatty acid, peroxide, acid and saponification content of soybean oil deodorizer distillate to be higher compared to refined soybean oil which suggests that it may require further pretreatment prior to transesterification. Although no transesterification reactions were conducted in this research, the high FFA Content and the origin of soybean oil deodorizer distillate as a byproduct of oil refining suggest its potential as a cost-effective and sustainable feedstock for biodiesel production, particularly within a waste valorization framework.

Keywords: Biodiesel, Feedstock, Free fatty acid, Refined soybean oil, Soybean oil deodorizer distillate

INTRODUCTION
Energy is a critical determinant in sustaining economic growth and ensuring the continued improvement of living standards. Globally, most energy is derived from petrochemical sources, including natural gas and coal (Xie *et al*., 2022). The transportation sector is the second-largest energy-consuming sector globally, following the industrial sector, and is responsible for approximately one-third of the total energy consumed globally. Over the years, there has been an increase in global energy demand because of fast industrialization and population growth (Holechek *et al*., 2022). More than 80% of this demand is generated from fossil fuels, which has led to the depletion of fossil fuels and increased environmental concerns, such as the emission of pollutants and greenhouse gases, which have stimulated the search for substitutes for renewable fuels that can meet the increasing energy demand. The deadly effects of fossil fuels on the environment are now known to people. Therefore, pursuing sustainable and clean energy sources has become significant for scientists and engineers with respect to SDG 7-Affordable and clean energy (Hassan *et al*., 2024). Biodiesel has attracted substantial attention in developed and developing countries as an excellent alternative to petroleum diesel fuel. Although renewable fuels hold promises for future economic growth, they must overcome the various scientific and technical hurdles to become environmentally viable and cost-effective to replace fossil fuels. This has made researchers and scientific communities worldwide focus on developing biodiesel and optimizing the process to meet the standards and specifications for the fuel to be used commercially (Akram *et al*., 2022).
Biodiesel is a renewable source of energy produced from different vegetable oils, animal fats and greases. Both the plant and animal feedstocks undergo conversion process in order to produce a suitable fuel for running the diesel engines (Das and Rokhum, 2024). Biodiesel can be used in compression ignition engines with minimal adjustments and blended in any proportion with petrodiesel. Biodiesel is being developed in 28 countries and is Europe's most extensively manufactured and used biofuel (Singh *et al*., 2021). The primary contributor to the production expenses is the choice of feedstocks, which account for 70-85 % of producing biodiesel (Pikula *et al*., 2020). Biodiesel can be obtained from three different generations of feedstocks. The first-generation feedstock is edible oils such as peanut, soybean, rapeseed, coconut, palm and sunflower oil (Singh *et al*., 2019). Second-generation feedstocks include non-edible oils like castor oil, jatropha oil and karanja oil (Pydimalla *et al*., 2023). The third-generation feedstocks are mostly waste oils, including used cooking oil and industrial byproducts like deodorizer distillates (Suzihaque *et al*., 2022).
The most common route for producing biodiesel is the conventional transesterification reaction, where triglycerides and short-chain alcohol (methanol) combine with a homogeneous or heterogeneous catalyst, forming fatty acid methyl esters (FAMEs) alongside glycerol as the by-product (Mathew *et al*., 2021). The transesterification method is a chemical process in which an alcohol displaces the ester from triglycerides which is the major component of the oil/fat, producing fatty acid alkyl esters and glycerol. This method is also known as alcoholysis. Similar to hydrolysis, this procedure uses alcohol in place of water (Babadi *et al*., 2022). Soybean oil is among the feedstocks used mostly in producing biodiesel due to its favorble acid profile and availability (Pikula et al., 2020). According to Nduka *et al*. (2021), research has shown soybean oil to be the most stable oil among the edible oils based on peroxide value which makes it to be considered in the production of biodiesel. Although the amount of waste oil used in the synthesis of biodiesel has increased to mitigate the cost of production in relation to SDG

12-Responsible consumption and production, and this has made soybean oil deodorizer distillate a suitable waste feedstock (Suzihaque *et al*., 2022). Soybean oil deodorizer distillate (SODD) is a cheap and readily available feedstock, a by-product obtained from soybean oil after the processing stages of deodorisation, stripping and refining (Benites *et al*., 2014). Besides the fact that soybean oil deodorizer distillate is regarded as an industrial waste used in biodiesel production, they are also valuable for the soap and food industries and as additives (Lv *et al*., 2021).

