Physicochemical and Trace Metals Characterization of Edible Vegetable Oils

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Author's contribution

The sole author designed, analysed, interpreted and prepared the manuscript.

Original Research Article

ABSTRACT

Edible vegetable oil samples purchased in Akure, Nigeria, were subjected to physicochemical properties and trace metals analysis. The physiochemical parameters such as acid value, iodine value and saponification value were carried out by standard methods and the levels of the trace metals (copper, zinc, iron, manganese, cadmium, nickel, lead and chromium) were determined by the atomic absorption spectrophotometric method after wet digestion. The range of mean values obtained for the physicochemical parameters of the oils: acid value (3.59 - 9.65 mg KOH/g); iodine value (32.99 - 75.04 g I_2/100g); and saponification value (187 – 207 mg/KOH/g), were within acceptable values for edible oils. The trace metals in the samples were in the range of values (mg/kg):0.10 - 0.60 (Cu); 0.07 - 2.49 (Zn); 0.13 - 0.55 (Fe); 0.01 - 0.48 (Mn); 0.01 - 0.09 (Cd); 0.01 - 0.08 (Ni); 0.07 - 0.26 (Pb); and 0.01 - 0.07(Cr). The concentration of trace metals such as Pb and Cd in the edible oils were beyond established permissible levels.

Keywords: Vegetable oils; saponification; trace metals; physicochemical.

1. INTRODUCTION

Vegetable oils are composed primarily of glycerides of fatty acids being obtained from vegetable sources. They are oils extracted from seeds, or fruits. Soya bean oil, rapeseed oil, and cocoa butter are examples of seed oils. Olive oil, palm oil and rice bran oil among others are examples of oils from fruits. They are beneficial and popular due to their cholesterol-lowering

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effect [1] and are used in domestic and industrial food preparations (frying, baking, salad dressings, margarine and ice cream productions), as raw materials in the production of cosmetics and biofuels as renewable energy sources [2,3].

The physicochemical properties of any vegetable oils clearly distinguish their utilization as edible or raw materials for external use. Edible oils are required to have physicochemical properties that indicate their quality in terms of consumer standards as regards their freshness, texture, storability and stability in the food products in which they are utilized [4]. Such properties include acid value (AV), iodine value (IV), saponification value (SV), refractive index, specific gravity, viscosity, and unsaponifiable matter which are used to monitor the compositional quality of oils [5-8].

The toxicity can be evaluated by determination of levels of trace metals in the oils. Levels of trace metals like Copper (Cu), Zinc (Zn), Iron (Fe), Manganese (Mn) and Nickel (Ni) are known to increase the rate of oil oxidation while other elements such as Arsenic (As), Cadmium (Cd) and Lead (Pb) are very important on account of their toxicity and metabolic roles [9]. Being of natural sources, vegetable oils contain trace amounts of metals, which at higher concentrations increase the rates at which they are oxidized. Metals present in vegetable oils can be from mineral uptake from the soil on which the plant is grown as well as from other sources such as agrochemicals utilized in agricultural applications, the use of metal equipment in the production processes, storage and transportation [10,11,12].

This study is aimed at evaluating the quality of edible palm olein and soybean oils through the physicochemical and trace metal content of the oils as sold in Akure, Nigeria. The two varieties of oils were selected based on their wide distribution and availability in the study area.

2. MATERIALS AND METHODS

2.1 Sample Collection

Edible vegetable oils made from refined palm olein or palm oil and soybeans were purchased from retail outlets in Akure, Ondo State, Nigeria and were coded as A1-A5 for five different brands of soybean oils and B1-B5 for five different brands of palm olein. The collected oil samples were stored away from sunlight until

analysis was carried out. The edible oils were made in CODEX-certified manufacturing facilities with National Agency for Food and Drug Administration and Control (NAFDAC) approval. The information on the packages did not specify the inclusion of additives and preservatives.

2.2 Reagents and Apparatus

All reagents used in this work were of analytical reagent grade unless otherwise stated. All laboratory glassware was cleaned with detergent solution, rinsed with distilled water, soaked in dilute nitric acid then rinsed thoroughly with deionized water and dried.

2.3 Methods

2.3.1 Physicochemical analysis

2.3.1.1 Specific gravity

The specific gravity of oil was determined using density bottle of 50ml capacity. The initial weight, W_1, of the bottle was noted, it was filled with the oil, covered with air stopper and reweighed, W_2. The density bottle was washed, drained, dried and weighed. It was filled with water, covered with its stopper and weighed, W_3. The experiment was carried out in triplicates. The specific gravity was calculated using equation 1.

$$Specific\ gravity = \frac{w_2 - w_1}{w_3 - w_1} \qquad (1)$$

Where,

W_1 is the weight of the density bottle,
W_2 is the weight of container and
W_3 is the weight of container with water

