



Characterization of Raw, Semi-processed and Completely Processed Palm Oils

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Authors' contributions

This work was carried out in collaboration between both authors. Author EY designed the study, wrote the protocol and the first draft. Author AKI performed the statistical analysis, managed the analyses of the study and the literature searches. Both authors read and approved the final manuscript.

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ABSTRACT

Knowledge of the acid value (AV), peroxide value (PV), iodine value (IV) and iron concentration (quality-index variables) in oils is an indication of the edibility of the oil because these are parameters of oil-deterioration. The semi-processed oil is used to prepare a delicacy called "banga soup" in the Niger Delta region (Bayelsa State, Nigeria) in the ratio of 7:3 of the oil and other components. It is also a common practice to eat raw oil directly from the fruit. In this work, the edibility of raw, semi-processed and completely-processed oils were tested by measuring the quality-index variables. The raw oil (R-OIL) was obtained by scrapping the oil-bearing out part of the fruit with plastic knife. Semi-processed oil (SM-OIL) was the skim resulting from the mixture of milled fruit and hot water. The skim was heat-purified to give the completely processed oil (CP-OIL). 5g of oil was used for each analysis but the sample for FAAS was digested with aqua regia. Analyses were done by titrimetric methods for acid value, peroxide value, and iodine value and FAAS was used to determine the concentration of iron. In the raw oils from both samples (*E. guineensis* and *E. oleifera*), the acid values, peroxide values, iodine values and concentration of iron were above the recommended maximum Codex Standard for edible oils but values were within recommended range for the semi- and completely processed oils. The results indicate the non-edibility of raw oil.

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ACRONYMS

AV- Acid Value; B- Blank; CP-OIL- Completely Processed Oil; FAAS- Flame Atomic Absorption Spectroscopy; IV- Iodine Value; M- Molarity; PV-Peroxide Value; R-OIL-Raw Oil; S- Sample Titre; SM-OIL- Semi Processed Oil.

1. INTRODUCTION

Lipid oxidation negatively affects taste, aroma, nutrition, and causes health hazard such as biological damage to living tissues and increase in the risk of cardiovascular diseases [1]. Oxidized oil affects the taste of food in which it is added negatively and also causes some health problem such as diarrhea and poor growth rate [2]. Some products of decomposition in used oil have been identified to have adverse effects on human health [3-5]. Oils are rich in unsaturated fatty acids especially linoleic and linolenic [6]. Although the rate of oxidation is greatly accelerated at higher temperatures, oxidative reactions that occur at higher temperatures may not follow precisely the same routes and mechanisms as the reactions at room temperatures [7]. Cooking oil deterioration is accelerated by light, heat, water, acids, enzymes and micro-organisms. Its effects result in the formation of off-odors and flavors, reduction or destruction of essential fatty acids, generation of glycerol, free fatty acids and toxic products. It has been established that frying causes oils to undergo hydrolysis, oxidation and thermal reaction, and consequently numerous by-products such as free fatty acids, alcohols, cyclic compounds, dimmers and polymers can be produced [8]. Moisture in foods induces and accelerates oxidation with the hydrolytic compounds [9]. The content of total polar compounds and acid value are the most predominant indicators for oil quality and are widely used in many international regulations [10,11]. The aim of this work was to determine acid value, peroxide value, iodine value and iron content in raw, semi-processed and completely processed oils in order to assess their edibility; because there is an increasing interest of eating raw and semi-processed oils in Bayelsa State of Nigeria. The semi-processed oil is used to prepare a delicacy called "banga soup" in the Niger Delta region (Bayelsa State, Nigeria) in the ratio of 7:3 of the oil and other components. It is also a common practice to eat raw oil directly from the fruit.

2. EXPERIMENTALS

2.1 Sample Collection and Preparation

The oil was produced by small-scale rural oil-producing method. It involves threshing of bunches to free the palm fruit, mashing the fruit and pressing out the crude palm oil. The crude palm oil is further treated by heating to purify and dried for storage.

2.2 Preparation of Raw Oil

The fresh fruit bunch consists of fruit embedded in spikelets growing on a main stem. Manual threshing was achieved by cutting the fruit-laden spikelets from the bunch stem with a machete and then separating the fruit from the spikelets by hand. The separated fruits were washed with water and air-dried. The oil-bearing outer part was scrapped with plastic knife and the raw oil was squeezed out. 5 g of this oil (raw oil, R-OIL) was collected for analysis.