Despite being waste oil, soybean oil deodorizer distillate (SODD) contains significant quantities of free fatty acids and residual triglycerides, making it a potential raw material for producing biodiesel (Shaah *et al*., 2021). More research on producing biodiesel with the use of soybean oil deodorizer distillate (SODD) as a feedstock needs to be conducted alongside crude or refined soybean oil. This will help in waste management and the production of more environmentally-friendly biodiesel (Yin *et al*., 2015). In countries like Nigeria, where the quality of feedstocks was found to be the major issue for the production of biodiesel, waste products such as SODD remain underutilized and should be considered (Igwebuike, 2023). However, detailed assessments of their physicochemical properties: such as acid value, free fatty acid content, iodine value, peroxide value, and saponification value are essential to determine their suitability as biodiesel feedstocks (Maroa and Inambao, 2020). Understanding these properties is crucial for optimizing the transesterification process and ensuring compliance with biodiesel standards. Therefore, the objective of this research is to compare the physicochemical properties of refined soybean oil and soybean oil deodorizer distillate to evaluate their potential for biodiesel production.

MATERIALS AND METHODS

Materials

The refined soybean oil and SODD (soybean oil deodorizer distillate) feedstocks were obtained from a local vegetable oil industry in Sagamu, Ogun State, Nigeria. The samples were collected in clean, airtight containers, labeled appropriately, and stored at 4 ^oC to prevent oxidation until analysis. All the reagents, chemicals used in this research were of analytical grade and obtained from BDH Chemical Ltd (Poole, England).

Physicochemical Analysis

The physicochemical parameters determined include: free fatty acid value, peroxide value, acid value, saponification value, and iodine value. Each analysis was performed in triplicate, and appropriate blank determinations were conducted for accuracy.

Determination of Free Fatty Acid Value

The procedure described by Febrianto *et al*. (2020) was followed in determining the free fatty acid value. Approximately 3 g of refined soybean oil and SODD were accurately weighed into a 250 mL Erlenmeyer flask, previously considered empty. Then 30 mL of ethanol was added and 2 mL of phenolphthalein indicator was introduced into the oil. The mixture was titrated against 0.1 N NaOH until a persistent pink colour appeared. Blank titrations were also conducted. For every sample, there were three titrations. The FFA value (expressed as oleic acid, 28.2 g/mol) was calculated using the formula:

$$\text{Free fatty acid} = \frac{V \times N \times BM}{m \times 1000} \quad (1)$$

Where:

V= volume of NaOH titrated(mL);
N= Normality of NaOH;
BM = molecular weight of fatty acid (28.2 g/mol);
m = Mass of oil sample.

Determination of Acid Value

Using the procedure outlined by Esan *et al*. (2024) the acid value was determined. 100 mL of ethanol was added to each 1 g of refined soybean oil and SODD, which were then weighed and dissolved in the flask. After adding two drops of phenolphthalein, the mixture was titrated with 0.1 N potassium hydroxide solution (KOH) until a faint pink endpoint was achieved. Blank titrations were performed. Each sample underwent three iterations of the titration. We used the following formula to determine each sample's acid value:

$$\text{Acid value} = \frac{56.1 \times v \times c}{m} \quad (2)$$

Where:

56.1 = Equivalent weight of KOH;
V = Volume of KOH titrated (mL);
C = Concentration of KOH (0.1N);
m = Mass of the oil sample (grams).

Determination of Iodine Value

The Hanus iodine solution method was used to determine the iodine value (Hilp, 2002). A 0.25 g of refined soybean oil and SODD were weighed accurately into a conical flask of 500 mL and distilled in 10 mL of chloroform. A measuring cylinder was used to add 25 mL of Hanus iodine solution and allowed to stand in the dark for 30 minutes, shaking occasionally (every 5 minutes) for an accurate result. Then 10 mL 15 % KI solution was added, after which it was shaken thoroughly, and 100 mL fresh boiled and cooled H_2O was added. The solution was then titrated with standard 0.1 N $Na_2S_2O_3$, which was added gradually while shaking continuously, until the yellow solution became nearly colorless. Afterwards, 0.5 mL of starch indicator was added and it was titrated continuously till the blue colour vanished completely indicating the end of the reaction. The mixture was shaken vigorously to ensure any remaining iodine in the chloroform solution was absorbed by the KI solution. The blank test was carried out in addition to the sample determination. Each sample underwent two titrations, and the iodine value was calculated using the formula below:

$$\text{Iodine value} = \frac{(B-S) \times N \times 12.69}{Wt.\ of\ sample} \quad (3)$$

Where:

B = volume of blank solution (mL);
S = volume of standard $Na_2S_2O_3$ (mL);
N = Normality of $Na_2S_2O_3$.