2.3.1.2 Refractive index

Refractive indices of the oils were determined using a refractometer in triplicates. The oil was placed on the cleaned and dried lens. The prism was closed and the instrument and lighting were adjusted to obtain most distinct reading possible. The refractometer number was obtained from which the refractive index was determined. The refractive index was calculated using the equation 2 [13].

$$R = R^1 + K(T^1 - T^0) \qquad (2)$$

Where,

R = Reading of the refractometer reduced to the specified temperature T^0C,

R^1 = reading at $_{T1}°$,

K = 0.0065 for fats and 0.00085 for oil (refractometer),

T^1 = temperature at which the reading R^1 is taking and

T^0 = absolute temperature (20°C)

2.3.1.3 Acid value

The acid value (AV) was determined by direct titration of the oil sample in alcoholic medium, against standard potassium hydroxide solution, in the presence of phenolphthalein as an indicator. About 1.0 g of the oil was dissolved in the neutralized solvent mixture and also warmed on hot plate for few minutes and removed from heat. Two drops of phenolphthalein indicator were added to the solutions, and the solution was titrated against standardized 0.1 M potassium hydroxide. The yellow colour of the solution became milky immediately the indicator was added and this later turned pink at the end - point. The process was carried out in triplicates and calculated using equation 3 [14].

$$Acid\ value = 56.18\ \times titre\ value\ (ml) \times$$
$$\frac{Molarity\ of\ Base}{Weight\ of\ the\ sample} \tag{3}$$

2.3.1.4 Iodine value (Wij's method)

The iodine of oil or fat is the weight of iodine absorbed by 100 parts by weight of the sample. 0.5 g of oil sample was weighed into a 250 ml conical flask and dissolved with 15 ml CCl_4 and 250 ml of Wij's reagent was added to the mixture. The flask was then stoppered and gently shaken and placed in the dark for 30 minutes. The excess iodine was determined by adding 20 ml of10% (w/v) KI solution and 150 ml water and titrating this with 0.1 M sodium thiosulphate using starch as an indicator. The titration was continued until blue color just disappeared after vigorous shaking. A blank determination was carried out. Iodine value was calculated using equation 4.

$$Iodine\ value = 12.69\ \times C\ \times (V_1 - V_2) \tag{4}$$

Where,

C = Concentration of sodium thiosulphate used,

V_1 = Volume of sodium thiosulphate used for blank,

V_2 = volume of sodium thiosulphate used for oil

2.3.1.5 Saponification value

About 1g of the oil was weighed into the flask and 12.5ml of alcoholic potassium hydroxide solution was pipetted into it. Reflux condenser was attached and heated in a boiling water bath for one hour with occasional shaking. The inside of the condenser was washed with distilled water to ensure that all the potassium hydroxide was in the flask. The entire content was titrated while still warm with 0.5M HCL using phenolphthalein as indicator. Procedural blank was prepared in similar way [15]. Saponification value was calculated using the equation 5.

$$Saponification\ value = (b - a) \times \frac{28.05}{Weight\ of\ sample} \tag{5}$$

Where,

b=Volume of HCL for blank,
a=Volume of HCL for sample,
28.05 value of 1/2 of molar mass of KOH (Since 0.5M KOH) was used

2.3.1.6 Unsaponifiable matter

After the determination of the saponification value, the unsaponifiable matter included the entire component after saponification. After the titration of the saponification value, neutralized liquid alkaline was prepared with 1ml of aqueous 3 M potassium hydroxide solution. This was transferred to a separator and wash in water (50ml less the volume of 0.5 M hydrochloric acid used). The solution was extracted with 50ml quantities of diethyl ether. The solution was then poured each into another separator containing 20 ml water. After the third extract was added, the combined ether extract was then shaken vigorously with 20 ml of the wash water. It was then calculated using the equation 6.

$$\frac{100\ (a-b)}{weight\ of\ sample} \tag{6}$$

Where;

a=mas in g of the residue from saponification value determination,
b= mass in g of

2.3.1.7 Trace metals analysis

Vegetable oil sample was weighed (0.5 g) into the conical flasks, 10 ml of concentrated HNO_3 (65%) and 5 ml of 70%, $HClO_3$was added and

the contents were heated at 70-80°C on a hot plate inside the fume cupboard for 30min after which 2 ml of H_2O_2 (30%) was added and boiled until white fumes disappeared, which signified that digestion was complete. The sample was allowed to cool and 20 ml of distilled water was added to bring the metals into solution. Sample was filtered using ashless Whatman filter into a 50 ml volumetric flask and made to mark with distilled water [16]. This solution was used to determine the trace metals concentration using atomic absorption spectrophotometer with acetylene-air flame atomization (FAAS, Buck Science 210 VGP). Calibration of the instrument was carried out using working standards prepared from commercially available metals standard solution (1000 Merck, Germany). The instrument parameters for the metals were selected based on the instrument guidelines. Procedural reagent blanks were prepared and analyzed and results of the metals concentrations were expressed as mean ± standard deviation from mean. Measurements were made within the linear range of the working standards used for calibrating the instrument.

