



ORIGINAL ARTICLE

Effect of heating time on the physicochemical properties of selected vegetable oils



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Abstract The determination of oil physicochemical properties is a useful strategy to monitor oil quality to avoid the use of potentially toxic oil. It is relevant in determining the physical and chemical changes that oil undergo during heating. In this work, the effect of prolong heating time from 1–7 h on the physicochemical properties of soybean oil, palmolein oil, groundnut oil and palm kernel oil was examined. Properties such as specific gravity, viscosity, saponification value (SV), iodine value (IV), free fatty acid and peroxide value (PV) were studied to evaluate the compositional quality of oils. Results revealed that heating at the different times did not have any significant effect on the physicochemical properties of the oils. There was however, significant differences of the physicochemical properties on the oil types. Results further reveal that based on the peroxide value, soybean oil was the most stable oil out of the four oils studied while palm kernel oil was found to be the least stable oil; as the stability of palm olein oil was found greater than that of groundnut oil.

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1. Introduction

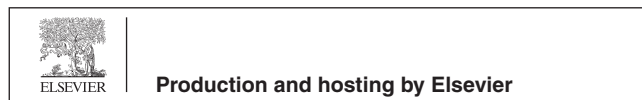
Vegetable oil is useful for the vitamins, antioxidants and energy it gives. It also functions as a transporter of several vitamins to the body cell (Kritchevsky, 2008). The use of veg-

etable oil is very common in food preparation. Deep frying is a common process in the preparation of food whereby the food is immersed in hot oil in the temperature range of 140–200 °C. During deep frying, simultaneous heat and mass transfer enable the desirable quality of fried food to be obtained. The compositional quality of oil is responsible for its physicochemical properties. Deep frying causes changes to occur in the physicochemical, nutritional and sensory properties of the oil (Che Man, 2000). During frying, myriads of complex reactions such as hydrolysis, isomerization, cyclization, oxidation and polymerizations occur which changes the flavour and decompose the compounds in the oil (Gómez-Alonso et al., 2003), thereby affecting the quality of oil and fried food. The oxidized

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products of fatty acids give off-flavours and odours. According to [Vieira et al. \(2015\)](#), high levels of free fatty acids especially linoleic acid are undesirable in finished oils because they can cause off-flavours and shorten the shelf-life of oils. The deteriorated frying oil can lead to adverse health effect on the human system due to accumulated toxic substances in the oil and fried product ([Tena et al., 2009](#)). Upon repeated frying, the oil can reach a stage where it is no longer suitable for use and required it to be discarded ([Weisshaar, 2014](#)).

Depending on the level of saturated or unsaturated fatty acid ratio, oil may be classified into hard or soft oil ([Ericson, 1996](#)). The implication is that soft oils have higher proportions of unsaturated fatty acids than saturated fatty acids, unlike hard oils which contain higher proportions of saturated fatty acids than unsaturated fatty acids. While soft oils are liquid at any temperature above 0°C, hard oils are solid or semi-solid at ambient temperature ([Naghshineh et al., 2009](#)). Therefore, in principle, soft oils are more easily oxidized and less stable than hard oils.

Recently, the effect of continuous deep-fat frying on the physicochemical properties of 10 brands of edible cooking oil was investigated by [Omara et al. \(2019\)](#). Their results showed that the reuse of oil for continuous frying of potatoe can be done only up to 7 times on average for hard oil and 6 times for soft oil carried out at frying time between 10–20 mins and a temperature of 140 °C.

In another study, [Zahir et al. \(2014\)](#) examined the physicochemical properties of corn and mustard oil after they have been used to fry potatoes three times. Two temperatures – 140 °C and 170 °C – were used for corn and mustard oil respectively. Their result suggested that repeated heating gradually diminishes the quality of the oil. They found secondary oxidized compounds with mustard oil.

Frying performance of palm olein oil and its blend with soybean oil, sunflower oil and canola oil was investigated ([Siddique et al., 2013](#)). Using fish frying, the process was carried out for 60 min in three stages, 20 min for each frying time at a temperature of 200 °C. Palm olein oil was found to be better against oxidation among other frying oils used in this study. Viscosity of pure oils and their blends decreased with increase in temperature. Since they also investigated rheological properties, they concluded that viscous property was higher than the elastic property of oil and blend. However, the critical stress for oil blends was found higher than that of pure oils.

In this study, the aim is to investigate the effect of deep frying on the physicochemical properties of four selected edible oils- palm olein oil, soybean oil, groundnut oil and palm kernel oil- which has not been compared before. Unlike the approach of previous studies, the oils were first heated up for the time periods ranging 1–7 h before they were used for frying.

2. Materials and methods

2.1. Oil samples and reagents

Hydrochloric acid (HCl), sodium hydroxide (NaOH), potassium hydroxide (KOH), ethanol (C₂H₅OH), chloroform (CHCl₃), iodine, sodium thiosulphate (Na₂S₂O₃·5H₂O), starch indicator were of analytical grade and purchased from Merck Darmstadt, Germany.

Vegetable oil (palm olein oil, soybean oil, groundnut oil and palm kernel oil) were purchased from a local supermarket in Benin City, Nigeria.

2.2. Frying process

Oil sample was first heated up to 185 ± 5 °C for different duration of 1 h, 3 h, 5 h, and 7 h, to give a total of four (4) samples for each oil and twenty samples altogether. Immediately after heating, the oil samples were used to fry akara (bean cake) as it is popularly referred to in Nigeria. Frying duration was for five (5) minutes.