Determination of Saponification Value

The method described by Triyasmono *et al*. (2022) was used to calculate the saponification value. Approximately 5 g of refined soybean oil and SODD were weighed into 50 mL of alcoholic hydroxide (KOH + ethanol) in a round-bottom flask. After adding a few drops of the phenolphthalein indicator, 0.5 N HCl was used to titrate the solution until the disappearance of the pink colour which indicates the saponification value. A blank determination (without oil) was conducted under the same conditions. The following formula was used to determine the saponification value for each sample:

$$\text{Saponification value} = \frac{28.05 \times (Vb - Vs)}{Ws} \quad (4)$$

Where:

V_b = Volume of standard HCl solution used for blank test (mL);

V_s = Volume of standard HCl solution used for sample (mL);
W_s = Weight of oil sample.

Determination of peroxide value

The methodology outlined by Hardy & Barrows (2003) was used to calculate the peroxide value. A conical flask containing 12 g of refined soybean oil and SODD was filled with 30 mL of a solvent mixture of glacial acetic acid and chloroform (3:2) and shaken vigorously. Then 1 mL of saturated KI was added, followed by the addition of 30 mL of distilled water and vigorously shaken. After 1 minute, 0.5 mL of starch indicator was added, and the mixture was titrated using a 0.01 N $Na_2S_2O_3$ solution until the black colour turned colourless. Alongside the oil samples, a blank was carried out to correct the values. Each sample's peroxide value was determined using the formula below:

$$\text{Peroxide value} = \frac{V \times N \times 1000}{Ws} \quad (5)$$

Where:
V = Volume of standard $Na_2S_2O_3$ (mL);
N = Normality of $Na_2S_2O_3$;
Ws = weight of oil.

Statistical Analysis

All experimental results were expressed as mean ± standard deviation (SD) of three replicates. Data were statistically analyzed using GraphPad Prism version 5.0 (GraphPad Software, Inc., La Jolla, CA, USA). Comparisons between refined soybean oil and SODD were made using unpaired Student's *t*-tests, with significance considered at $p < 0.05$.

RESULTS AND DISCUSSION

The physicochemical parameters of refined soybean oil and soybean oil deodorizer distillate in this research are displayed in Table 1 below. Each result was expressed as the mean ± standard deviation (n = 3). Free fatty acid value indicates the level of free fatty acid in the feedstock. The determination of FFA content is an important analysis for assessing the quality of the oil, as well as monitoring its degradation (Bouaid *et al*., 2016). The FFA value obtained for refined soybean oil was 0.63 ± 0.11 mg KOH/g while that of soybean oil deodorizer distillate was 47.70 ± 1.00 mg KOH/g ($p < 0.01$). The FFA value for refined soybean was low which is within the typical range for high-quality edible oils, indicating effective neutralization during the refining process. The high FFA in SODD is attributed to thermal degradation and significant hydrolysis of triglycerides during the deodorization process. This makes SODD more suitable for biodiesel production via acid-catalyzed esterification prior to transesterification (Lv *et al*., 2021). However, the FFA content of refined soybean oil in this research is close to that reported in Dave *et al*. (2014), which varied between 0.03 and 1.23 mg KOH/g.

Acid value is a key parameter that monitors the level of corrosiveness of the feedstock, so as to know whether it will require a neutralization process to reduce the acidity. This determines the quality of biodiesel produced (Wazed *et al*., 2023). The acid value obtained for the two oils differed significantly ($p < 0.01$), with 10.33 ± 1.53 mg KOH/g for refined soybean oil and 115.69 ± 0.85 mg KOH/g for soybean oil deodorizer distillate.

Table 1: Physicochemical Properties of Refined Soybean Oil and Soybean Oil Deodorizer Distillate

Parameters	Refined soybean oil	Soybean oil deodorizer distillate (SODD)
Free fatty acid value (mg KOH/g)	0.63 ± 0.11	47.70 ± 1.00
Acid value (mg KOH/g)	10.33 ± 1.53	115.69 ± 0.85
Peroxide value (meq O_2/kg)	4.08 ± 1.02	248.55 ± 0.89
Saponification value (mg KOH/g)	34.70 ± 1.00	64.95 ± 0.65
Iodine value (g I_2/100 g)	11.58 ± 0.38	5.58 ± 0.51

Each value represents the mean ± the standard deviation based on three independent measurements (mean ± SD, n = 3)

The acid value for refined soybean oil revealed to be in alignment with the acceptable limit for edible oils which is ≤10, indicating minimal free acid content and effective refining. In contrast, the high acid value in SODD suggests the need for pre-treatment to reduce acidity and improve biodiesel yield (Ibeto *et al*., 2012). Moreover, the acid value of refined soybean oil in this research is comparable to that of Cashew nut seed oil and palm kernel seed oil reported in Evbuowman *et al*. (2013) with average values of 10.70 and 10.71 mg KOH/g respectively.

