



# Physicochemical assessment of vegetable oil used to fry some Nigerian food items

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**Abstract.** The escalating global energy demand, projected to rise from 442 EJ in 2023 to between 623 and 710 EJ by 2035, has intensified the search for sustainable energy alternatives to fossil fuels. Biodiesel, particularly from waste cooking oil (WCO), presents a promising solution due to its affordability and environmental benefits. This study investigates the physicochemical properties of neat and waste sunflower oil used to fry bean cakes (Akara), fish, and puff-puff in Nigeria, aiming to identify the impact of frying duration and fried substances on oil quality. The study's findings reveal significant changes in the oil's properties post-frying, with notable variations in pH, moisture content, boiling point, density, viscosity, flash point, saponification value, protein content, fat content, acid value, free fatty acids (FFA), and fatty acid methyl esters (FAME). The results indicated that the moisture content increased from 34.6% in neat oil to 47.9% in oil used for frying bean cakes, while the boiling point decreased from 243 °C to 226 °C in oil used for frying fish. The kinematic viscosity and density also varied, with the lowest viscosity (26.48 mm<sup>2</sup>/s) observed in oil used for frying fish and the highest density (911 kg/m<sup>3</sup>) in oil used for frying bean cakes. The flash point of WCO samples ranged from 248 to 258 °C compared to 260 °C in neat oil, and the saponification values varied significantly, indicating different levels of oxidation and deterioration. The study concludes that frying fish has the most detrimental effect on oil quality, as evidenced by the highest acid value (7.2 mgKOH/g) and significant changes in FFA and FAME concentrations. These insights into the physicochemical alterations of WCO highlight the potential for its optimized use in biodiesel production, offering a sustainable energy source while addressing waste disposal challenges.

**Keywords:** Waste vegetable oil, Physicochemical properties, Waste cooking oil (WCO), Biodiesel feedstock, Characteristic properties.

## 1 Introduction

Over recent decades, the energy demand has surged, driven by population growth and ongoing industrial expansion. The global economy is deeply dependent on fossil fuels to power industries and supply essential raw materials. As evidence of this reliance, global *Total Final Energy Consumption* surged to 442 EJ in 2023 [1]. According to the International Energy Agency, projections indicate that this upward trend will persist, with energy demand expected to climb further to a range of 623–710 EJ by 2035 [1, 2]. However, this rise in energy consumption has come at a cost, with mounting evidence revealing the adverse environmental impacts of continued reliance on fossil fuels. Consequently, there has been a global shift towards embracing renewable energy sources [3, 4]. A shift worth noting is

the development of Biodiesel which garnered much interest and study recently. In light of the volatility of the price of diesel fuel derived from petroleum and the lack of robust regulations aimed at reducing the use of fossil fuels, biodiesel fuel is affordable, environmentally benign, and sustainable [5]. The search for a conveniently available, affordable, and environmentally acceptable feedstock for sustainable biodiesel production has become essential due to the growing demand for environmentally friendly and sustainable alternatives to fuel derived from fossil fuels [6]. Literature has shown that both edible and non-edible oils can be utilized as feedstock for biodiesel [2, 7]. Albeit, the commercialization of biodiesel faces a significant hurdle due to its comparatively higher production cost compared to petroleum-based diesel. Typically, biodiesel costs about one and a half times more than petroleum-based diesel, contingent upon the feedstock oils utilized [8]. A substantial portion, approximately 70–95%, of the total production

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**Table 1.** Global vegetable oil consumption (million metric tonnes) [11].

Year	2015/16	2017/18	2019/20	2021/22	2023/24
Palm oil	59.38	65.99	71.07	71.12	77.99
Soybeans oil	52.09	54.41	56.97	59.3	60.72
Rapeseed oil	28.27	28.92	28.18	29.44	32.82
Sunflower seed oil	15.02	17.46	18.92	17.88	20.27
Palm Kernel oil	7	7.8	8.15	8.29	8.89
Peanut oil	5.41	5.71	6.22	6.51	6.29
Cottonseed oil	4.36	5.03	5.07	4.97	4.96
Coconut oil	3.24	3.32	3.52	3.51	3.69
Olive oil	2.81	2.87	3.03	3.15	2.36

cost of biodiesel is attributed to the expense of raw materials, such as vegetable oil or animal fats [8]. Consequently, the utilization of waste cooking oil (WCO) presents a promising solution to this challenge, as it is obtainable at 40% of the pure oil [9, 10], thereby potentially reducing the overall cost of biodiesel production significantly even with the ever-increasing global consumption of edible oil [6] (see Table 1).