2.3.1.8 Statistical analysis

Statistical analysis of data was carried out using SPSS 22.0 Statistical Package for Social Science. Descriptive statistics were employed in analyzing the levels of physicochemical properties and trace metals concentrations in the edible vegetable oils.

3. RESULTS AND DISCUSSION

3.1 Physicochemical Properties of Edible Vegetable Oils

The quality of edible vegetable oils was examined by determining the acid value, iodine value, saponification, specific gravity, refractive index and unsaponifiable matter. The results are presented in Figs. 1 to 6.

3.2 Acid Value (AV)

The levels of edibility of the oils were determined by the acid value method. This method indicated the extent of hydrolytic or enzymatic degradation in the oils. The results recorded in the oil samples ranged from 3.91 - 9.25 mg KOH/g in soy oil varieties and 3.69 - 9.65 mg KOH/g in palm olein varieties. The values obtained in this study across the oil varieties were higher than the FAO/WHO threshold limit (0.6 mg KOH/g) for

all edible oils [17,18]. The high AV obtained for the oil samples is indicative of high levels of free fatty acids, which may be associated with incomplete or inappropriate refining processes of edible oils; length and temperature of storage of raw materials and finished products [19,20]. The results obtained from this study were higher than 0.082 mg KOH/g and 0.091 mg KOH/g reported for Ethiopian vegetable oils of palm oil variety by [21]. The handling of the oils by retailers may also have affected their acid values, as most retailers display their wares in the open market directly under sunlight. According to the assertion of [22], edible oils quality and preservation are upheld when kept in air-tight amber-coloured glass ware rather than in polyvinylchloride containers in cool places.

3.3 Iodine Value (IV)

Iodine value (IV) evaluates the level of unsaturation in vegetable fat or oil and thus indicates the susceptibility of the oil to oxidation [23]. The IV in the edible oil samples ranged from 44.44 to 75.04 (g I_2/100g) in soy oil varieties and from 32.99 to 50.30 (g I_2/100g) in palm oil varieties. The soy oil varieties had higher IV when compared with those of palm olein varieties indicating more unsaturation in the soy oils than the palm olein varieties. The palm olein varieties would, therefore, be more stable to oxidation than the soy oils. The range of values obtained for the soy oil varieties agrees with the findings of [24[(61.91 and 89.86 g I_2/100g) for sesame oil varieties of Tanzania origin. The range of values obtained in this work agrees with the values (48.85 – 125.4 g I_2/100g) reported for the different brands of vegetable oils by [25].