Iodine value is an important property that shows the unsaturation level (that is the total number of double bonds) in the oil samples. The iodine value obtained for refined soybean oil was 11.58 ± 0.38 g I_2/100 g, which indicates a higher degree of unsaturation, thereby suggesting a greater proportion of unsaturated fatty acid composition in the oil. Soybean oil deodorizer distillate shows a low iodine value of 5.58 ± 0.51 g I_2/100 g, suggesting that the deodorization process has removed some of the unsaturated components which signifies the presence of more saturated fatty acids in the distillate. The iodine value obtained in this research is lower than that of jatropha kernel obtained in Akbar et al. (2009) which is 103.62 ± 0.02 g I_2/100 g. Although the EN 14214 specification for iodine value (<120 g I_2/100 g) applies to biodiesel fuel rather than feedstocks, the values reported in this research are within a suitable range for processing (Osarumwense *et al*., 2020). The iodine value of refined soybean oil was also close to almond seed oil which is an edible oil reported by Ogunsuyi & Daramola (2013) with an average value of 12.46 g I_2/100 g.

Saponification value is an index that shows the amount of potassium hydroxide (KOH) needed to saponify the oil and it also estimates the average molecular weight of the fatty acid present in the oil. A high saponification value indicates a low average fatty acid and a low saponification value suggests superior quality (Olabanji *et al*., 2016). The saponification value obtained for refined soybean oil was 34.70 ± 1.00 mg KOH/g, which is typical for oils that have a comparatively large amount of long chain fatty acids while that of soybean oil deodorizer distillate was higher (64.95 ± 0.65 mg KOH/g) which indicates that it contains more short and medium-chain fatty acids compared to the refined oil. The saponification value of SODD in this research was found close with the study of Ibeto *et al*. (2012) which found the saponification value of *Luffa cylidrica* seed oils to be 65.92 mg KOH/g and the saponification value above 100 has the higher tendency to form soap and difficulty in the separation of products thereby causing low FAME yield. However, the saponification value of SODD reported in this research was lower than 100 which indicates that it does not tends to form soap which makes it suitable for the production of biodiesel.

Peroxide value indicates the extent of oxidation (indicating how oxidized the oil is) affecting the flavour, colour and shelf life. The higher the peroxide value, the more exposed it is to deterioration which also affects the oil's shelf life (Sana & Shouriehebal, 2023). The peroxide value obtained for refined soybean oil was 4.08 ± 1.02 meq O_2/kg which indicates that the oil has been well-protected from oxidation during refining, thereby preserving their quality while that of soybean oil deodorizer distillate was 248.55 ± 0.89 meq O_2/kg ($p < 0.01$), reflecting extensive oxidation during high-temperature deodorization. While such a value renders it unsuitable for food use, it supports the valorization of SODD as a waste-based biodiesel feedstock, where oxidative stability is less critical than in edible applications (Sana & Shouriehebal, 2023). The range of peroxide values for non-edible oil is between 4.36-9.82 meq O_2/kg, which was higher than the refined soybean oil in this research and this confirms the suitability of soybean oil deodorizer distillate for biodiesel production without affecting the consumption factor. The peroxide value of refined soybean oil in this research is comparable to the result obtained in Eze, (2012) for Bambara groundnut oil with an average value of 4.3421 meq O_2/kg.

CONCLUSION

This research investigated the physicochemical characteristics of refined soybean oil and soybean oil deodorizer distillate (SODD) to assess their suitability as a feedstock for producing biodiesel. All the physicochemical properties determined in this research including free fatty acid, acid, peroxide, iodine, and saponification content were determined to identify the feedstock that meet ASTM D6751 standards required for biodiesel production. The findings from this research indicate that while refined soybean oil is chemically stable, its low free fatty acid content and higher market cost limit its feasibility as a biodiesel feedstock. In contrast, SODD, a low-cost by-product of the soybean oil refining process, exhibits significantly higher free fatty acid content, making it more suitable for biodiesel production via acid-catalyzed esterification. This suggests a promising opportunity for waste valorization and resource efficiency, contributing to circular economy initiatives and the reduction of industrial waste in alignment with SDG-12 (Responsible Consumption and Production).

Importantly, the conversion of SODD into biodiesel has the potential to enhance the economic and environmental sustainability of biofuel production, especially in regions where soybean oil processing generates large volumes of by-products. Future research should focus on process optimization for biodiesel production from SODD, including the selection of effective acid or heterogeneous catalysts, evaluation of reaction parameters (such as temperature, molar ratio, and reaction time), and investigation of the resulting fuel properties from this distillate to further assess its viability as an alternative energy source.

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