2.3 Preparation of Semi-processed Oil

The separated fruits were cooked in water and allowed to boil for 10 minutes in order to liquefy the oil in the fruit cells and facilitate its release during extraction. The soft fruits were milled by using mortar and pestle. The milled fruits were mixed with hot water and boiled to allow the oil to float and the oil was skimmed off. 5 g of the skimmed oil (semi-processed oil, SM-OIL) was collected for analysis.

2.4 Preparation of Completely Processed Oil

The skimmed oil was further purified by boiling for about 120 minutes followed by filtration to give the final product which was designated as completely processed oil (CP-OIL). 5 g of the heat-treated and filtered oil weighed for analysis.

2.5 Acid Value Determination

Solvent mixture of 1/1 (v/v) of 95 per cent (v/v) ethanol and diethyl ether was prepared. Potassium hydroxide (0.1 mol/L) solution was prepared in ethanol. Five grams of oil was dissolved in 50 ml of the solvent mixture of ethanol and diethyl ether. Phenolphthalein solution (0.2 ml) was added and titrated against potassium hydroxide in ethanol, to pink colour.

The acid value was calculated with Equation 1 below:

$$AV(\text{mgKOH/g}) = [\text{volume of KOH (ml)} \times \text{Molarity} \times 56.1\text{mg}]/[\text{Weight of sample}] \quad (1)$$

The titration was stopped when the colour persisted for 15 seconds. A blank test was also carried out.

2.6 Peroxide Value

30 ml of a mixture of chloroform and glacial acetic acid in a ratio of 2:3 was prepared. 5 g of oil (test sample) was mixed with this mixture in a 250 ml conical flask and agitated to effect dissolution of the sample. 0.5 ml of saturated potassium iodide was added, agitated and 30 ml of water was also added. This mixture was titrated against 0.01 M sodium thiosulphate until the yellow colour almost disappeared.

The peroxide value was calculated with Equation 2 below:

$$\text{Peroxide value (Meq Peroxide/kg)} = [(S - B) \times M \times 1000]/[\text{Sample weight}] \quad (2)$$

Where:

- S = Sample titre
- B = Blank
- M = Molarity of sodium thiosulphate

The titration was carried out slowly with vigorous agitation. A blank titration was also carried out.

2.7 Iodine Value

Two grams of oil sample was weighed into a dry glass stopper bottle of 250 ml capacity and 10 ml of carbon tetrachloride was added to the oil. About 20 ml of Wij's solution was then added and allowed to stand in the dark for 30 minutes. 15 ml of (10%) potassium iodide and 100 ml of water

was added and then titrated with 0.1 M sodium thiosulphate solution using starch as indicator just before the end point.

The iodine value was calculated with Equation 3 below:

$$\text{Iodine value} = [(B - S) \times N \times 12.69]/[\text{Sample weight (g)}] \quad (3)$$

Where: B = blank titre value; S = sample titre value; N = normality of $\text{Na}_2\text{S}_2\text{O}_3$; 12.69 is used to convert from meqthiosulphate to g iodine; molecular weight of iodine = 126.9.

A blank was also prepared alongside the oil samples.

2.8 Iron Concentration

Five grams of oil was digested with acid and the concentration of iron was determined with flame atomic absorption spectrophotometer as described by Farzin and Moassesi [12].

All laboratory ware including pipette tips were cleaned thoroughly with detergent and tap water, rinsed with distilled water, soaked in dilute nitric acid then rinsed thoroughly with deionized distilled water.

The calibration curve was based on five standards. The Fe standard solutions used for calibration were freshly prepared by diluting stock standard solutions (1000 mg/L) in a nitric acid solution (0.1% v/v) immediately before use. The calibration curve ranged from (2-10) mg/l of Fe. This was based on the expected concentrations of Fe.

5 g samples were digested with 10 ml of concentrated HNO_3 (65%) and 2 ml of concentrated H_2O_2 (30%) in microwave digestion system for 30 min and finally diluted to 10 ml with 2% HNO_3 . All sample solutions were clear. A blank digest was carried out in the same way. Digestion conditions for microwave system were applied as 5 min for 500 W, 5 min for 800 W, 10 min for 1000 W, 10 min for 1300 W, vent: 10 min, respectively.

Blank solutions were also prepared by using the same digestion procedure.