3.4 Saponification Value (SV)

Saponification value of edible oil reveals the average molecular mass of fatty acids in the oil [26] and the deterioration of the oils. The SV obtained in this work ranged from 185.13 to 193.00 (mg KOH/g) in soy oil and 199.18-207.00 (mg KOH/g) in palm oil varieties respectively. The values were agreeable with the range of values (194.75 - 213.19 mg KOH/g) reported for some Ethiopian edible oils by [27] and the standard values (195-205 mg KOH/g) for palm oil in its refined and processed forms by SON, (2000). The results of this study were higher than the findings of [26] (125.6 and 153.8 mg KOH/g) who worked on the physicochemical properties of Pakistani mustard and corn oils respectively. The authors adduced their findings to a smaller
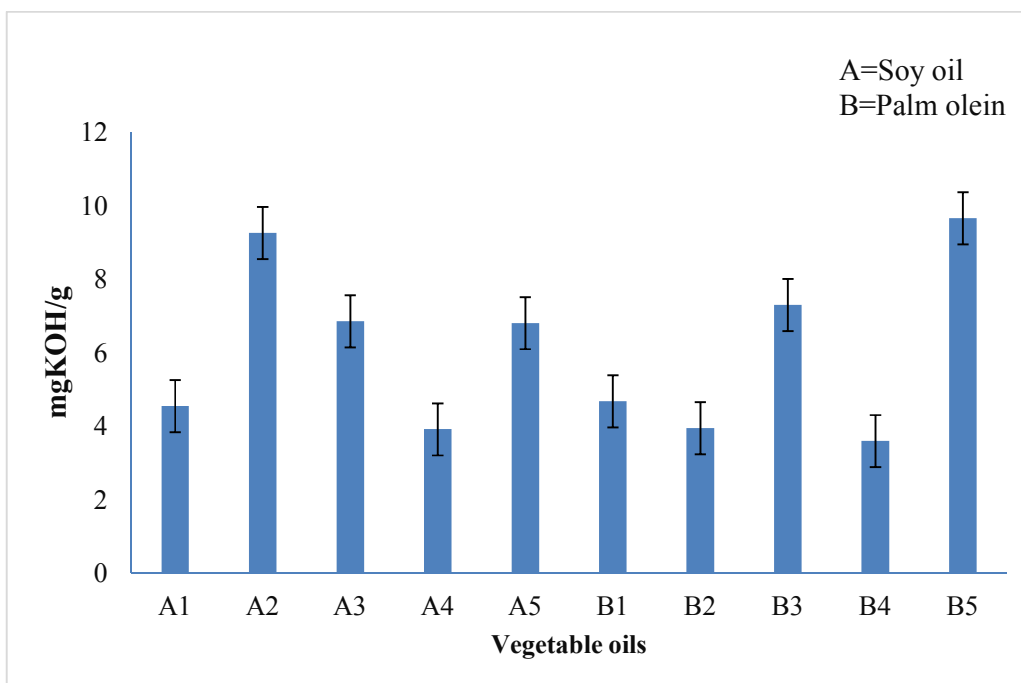
Fig. 1. Acid values of edible vegetable oils of soy and palm olein varieties
A1-A5 and B1-B5 represent five different brands of soybean oils and palm olein respectively
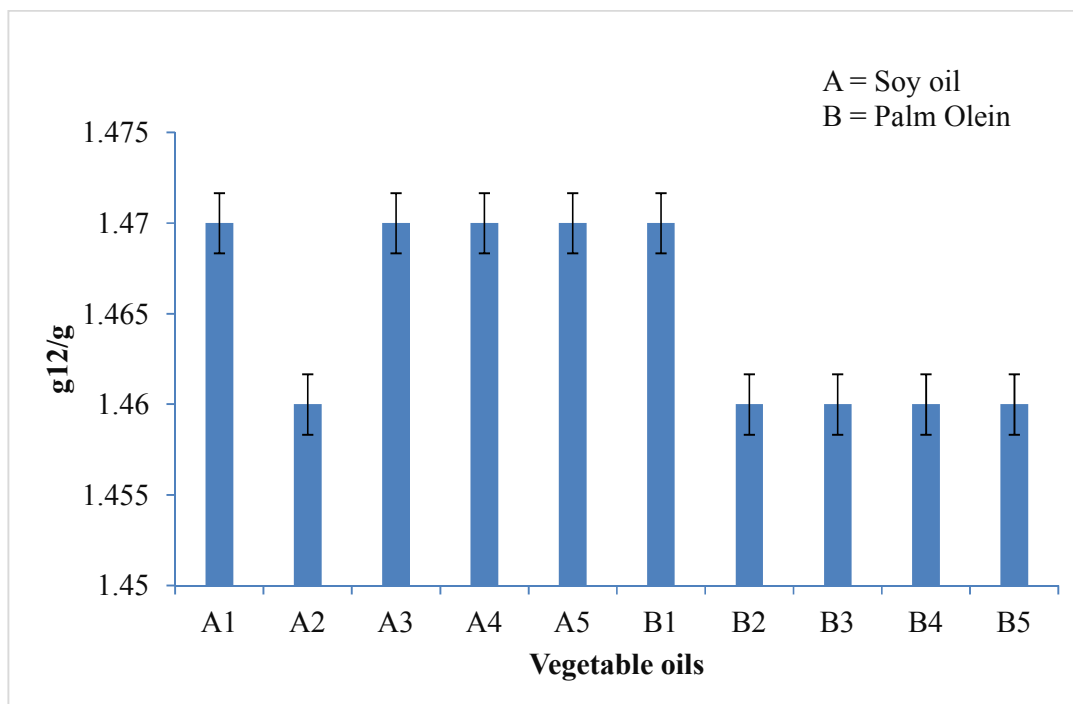*Error bars indicate the standard error of mean*