Utilizing waste cooking oil (WCO) as feedstock mitigates the disposal challenges associated with used frying oils. Every year, tons of waste oil are discarded, leading to water and soil pollution, health concerns, and disturbances in aquatic ecosystems [9]. WCO is generated from repeated cooking or frying using edible vegetable oils (such as palm, soybean, sunflower, cottonseed, olive, palm kernel, rapeseed) or animal fat (butter, fish oil, and tallow) [12]. The oil experiences significant physical and chemical changes such as increased viscosity, specific heat, altered surface tension, colour change, and increased foaming tendency due to excessive heating which usually ranges between 160 and 200 °C [5, 13].

Literature highlights three primary reactions occurring in frying oil: thermolytic, oxidative, and hydrolytic, which influence the oil's composition and the quality of fried foods [14, 15]. While further exploration of the different properties of WCO from different sources was also explored and reported [16], the effect of heating duration was documented [13], there exists a gap yet to be explored about the effect of fried substance, frying duration and the frying temperature on the property of WCO from a single edible cooking oil source.

The aim is to determine how the duration of usage and the type of food fried in the oil affect some properties and the FA composition of WCO compared with their neat vegetable oil source. Thus, the objective of this research is to compare the properties of WCO from the same source but utilized for different time durations and different fried substances and compare the composition with the neat vegetable oil. Though some studies have been conducted on the effect of usage on the physical and chemical properties of some Nigerian vegetable oil, in the opinion of the authors, there has been no study to investigate the effect of usage of Nigerian to fry bean cake, fish, and puff puff. This forms the novelty of the current study. The scope of

**Table 2.** Description of the samples.

Sample notation	Description
A	Waste sunflower used to fry bean cake (Akara)
F	Waste sunflower oil used to fry fish
P	Waste sunflower oil used to fry puff-puff
N	Neat sunflower oil

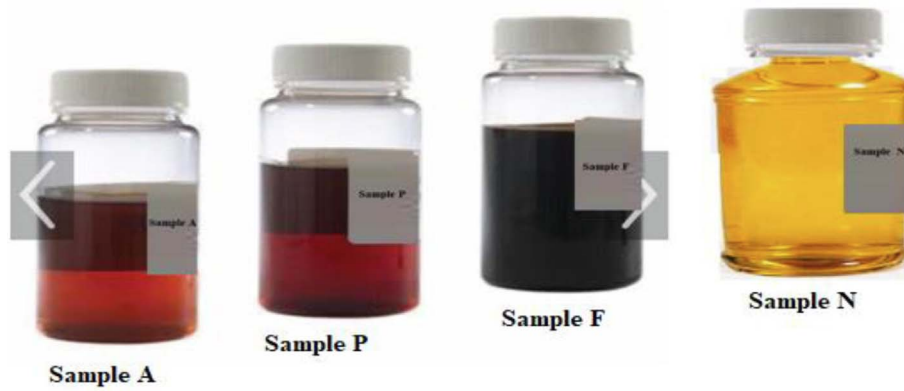
the study is limited to the determination of the physical and chemical properties of the Nigerian sunflower oil used to fry bean cake, fish, and puff puff. The thermal and spectrographic analyses, fatty acid composition, and conversion of the waste frying oil to other products are outside the scope of the current study. The outcome of the study will provide data on the properties of waste sunflower oil used to fry bean cake, fish, and puff puff to researchers in the food and waste conversion research domain.

## 2 Materials and method

This research employs a case study approach to investigate the effect of different common fried food types on the characteristic properties of WCO in the context of Nigeria. The study incorporates a combination of laboratory analyses, statistical assessments, and sensory evaluations to examine the relationship between fried foods and WCO quality comprehensively.