Flame atomic absorption spectrometry (FAAS) (Spectr AA 220, Varian, Australia), equipped with

deuterium background correction was used for the determination of Fe. The instrument parameters for the determination of Fe were: Wavelength: 248.3(nm); Slit width: 0.2(nm); Lamp current: 5.0(mA); Fuel gas: Acetylene; Oxidant gas: Air.

The analytical characteristics for the determination of Fe oil from the method validation were:

Recovery: 99%; Method detection limit: 0.04 mg/L; Method quantification limit: 0.13 mg/L; Minimal precision for 10 replicates: 2.2% R.S.D.; Intermediate precision with analyses carried out in 1-week periods for 5 weeks: 3.5% R.S.D.

3. RESULTS AND DISCUSSION

The means and standard deviations of triplicate determinations for the titrimetric and FAAS analyses of the raw oil (R-OIL), semi-processed oil (SM-OIL), and completely processed oil (CP-OIL) in comparison with the Codex standards are

as shown in Table 1. The acid values, peroxide values, iodine values, and iron concentrations for CP-OIL were lower than those of the SM-OIL and those of SM-OIL lower than those of R-OIL. This trend is seen in both samples (*Elaeis guineensis* and *Elaeis oleifera*). The decreasing trend could be due to the heating involved in processing the raw oil towards a completely processed level. Comparing the Codex Standards for these quality-index variables in edible oils with the test results showed that R-OIL values were above the recommended maximum values while SM-OIL and CP-OIL values are within acceptable ranges with respect to the standards. Fig. 1 is bar charts showing the variation of acid value, peroxide value, iodine value and iron concentration in raw oil, semi-processed oil and completely processed oil for *Elaeis guineensis* and *Elaeis oleifera*. It is clear from the chart that all test variables in semi- and completely-processed oils were within WHO-acceptable limits except the raw oil data. R-OIL should therefore not be taken directly as food; however, its usefulness still remains in industrial processes, like in the production of soap.

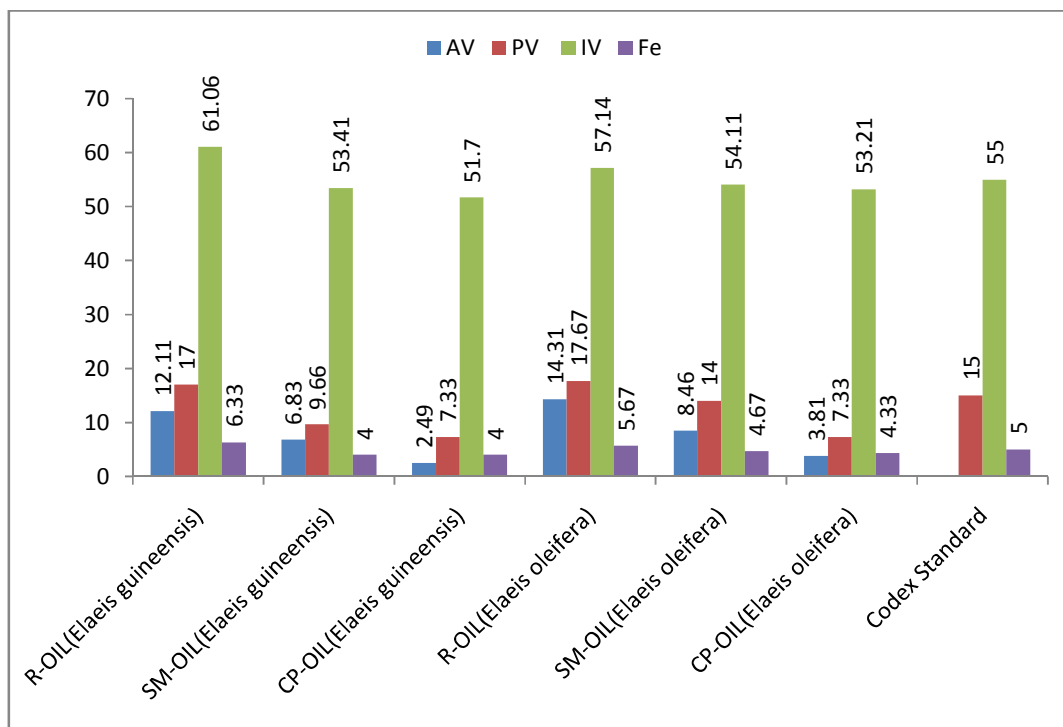


Fig. 1. Comparison of AV, PV, IV and Fe in *E. guineensis* and *E. oleifera* with the recommended codex standard for edible oil