Fig. 2. Iodine values of edible vegetable oils of soy and palm olein varieties
A1-A5 and B1-B5 represent five different brands of soybean oils and palm olein respectively
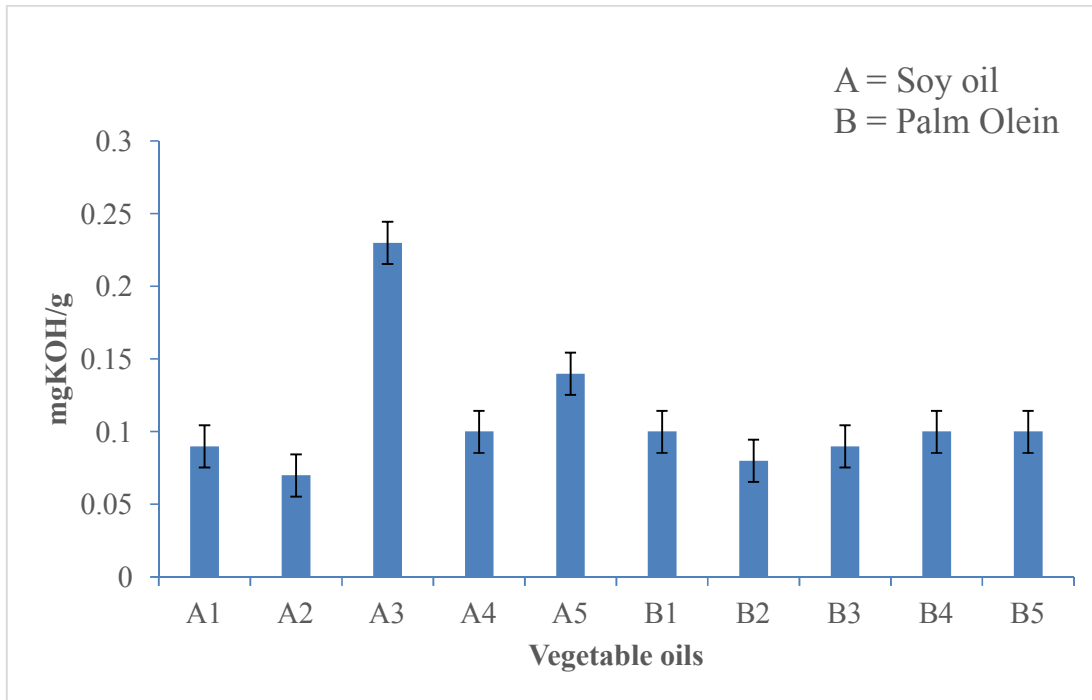*Error bars indicate the standard error of mean*

Fig. 3. Saponification values of edible vegetable oils of soy and palm olein varieties
A1-A5 and B1-B5 represent five different brands of soybean oils and palm olein respectively
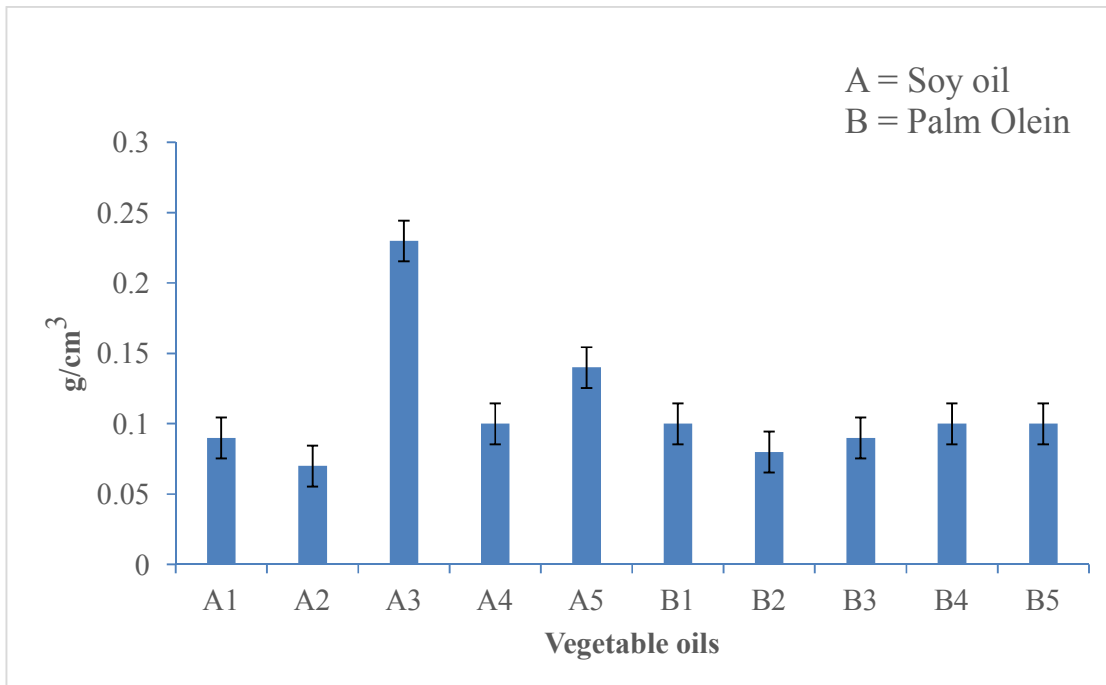*Error bars indicate the standard error of mean*

Fig. 4. Specific gravity of edible vegetable oils of soy and palm olein varieties
A1-A5 and B1-B5 represent five different brands of soybean oils and palm olein respectively
Error bars indicate the standard error of mean

number of ester bonds and low molecular weight of fatty acids in the edible oil. The fatty acids in the soy varieties were of lower molecular mass than those present in the palm olein varieties.

3.5 Specific Gravity (SG)

The specific gravity (SG) of oil depends on the temperature and different types of oil. The SG of the edible oil samples ranged within 0.910 and 0.920 (g/ml) at $30\,^0C$ for both brands and were within the threshold levels established by FAO/WHO (0.919 - 0.925 g/ml). This shows that the oil samples were pure and well refined. The values obtained in this work were similar to the results of [28,27] for edible oils. The higher SG value obtained in soy oils when compared to the palm oil varieties could be adduced to the presence of linoleic acid and other unsaturated fatty acids in the brand [20].