### 2.1 Material selection and collection

A diverse range of commonly consumed fried foods/snacks from Nigerian cuisine are selected based on their cultural significance and popularity. The explored fries include bean cake (akara), fish, and puff-puff. Three (3) litres of sunflower oil were distributed for use in three (3) frie establishments. It should be noted that the same frying conditions including frying duration of 20 h, and frying temperature ranging between 160 °C and 180 °C were maintained. Frying experiments were conducted in triplicate.



**Figure 1.** Vegetable oil samples.

The WCO samples were labelled as described in Table 2 and shown in Figure 1.

## 2.2 Frying procedure

The bean cakes were prepared by soaking dried black-eyed peas for 30 min to soften the bean seed coat. Subsequently, the soaked beans were blended with onions, peppers, and seasonings to achieve a smooth batter before frying. Puff puff was made by mixing flour, sugar, yeast, salt, and water to form a thick, smooth batter, which was left to ferment for a period to enhance flavour and texture prior to frying. The fish, on the other hand, underwent cleaning and seasoning with salt, pepper, and flour to enhance crunchiness before frying. In all frying processes, the oil samples were preheated to 175 °C to prevent oil-soaking of the food. Each frying cycle lasted approximately  $6 \pm 2$  min. Throughout a 10-h sampling period, a total of 8 kg of bean cakes, 5 kg of puff puff, and 7 kg of fish were fried. Between frying cycles, no fresh oil was added. This decision was made to mitigate the formation of polar compounds and free fatty acids, as noted by Jurid *et al.* [17].

## 2.3 Physico chemical properties method

Before characterisation of the WCO samples were heated to 175 °C for 10 min to eliminate moisture. The samples were air-cooled afterward and stored undisturbed in an airtight plastic container for three (3) weeks to allow sedimentation of food particles.

The conventional physicochemical attributes of the specimens were acquired through the utilization of pertinent methodologies, procedures, and instrumentation. These encompassed the assessment of various parameters including free fatty acid (FFA) content, boiling point, flash-point, viscosity, fatty acid methyl ester (FAME) concentration, peroxide value (PV), Iodine Value, Specific Gravity, and Moisture Content.

### 2.3.1 Moisture content

10 g of oil sample was placed in a weighed crucible. The samples were dried for 1 h to constant weights in an oven set at 105 °C and then allowed to cool in desiccators

for 15 min and finally, the difference was calculated using equation (1).

$$\% \text{ Moisture} = \frac{W1 \times 100}{W2} \quad (1)$$

where, W1 = weight loss (g) upon drying, W2 = weight (g) of the oil sample.

### 2.3.2 Boiling point

The distillation method was utilized in determining the boiling point of the samples. This method involves subjecting the oil to gradual heating while continuously measuring the temperature. As the oil undergoes vaporization, the temperature is observed to ascertain the point at which it remains constant. This stabilization of temperature signifies the attainment of the boiling point. The boiling point is derived directly from the thermometer readings obtained at this juncture.

### 2.3.3 Specific gravity

A dry pycnometer was used to determine specific gravity. Specific gravity was measured by the Relative Density of oil to water. Distilled water was added to the pycnometer followed by measurement using an electronic balance. Similarly, oil weight was measured. Care was taken to avoid leakage of air into the pycnometer. The specific gravity value was calculated using equation (2):

$$\text{specific gravity} = \frac{\text{Weight of the oil (g)}}{\text{Weight of distilled water (g)}} \quad (2)$$

### 2.3.4 Flash point

Two primary methods are employed for flash point determination: open and closed. However, in the context of these experiments, the open-cup method was adopted.

In this procedure, a 25 ml beaker housing the oil sample was situated upon an electric heating source. A thermometer was vertically suspended within the beaker, positioned with its bulb approximately 6.35 mm above the base. The sample underwent rapid heating until reaching approximately 40 °C, followed by controlled heating at a rate of

60 °C per minute. A flame was continuously directed toward the vapour space above the beaker until the ignition of the oil occurred. Subsequently, the temperature indicated by the thermometer at the instance of ignition was meticulously recorded.

### 2.3.5 Saponification value

To ascertain the saponification value, 1.0 g of oil sample is placed in a conical flask, along with 15 mL of 1 M KOH and 10 ml of distilled water. The mixture is then heated under a reserved condenser for 30–40 min to guarantee that the sample is completely dissolved. Phenolphthalein was added and titrated with 0.5 M of HCl until a pink endpoint was obtained when this sample had cooled. The identical time conditions were used to determine a blank.