AV = acid value (mg KOH/g oil), PV = peroxide value (mEq of active oxygen/kg oil), IV = iodine value, Fe = iron concentration (mg/kg)

Comparing the values for AV, PV and IV gotten from this study with values gotten from previous studies (Table 2) shows that the AV for R-OIL in this study, (12.11±0.14) mgKOH/g for *E. guineensis* and (14.31±0.32) mgKOH/g for *E. oleifera* were much higher than studies conducted in Kogi State [13], they recorded a range of (1.98-3.63) mgKOH/g; Plateau State [14], they recorded (2.67-4.20) mgKOH/g and Osun State [15], they recorded (0.77-1.15) mgKOH/g for crude palm produced by semi-mechanized mill. However, the results obtained in this study, though high, were close to results obtained from other studies conducted in Bayelsa State [16], they recorded (8.43±0.22) mgKOH/g; Delta State [17], they recorded a range of (7.04-12.24) mgKOH/g and Rivers State[18], they recorded (8.44-10.30) mgKOH/g. According to Tagoe et al. [19], the high AV recorded in this study could have varied from other studies because of the duration of storage before analysis, since the AV of processed oil increases with duration of storage. In essence, the palm oil in this study must have been kept longer before analysis than for the other studies.

The PV values obtained in this study (17.00±1.00 and 17.67±0.58) mEq O₂/kg for *E. guineensis* and *E. oleifera* respectively, were higher than studies conducted in Kogi State [13], they recorded the recorded (7.1-8.8) mEq O₂/kg; Delta State [17], they recorded (2.7-4.7) mEq O₂/kg; Osun State [15], they recorded (4.94-6.00) mEq O₂/kg and Rivers state [18], they recorded (1.20- 1.93) mEq O₂/kg. Though, lower when compared with findings from a similar study conducted in Bayelsa state by Ohimain et al. [16]. They recorded (43.90±0.06) mEq O₂/kg.

Peroxide value is a measure of oxidation during storage and the freshness of the lipid matrix [16]. The active oxygen combines with double bonds of the fatty acids in the triglycerides, which are useful indicators of the early stages of rancidity [20]. Oils with high PV could be harmful to human health due to the free radicals that can be generated in the oxidative process [19]. It is therefore a necessity for raw oil to be processed before consumption.

Iodine value is the amount of iodine required to saturate the unsaturated multiple bonds in one gram of oil. The iodine value determines the stability and shelf life of oil. The higher the iodine value, the shorter the shelf life.

The IV for this study (61.04±0.42) and (57.14±0.77) for *E. guineensis* and *E. oleifera* respectively, was higher than the values recorded in other studies (Table 2), though very close to a similar study conducted in Bayelsa State [16]. They recorded 51.17±1.78. Also, a study in Plateau State recorded a range of 46.74-63.59 [14]. Processing of raw oil is essential to bring the iodine value within regulatory limits as to increase the shelf life of the oil.

Comparing the AV, PV, IV and Fe values of R-OIL for *E. guineensis* and *E. oleifera* showed that *E. guineensis*<*E. oleifera*; *E. guineensis*<*E. oleifera*; *E. guineensis*>*E. oleifera* and *E. guineensis*>*E. oleifera* for AV, PV, IV and Fe, respectively. However, applying t-test at 95% confidence limit showed that there was no significant difference for PV and Fe, while there was a significant difference for AV and IV between *E. guineensis* and *E. oleifera*.

$$t = \frac{(\bar{x}_1 - \bar{x}_2)}{s \sqrt{\frac{1}{n_1} + \frac{1}{n_2}}} \tag{4}$$

$$s^2 = \frac{(n_1 - 1)s_1^2 + (n_2 - 1)s_2^2}{n_1 + n_2 - 2} \tag{5}$$

Equation 4 enables the calculation of “t” while Equation 5 is used to calculate the pooled standard deviation, since the same variation is expected from both *E. guineensis* and *E. oleifera*. The comparisons of the means of the parameters studied are as stated in Table 3.

The statistical evaluation of the mean differences indicates that *E. guineensis* should be more unsaturated than *E. oleifera*, since it has significantly higher IV and therefore its shelf life is likely to be shorter.