3.6 Refractive Index (RI)

The refractive indices(RI) of the edible vegetable oils of soy and palm oil varieties ranged within 1.46 and 1.47 at 20°C, and were within the recommended indices of refraction for edible vegetable oils (1.46 - 1.47) [29]. The values obtained in this work were also comparable with the values (1.466 – 1.475) reported by [27] for some Ethiopian edible vegetable oils.

3.7 Unsaponifiable Matter

The unsaponifiable matter is the non-glyceride components of vegetable oils which mostly form naturally or during oil processing or degradation. The unsaponifiable contents in the oil samples in this work ranged from 0.07 to 0.23 in soy oil and 0.08 to 0.10 in palm oil varieties respectively. The values were lower to the FAO/WHO standard (1.1 – 1.2) for unsaponifiable matter in edible oils.

3.8 Trace Metals Concentration in Edible Vegetable Oils

The concentration of trace metals in edible vegetable oils examined is presented in Table 1. The concentration of metals showed slight variations across the brands of edible vegetable oils.

The concentration of copper in the different samples of soy and palm oil varieties analyzed were within and some were above the [17] recommended limits. All the tested samples contained a significant amount of the trace metal.

In soy oil samples, the concentration of Cu occurred within 0.10 - 0.17 and 0.10 - 0.60 mg/kg in the palm olein samples. The highest concentration of Cu (0.60) was found in the palm olein varieties. Although copper is an essential element for human life, excessive intake can result in undesirable health conditions, such as accumulation in the liver, thus producing liver and kidney damage. Other effects include gastro-intestinal problems and anaemia (Manju, 2015). In addition, [28] opined that an increase in the concentration of copper in edible vegetable oils can act as catalyst, thus accelerating the oil rancidity. The finding of this study was similar to the results of [30] who worked on the Physico-chemical characteristics and levels of some selected metals in edible oils of Ethiopian origin and stated it to be acceptable to human consumption at nutritional levels.

As a basic element of varieties of enzymes, zinc plays numerous functions (regulatory, structural and catalytic) in the human body, helps in developing the brain, synthesizing DNA, the formation of bone, wound healing and body growth [31]. The concentration of zinc in the different samples of soy and palm olein were below the [17] recommended limits, except for sample B_1 which was extremely higher than the recommended limit. Zn concentration in the soy oil ranged from 0.06 - 0.30 and 0.11 - 2.46 mg/kg in palm olein. Deficiencies in the concentration of zinc is often associated with retarded growth and abnormalities in the immune system.

Iron has been reported to reduce the oxidative stability of edible oils, with its deficiency resulting in anaemia and impaired intellectual development [1]. Iron content ranged from 0.11 to 0.46 and 0.13 to 1.14 mg/kg in soy oil and palm oil respectively, with the highest concentration recorded in the palm olein oil varieties. Iron concentration in the different samples of soy and palm olein was below the FAO/WHO, (2011) recommended limits. The iron content recorded in this study was in agreement with the findings of Schumann et al. (2007).