### 2.3.6 Viscosity

Viscosity, an intrinsic characteristic of fluid substances, denotes the degree of resistance to flow they exhibit. Typically, a viscometer serves as the instrument of choice for quantifying viscosity.

In this study, the dynamic viscosity ( $\mu$ ) of waste fish oils (WFOs) was determined utilizing a Brookfield Viscometer outfitted with a thermo-container and programmable temperature control system. The thermo-container featured a sample chamber and a spindle, while cooling was facilitated through a cooling plug assembly linked to a pressurized air nozzle. The temperature controller was programmed to execute a heating protocol, starting from 23 °C and progressing incrementally until reaching the smoke point of each oil, maintaining each temperature level for 5 min. Subsequently, dynamic viscosity measurements were conducted and converted to kinematic viscosity. The conversion was performed using equation (3).

$$\mu = v\rho \quad (3)$$

Where  $\mu$  represents the dynamic viscosity (mPa·s),  $v$  represents the kinematic viscosity (mm<sup>2</sup>/s) and  $\rho$  represents the density (g/cm<sup>3</sup>).

### 2.3.7 Free fatty acid (FFA) content

Titration methods are employed to determine the level of FFA, an indicator of hydrolysis of oils and fats. Thus, FFA is a key feature linked with the quality and commercial value of oils and fats. It is also a key health determinant as an elevated FFA level in the blood is thought to raise the risk of cardiovascular disease by causing insulin resistance (in some cases leading to diabetes), raising blood pressure, and other effects.

The Percentage FFA is determined by measuring one gram (1 g) of the oil sample was accurately weighed into a conical flask. This was followed by the addition of 10 cm<sup>3</sup> of neutralized 95% ethanol and Phenolphthalein. This was then titrated with 0.1 M NaOH, with constant shaking until a pink colour persisted for 30 s. The percentage of free fatty acid was calculated as shown in equation (4). Where: V is the Volume of NaOH, M is the Molarity of NaOH and CF is the conversion factor

$$\text{Free Fatty Acid} = \frac{V \times M \times CF}{\text{Weight of Sample}} \quad (4)$$

### 2.3.8 Fatty acid methyl ester analysis (FAME)

In this particular experimental protocol, 50 mg of the extracted fat content from the sample underwent saponification (esterification) for five (5) minutes at 95 °C, employing 3.4 ml of 0.5 M KOH in dry methanol. Following this, neutralization was achieved using 0.7 M HCl, and subsequently, 3 ml of 14% boron trifluoride in methanol was introduced. The resulting mixture underwent concentration for 5 min at a temperature of 90 °C to ensure completion of the methylation process. The Fatty Acid Methyl Esters were then extracted from the mixture using redistilled n-hexane. The resulting content was concentrated to a volume of 1 ml for subsequent analysis via gas chromatography, with an injection volume of 1  $\mu$ l into the injection port of gas chromatography with flame ionization detection (GC FID).

The gas chromatography conditions utilized for analysis are outlined in the attached analysis printout. Specifically, for the GC FID instrument, a Buck Scientific 910 model was employed, utilizing nitrogen as the carrier gas. The initial and final temperatures were set at 50 °C and 250 °C, respectively. The column type utilized was RESTEK 10 m MXT-2887, coupled with an FID detector. Hydrogen gas pressure, compressed air, and nitrogen gas were adjusted to 15 psi, 35 psi, and 10 psi, respectively, to optimize instrument performance.

### 2.3.9 Statistical analysis

The data were subjected to descriptive statistical analysis. The descriptive statistical analysis involves the calculation of the mean, standard deviation (SD), and coefficient of variation (CV %) of the data obtained from the results of the physicochemical properties.

$$CV\% = \frac{SD}{\text{Mean}} \times 100 \quad (5)$$

## 3 Results and discussion

The inherent properties of the neat sunflower oil and waste sunflower oil samples were investigated and subsequently documented in Table 3.