Table 1. Values of AV, PV, IV and Fe in raw, semi-processed and completely processed oils

Parameter	<i>Elaeis guineensis</i>			<i>Elaeis oleifera</i>			Codex standard
	R-oil	SM-oil	CP-oil	R-oil	SM-oil	CP-oil	
AV(mgKOH/g)	12.11±0.14	6.83±0.20	2.49±0.08	14.31±0.32	8.46±0.46	3.81±0.10	NA
PV (mEq O ₂ /kg)	17.00±1.00	9.66±0.58	7.33±0.58	17.67±0.58	14.00±1.00	7.33±0.58	15
IV	61.06±0.42	53.41±0.72	51.70±0.98	57.14±0.77	54.11±0.49	53.21±0.33	50.0-55.0
Fe (mg/kg)	6.33±0.58	4±0.00	4±0.00	5.67±0.58	4.67±0.58	4.33±0.58	5

R = raw oil; SM = semi-processed oil; CP = completely processed; AV = acid value; PV = peroxide value; IV = iodine value; Fe = iron concentration; NA= not available

Table 2. Comparison of AV, PV and IV obtained from this study with values obtained from other studies

Oil quality parameter	This study (mean±s.d.)		Ohimain et al.	Akubor and Ogu,	Aleto et al.	Okechalu et al.	Akinola et al.	Ohimain et al.	Codex value
	<i>E. guineensis</i>	<i>E. oleifera</i>	2013 [16]	2012 [13]	1990 [17]	2011 [14]	2010 [15]	2012 [18]	
	Bayelsa	Bayelsa	Bayelsa	Kogi	Delta	Plateau	Osun	Rivers	
AV	12.11±0.14	14.31±0.32	8.43±0.22	1.98-3.63	7.04-12.24	2.67-4.20	0.77-1.15	8.44-10.30	-
PV	17.00±1.00	17.67±0.58	43.90±0.06	7.1-8.8	2.70-7.40	23.2-35.5	4.94-6.00	1.20-1.93	15
IV	61.06±0.42	57.14±0.77	51.17±1.78	30.4-42.8	-	46.74-63.59	47.42-48.44	-	50-55

AV = acid value(mgKOH/g); PV = peroxide value(mEq O₂/kg); IV = iodine value; - = Not Available

Table 3. Comparison of the means of AV, PV, IV and Fe between *E. guineensis* and *E. oleifera*

	Means and standard deviations		Calculated "t"	Critical "t" at 95%, dF=4 (2-tailed)	Remark
AV	$\bar{X}_1=12.11$	S1=0.14	5.3627	2.776	Significantly different
	$\bar{X}_2=14.31$	S2=0.32			
PV	$\bar{X}_1=17.00$	S1=1.00	0.5357	2.776	Not significantly different
	$\bar{X}_2=17.6$	S2=0.58			
IV	$\bar{X}_1=61.06$	S1=0.42	3.8783	2.776	Significantly different
	$\bar{X}_2=57.14$	S2=0.77			
Fe	$\bar{X}_1=6.33$	S1=0.58	0.7586	2.776	Not significantly different
	$\bar{X}_2=5.67$	S2=0.58			

X1= *E. guineensis*; X2= *E. oleifera*; dF= degree of freedom

4. CONCLUSION

Following the rate of consumption of raw and semi processed palm oil in Bayelsa State and hence the need to ascertain the fitness of these oils for consumption as food, this study was conducted by determining the acid value, peroxide values, iodine values and concentration of iron in the oils.

The acid values, peroxide values, iodine values, and concentration of iron of raw oil (R-OIL) were above recommended maximum values by Codex Standard for edible oils; therefore raw oil is not suitable for food, however, it can be used for other industrial processes, like in the production of soaps and cosmetics. This information if adequately cascaded will dissuade locals within Bayelsa State to stop the habit of eating raw palm oil. The acid values, peroxide values, iodine values, and concentration of iron for Semi-processed oil (SM-OIL), and completely processed oil (CP-OIL) were within acceptable ranges, by the same Codex Standard and therefore fit to be taken as food.

The significantly higher iodine value of *E. guineensis* when compared with *E. oleifera* indicates that it will be more unsaturated and would have a shorter shelf life.

COMPETING INTERESTS

Authors have declared that no competing interests exist.

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