Manganese contents in the different brands of edible vegetable oil range from 0.01±0.00 to 0.48±0.06 mg in soy oil and 0.14±0.04 to 1.01±0.20 mg in palm oil. The Mn level in the two samples was below the FAO/WHO, (2011) recommended limit except for the brand B_1 palm oil which was above the safety limit. Although the high concentration of Mn in the human diet has been attributed to severe effects on some
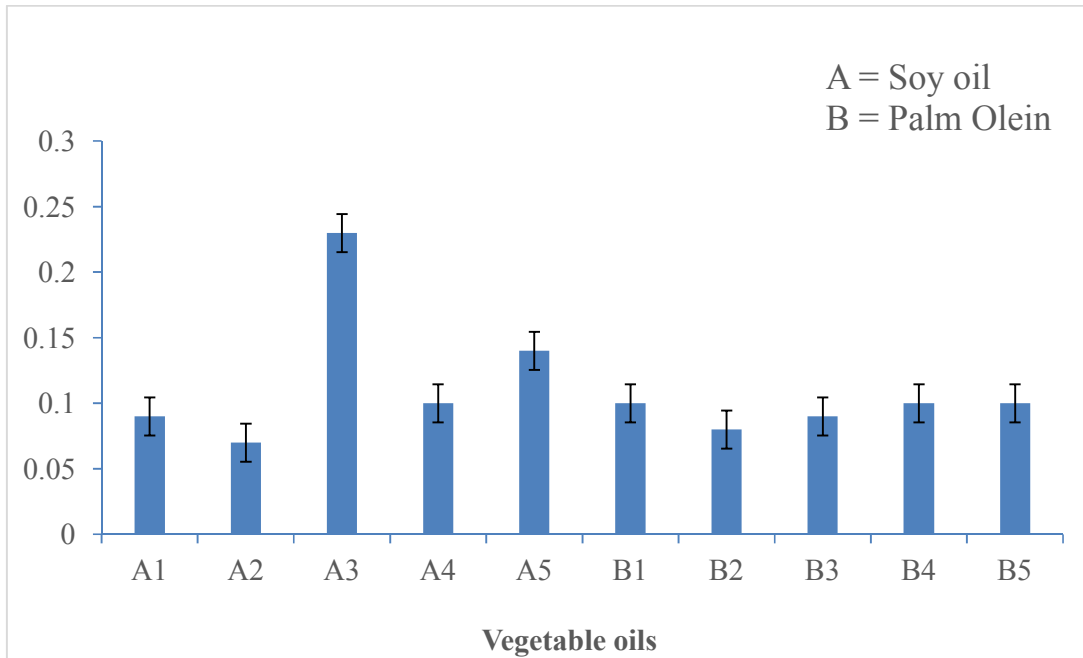
Fig. 5. Refractive index of edible vegetable oils of soy and palm olein varieties
A1-A5 and B1-B5 represent five different brands of soybean oils and palm olein respectively
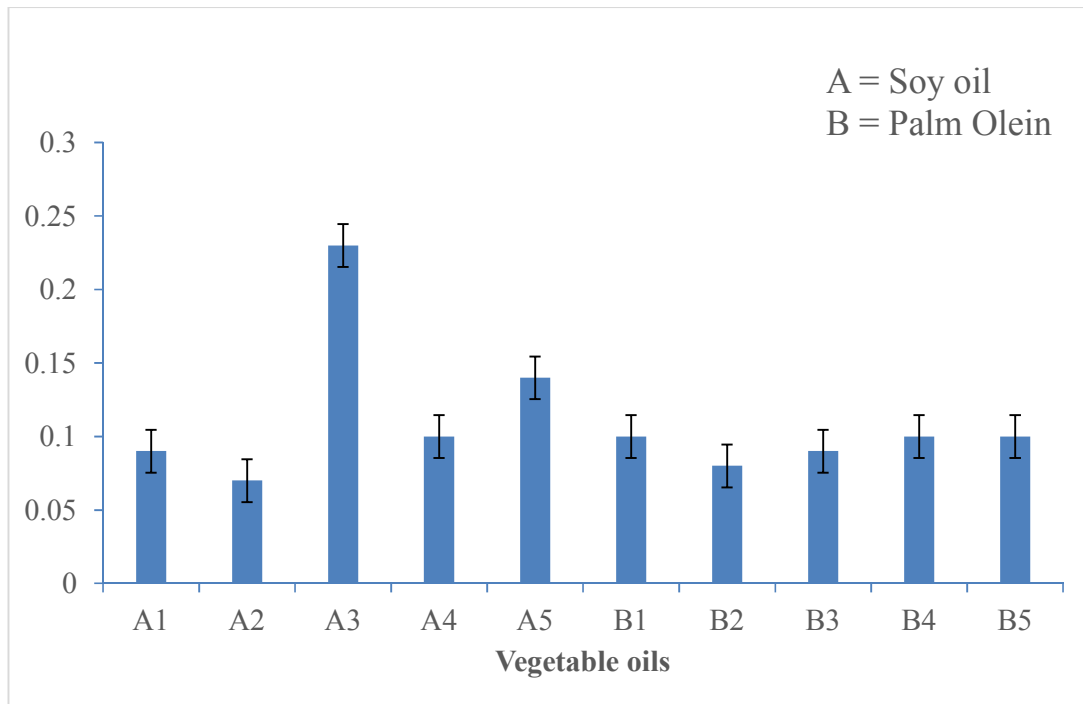*Error bars indicate the standard error of mean*

Fig. 6. Unsaponifiable matter of soy and palm olein varieties of edible vegetable oils
A1-A5 and B1-B5 represent five different brands of soybean oils and palm olein respectively
Error bars indicate the standard error of mean

Table 1. Heavy metal concentrations in soy and vegetable oils (mg/g)