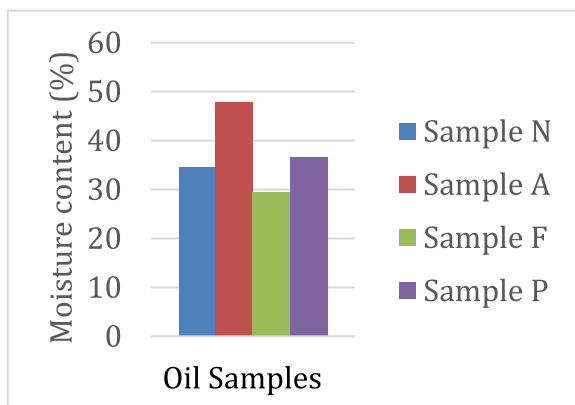
### 3.1 Moisture content of oil

The frying process entails various mass transfer phenomena, encompassing water loss, oil absorption, and heat transfer [18, 19]. Both food and oil moisture levels play crucial roles, expediting oil hydrolysis while concurrently offering protection against oil oxidation during frying [17]. Analysis of Table 3 and Figure 2 reveals discernible effects of moisture content in the fried food samples, exhibiting higher values compared to the pristine oil sample, which recorded a moisture content of 34.6%. Notably, Sample A displayed the highest moisture content at 47.9%, likely

**Table 3.** Characteristic properties of neat and waste cooking oil.

Properties	Unit	Samples				Mean of WCO	SD of WCO	CV% of WCO
		N	A	F	P			
pH		7.38	5.33	6.02	5.27	6.000	0.981	16.39
Water content	%	34.6	47.9	29.4	36.6	37.125	7.798	21.01
Boiling point	°C	243	237	226	235	235.25	7.042	2.99
Density	g/cm <sup>3</sup>	902	911	909	902	906	4.690	0.52
Kinematic viscosity@40 °C	mm <sup>2</sup> /s	34.35	30.5	26.48	32.9	31.058	3.440	11.08
Flash point	°C	260	256	248	258	255.5	5.260	2.06
Saponification value	mgKOH/g	188.9	208.9	310.63	193.7	225.53	57.37	25.44
Ash	%	0.18	0.06	0.06	0.33	0.158	0.128	81.37
Protein	%	5.88	1.82	2.69	1.83	3.055	1.927	63.08
Fat	%	33.6	40.27	39.8	38.26	37.983	3.045	8.02
Acid value	mgKOH/g	3.7	3.96	7.2	5.33	5.048	1.603	31.76
FFA	ppm	49.7	26.9	21.8	30.9	32.325	12.17	37.64
FAME	ppm	51.94	30.119	23.37	35.48	35.227	12.19	34.61

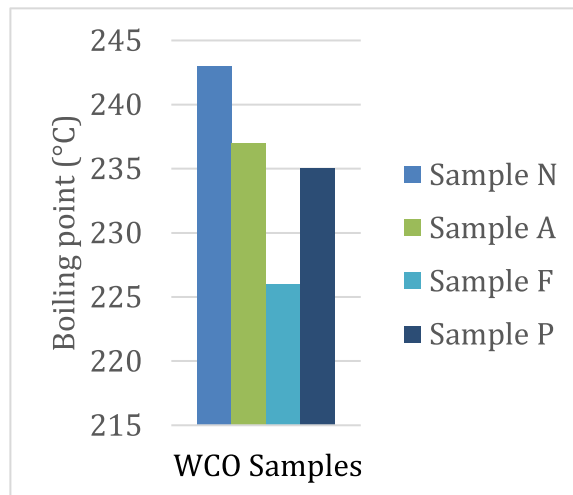
FAME = Fatty Acid Methyl Ester Analysis, FFA = Free Fatty Acid.

**Figure 2.** Moisture content against WCO samples.

stemming from its preparation method involving soaking the beans in water. Additionally, the average frying temperature for commercial bean cakes typically ranges between 140 and 175 °C due to challenges in regulating fire intensity and airflow. Furthermore, Sample A's extended frying duration exposes the oil to prolonged ambient humidity. Consistent with existing literature, the escalation in oil moisture content may result from exposure to food moisture and surrounding air humidity, thereby elevating the risk of oil rancidity and oxidative stress [20].

### 3.1.1 The boiling temperature

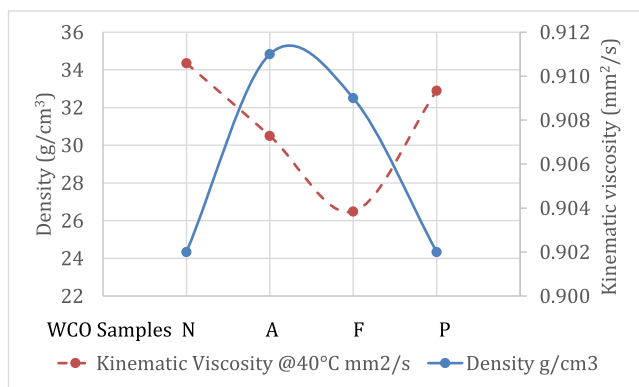
Figure 3 shows the graph of the explored WCOs against corresponding boiling point temperatures. The boiling point of edible oils is intricately linked to the degree of unsaturation present within the fatty acid composition [21]. Unsaturated fatty acids are characterized by the presence of one or more double bonds between carbon atoms within their carbon chains. These double bonds introduce kinks or bends

**Figure 3.** Boiling point versus WCO samples.

in the fatty acid structure, thereby reducing the intermolecular forces between adjacent fatty acid molecules. As a result, unsaturated fats have a lower boiling point compared to saturated fats, which lack double bonds and therefore exhibit a more linear molecular structure. As depicted in Table 3, a reduction in the boiling temperature as compared to the neat unused vegetable oil Sample N from 243 °C was observed with the lowest boiling temperature of 226 °C recorded for Sample F. This might be the effect of fish frying on the characteristic properties of edible oil has been documented after 10 h of use [22].

### 3.1.2 Kinematic viscosity and density

Oils consist of triglycerides (TGs), and their viscosity is contingent upon the composition of TGs within the oil. Variations in viscosity occur as a consequence of the



**Figure 4.** Kinetic viscosity and density versus WCO samples.

distinct arrangements of fatty acids along the glycerol backbone of the triglyceride molecule. Consequently, viscosity is intricately linked to the chemical characteristics of oils, including the length of fatty acid chains, molecular weight, degree of saturation or unsaturation, and temperature [23, 24]. It could be said that viscosity increases with molecular weight and decreases with increased level of unsaturation [21]. As observed in Table 3 and Figure 4, the kinematic viscosity of the investigated oil samples ranges between 26.48 and 34.35 mm<sup>2</sup>/s with the highest and lowest values recorded for Sample N and Sample F respectively. The comparatively lower viscosity observed for the explored WCO might indicate that they are lighter, and contain a high level of unsaturation in addition to a high level of suspended particles within the WCO. This signifies that Sample F is the most deteriorated.

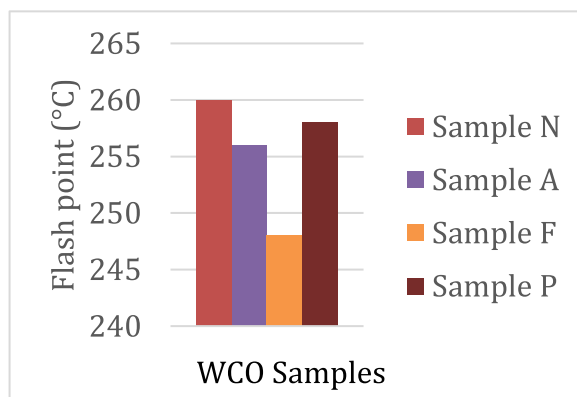
Comparatively, the observed densities recorded for the explored WCO (ranging between 0.902–0.911 g/cm<sup>3</sup>) are higher in value as compared to the unused oil (Sample N) at 0.902 g/cm<sup>3</sup>. The values obtained show that the most-dense oil is the Sample A. For this set of experiments, the increase in density might be due to the presence of suspended food particles in the oil in addition to the refined and unrefined characteristics of the oil [24].