Oil name	Sample	Cu	Zn	Fe	Mn	Cd	Ni	Pb	Cr
Soy oil	A_1	0.17±0.03	0.07±0.03	0.29±0.11	0.16±0.05	0.02±0.01	0.03±0.01	0.12 ±0.03	0.01±0.00
	A_2	0.12±0.03	0.30±0.07	0.20±0.11	0.22±0.19	0.04 ±0.01	0.06 ±0.04	0.10±0.04	0.03±0.01
	A_3	0.10±0.03	0.10±0.06	0.11 ±0.04	0.01±0.00	0.04±0.06	0.03±0.02	0.10±0.02	0.03±0.02
	A_4	0.12±0.01	0.25±0.14	0.14±0.04	0.48±0.06	0.01±0.01	0.08±0.02	0.15±0.02	0.05±0.01
	A_5	0.10±0.01	0.06±0.02	0.46±0.24	0.15 ±0.06	0.03±0.05	0.03±0.01	0.13±0.01	Bd
Palm olein	B_1	0.60±0.19	2.49±0.55	1.14±0.30	1.01±0.20	0.06±0.03	0.08±0.04	0.26±0.01	0.07±0.05
	B_2	0.11±0.03	0.12 ± 0.00	0.51±0.24	0.18±0.08	ND	0.07±0.03	0.09±0.01	0.01±0.00
	B_3	0.10±0.01	0.21±0.02	0.55±0.14	0.26±0.13	0.09±0.02	0.06±0.02	0.10±0.02	0.02±0.01
	B_4	0.10±0.01	0.11±0.01	0.44±0.04	0.21±0.02	0.02±0.01	0.04±0.01	0.22±0.01	0.07±0.01
	B_5	0.12±0.08	0.18±0.04	0.13±0.06	0.14±0.04	0.01±0.01	0.01±0.01	0.07±0.03	0.01±0.01
	FAO/WHO, (2011)	0.10	0.30	1.50	0.50	0.05	0.004	0.10	0.10

Mean± standard deviation
Bd indicate below detection limit
ND indicate not detected
A1-A5 and B1-B5 represent five different brands of soybean oils and palm olein respectively

organs, the lungs and on the brain, yet its deficiency can produce adverse and prolong reproductive and skeletal abnormalities.

The concentration of Cd recorded in this study ranges from 0.01±0.01 to 0.04±0.06mg in soy oil and 0.01±0.01 to 0.09±0.02mg in palm oil. When compared with the FAO/WHO recommended limit, most of the brands were below the safety limit for consumption except for brand B_1 and B_3 that was above the recommended limit. The highest concentration was also recorded in the palm oil sample. Cadmium concentrates largely in the lungs, kidney and blood-forming organs. However, its toxicity results in various human health disorders such as kidney dysfunction and damage, metabolic anomalies, damage to the skeletal system, reproductive deficiencies and problems and cancer [32].

It ranges from 0.03±0.01 to 0.06±0.04 mg in soy oil and 0.01±0.01 to 0.08±0.04 mg in palm oil. The highest concentration was recorded in the palm olein oil samples. Although, Ni at low concentration in the human system serves as enzymes activator, it becomes toxic to human health at high concentration. The Ni concentration in all the edible oil samples was above the FAO/WHO recommended limit. The high concentration could have an adverse effect on the human systems. Effects such as; organs dysfunction, metabolic abnormalities and immunological disorders.

The concentration of Pb observed in all the brands of edible soy and palm oil were above the FAO/WHO, (2011) recommended limit. It ranges from 0.10±0.02 to 0.15±0.02 mg in soy oil and 0.07±0.03 to 0.26±0.01 mg in palm oil. The highest concentration was recorded in the palm oil samples. Although lead has no beneficial role in the human system, toxic effects may arise from its presence and accumulation in the body. Acute and chronic symptoms of food poisoning occur from the consumption of both slight and a heavy dose of lead infiltrated foodstuffs. Lead also creates health disorders such as sleeplessness, tiredness, hear and weight loss. The lead levels were higher than those in previous reports [33,34, 35].

Chromium has been regarded as an essential mineral needed in the human diet for lipid metabolism and insulin functions [36]. Chromium concentration in all the brands was below the FAO/WHO recommended limit. It ranges from 0.01±0.00 to 0.05±0.01 mg in soy oil and

0.01±0.01 to 0.07±0.05 mg in palm oil, with the highest concentration recorded in the palm oil sample.

The comparison between the concentration of heavy metals in the edible soy and palm olein oils shows that the concentration of metals in the two oils was significantly higher ($P<0.05$) in the palm olein oils than in the soybean oils. This is in consonance with the report of [10] who stated that the principal source of oils contamination with heavy metals is their migration from arable soil into oil plants.

4. CONCLUSION

The present study examined the physicochemical properties and toxic potential of trace metals in commercially vegetable oils in Nigeria. The physicochemical analysis revealed that the parameters were all within the standard values for edible oils with the exception of the acid values. The high acid values are indicative of high levels of free fatty acids which may be associated with incomplete or inappropriate refining processes of edible oils; length and temperature of storage of raw materials and finished products. The handling of the oils by retailers may also have affected their acid values, as most retailers display their wares in the open market directly under sunlight. The trace metal concentration also revealed that the levels of Ni and Pb were above the threshold limit and may have a detrimental impact on the consumer's health. The metal contents were higher in the palm oil sample than the soy oil which may be attributable to agricultural or transportation factors. It is therefore recommended that the heavy metals in oils be monitored in order to prevent excessive build-up of the metals in the human food chain. Careful handling practice, processing and storage of raw materials and finished products are essential for quality maintenance.

COMPETING INTERESTS

Author has declared that no competing interests exist.

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