### 3.1.3 Flash point

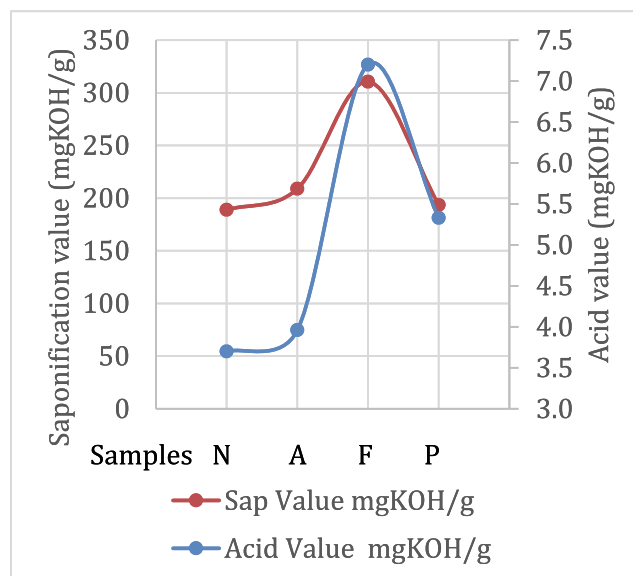
As shown in Table 3 and Figure 5, the flash point between 248 and 258 °C was recorded for the WCO and 260 °C for Sample N. For use as feedstock for biodiesel, a higher flash-point is desired as the risk associated with fuel transportation is greatly minimized [25]. This is attributed to the inability of combustible air-fuel vapour mixture to ignite below the flash point.

### 3.1.4 Acid value

As observed in Table 3 and Figure 6, a comparatively lower acid value of 3.7 mgKOH/g was recorded for Sample N which indicates higher stability of the oil over a long period and resistance against rancidity and peroxidation [24]. The recorded increase in the acid value for the WCO ranges from 3.96 to 7.2 mgKOH/g with the highest value at Sample F observed. The increment might be due to the



**Figure 5.** Flashpoint against WCO samples.



**Figure 6.** Saponification and acid value against WCO samples.

infusion of water during frying [26], especially for Sample F in which the sporadic deterioration of the oil due to fish frying has been documented by Manral *et al.* [22].

### 3.1.5 Saponification value

For the examined samples, the saponification values, as tabulated in Table 1 and depicted in Figure 6, were 188.9 mgKOH/g for the neat unused oil, while for the WCO oils 208.9, 193.7, and 310.63 mgKOH/g were recorded for Samples A, P, and F respectively. This divergence in saponification values suggests disparate levels of oxidation and deterioration among the samples. The higher saponification values observed in Samples A, P, and F may indicate increased volatility and potentially heightened energy-yielding potential due to the presence of lower molecular weight components [27]. Additionally, these findings align with the assertion by Pearson (1976) [28, 29] that oils with elevated saponification values tend to contain

higher proportions of lower fatty acids, a characteristic often associated with fats and hydrogenated oils. Notably, saponification values exceeding 200 mgKOH/g are typically observed in fats and hydrogenated oils, while oils typically exhibit values below 195 mgKOH/g.

### 3.1.6 Free fatty acid (FFA)

The observed FFA values for the explored oils are reported in Table 1. Notably, for the unused oil, a comparatively higher value of 49.7 ppm was observed compared to WCO, in which 26.9 ppm, 21.8 ppm, and 30.9 ppm were recorded for Samples A, F, and P respectively. This significant difference in FFA content can be attributed to the high cooking temperature, impurities, and moisture present in WCO [30, 31], which promote the hydrolysis of triglycerides followed by the conversion into FFAs. It's important to recognize that while FFAs in WCO are advantageous for soap manufacturing due to their reactivity with catalysts, this poses challenges in biodiesel production [32]. Additionally, the presence of FFAs can lead to changes in acidity and viscosity levels.

## 4 Conclusion

The physicochemical analysis of the neat and waste cooking oil samples revealed that the quality of frying oil deteriorates with use viz-a-viz, the pH value, water content, coiling point, density, kinematic viscosity, flash point, saponification value, ash content, protein, fat, acid value FFA and FAME. It is ascertained that the fried substances affect the used oil sample property differently. The findings, however, showed there were significant differences in the physicochemical properties when comparing the WCO types with Sample F demonstrating the detrimental effect of fish on oil.

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### Conflicts of interest

The authors declare that they have no conflicts of interest to report regarding the present study.

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