2.3. Specific gravity measurement

The densities of oil samples before and after frying were measured using a pycnometer with a 50 mL capacity. The empty bottle was weighed (W₀) and filled with oil sample. Thereafter, the content was emptied and filled with water and reweighed as W₂. The specific gravity was determined as follows using Eq. (1)

$$s.g = \frac{w_1 - w_0}{w_2 - w_0} \quad (1)$$

2.4. Viscosity measurement

The viscosity of oil samples before and after frying was measured by an Ostwald Viscometer techniconomial constant 0.05Cs/c, ASTMAD 445 England. The flow time (in seconds) of oil samples was recorded with a stop watch. Viscosity was calculated using Eq. (2)

$$viscosity = \frac{4.39 \times t}{8} \quad (2)$$

2.5. Saponification value measurement

Saponification value was determined with slight modification according to [ASTMD464](#). Typically, 2.0 g of oil sample was weighed into a conical flask, 25 mL of 0.5 N ethanolic KOH was added into the flask containing the oil and mixed thoroughly. A condenser was fitted to the conical flask and the mixture heated between 60 and 70 °C for an hour. The hot mixture was titrated with 0.5 N HCl using 2 drops of 1% phenolphthalein indicator until a colourless solution was obtained. A blank was determined in the same manner. The SV was obtained with Eq. (3)

$$S.V = \frac{A - B \times N \times 56.1}{W} \quad (3)$$

where A = HCl(mL) for blank, B = HCl (mL) for sample, W = weight of sample (g), N = normality of HCl

2.6. Peroxide value measurement

Peroxide value was determined according to the method described by [Cox and Pearson \(1962\)](#). 1 g of oil sample was placed in tube, followed by 1 g of powdered KI and 20 mL of solvent mixture (2:1 of glacia acetic acid and chloroform). Then the tube was transferred to boiling water and allowed

to boil for 30 s. Thereafter, the content of the tube was transferred to a conical flask containing 20 mL of 5% KI solution. 35 mL of distilled water was added to the mixture in the conical flask. The mixture was titrated with 0.002N $\text{Na}_2\text{S}_2\text{O}_3$ until a pale yellow colour was obtained. 0.5 mL starch was added to the pale mixture and titrated till a colourless mixture was obtained. A blank was determined in the same manner. The peroxide value was calculated using Eq. (4).

$$P.V = \frac{(S - B) \times 1000}{W} \quad (4)$$

where S = $\text{Na}_2\text{S}_2\text{O}_3$ (mL) for sample, B = $\text{Na}_2\text{S}_2\text{O}_3$ (mL) for blank, W = weight of oil (g)

2.7. Acid value measurement

The acid value was determined using the ISO 660:1996 method. 2 g of oil was mixed with 10 mL ethanol, followed by two (2) drops of phenolphthalein. The mixture was boiled for about five minutes and then titrated with 0.1N KOH until a pale pink colour appeared. The acid value was calculated using Eq. (5) and FFA was calculated as follows in Eq. (6)

$$\text{acid value} = \frac{T \times N \times 56.1}{W} \quad (5)$$

$$\text{FFA} = \frac{\text{acid value}}{2} \quad (6)$$

where T = volume of titrant (mL), N = normality of KOH, W = weight of oil sample (g)

2.8. Iodine value measurement

The iodine value (IV) was determined using the Hubl method. Typically, 0.5 mL of oil was mixed with 5 mL of chloroform and dissolved by gentle swirling. Hubl's Iodine was added slowly from the burette until the colour of iodine matched with the control. Then the burette reading was noted, and the experiment was repeated. Iodine value was calculated using Eq. (7).

$$IV = \frac{260x}{45.150} \quad (7)$$

where x is the volume of Hubl's iodine in mL

2.9. Statistical analysis

The obtained data were subjected to one way ANOVA using Microsoft Excel 2013. The effect of time, temperature on the physicochemical properties of oil was investigated including analysing the effect of variation of physicochemical properties among the oil samples defined at $p < 0.05$.

3. Results and discussion

The quality of oil samples before heating (that is at room temperature) was analyzed by evaluating physicochemical properties such viscosity, specific gravity, peroxide value, iodine value, free fatty acid and saponification value. Results are presented in Table 1. The effect of heating carried out at 185 °C under varying times was also investigated and the results are presented in Table 2.

3.1. Specific gravity and viscosity

The specific gravity is an important property affecting the heat transfer by natural convection. The specific gravity of the oils differ due to their different structural composition. Although there were changes on heating, heating at 185 °C at different times (1–7 h) did not have any significant effect on the specific gravity of the oils as can be found in Table 3. In Table 2, the highest values of specific gravity is attributed to soy bean oil, ranging in values from 0.919–0.922 followed by palm kernel oil with value of 0.918–0.921, then palm olein oil with value of 0.910–0.912 while groundnut oil had the least specific gravity range of 0.910–0.911. The values obtained were closely related to the standard range of 0.898–0.907 approved by Standard Organization of Nigeria (SON, 2000). During frying, due to hydrolysis, oxidation and polymerization processes, the composition of the oil degrades, which affects the stability of its compounds (Gloria and Aguilera, 1998). In general, there was reduction of specific gravity as the number of frying times increased except for the specific gravity of palm kernel oil which increased as the frying time increased. The reduction in specific gravity might be due to breakdown of the oil's structure following heating at high temperature and consequently reducing the density of the oil as frying time progresses. The higher specific gravity observed for soybean oil can be attributed to the higher content of linoleic acid it contains.

Viscosity is another parameter used to measure physical changes. It depends on the specific gravity, molecular weight, temperature and degree of unsaturation. In general, the viscosity of the oils both increased and decreased without a trend when heated at increasing frying times as shown in Table 2. However, changes in viscosity at room temperature (27 °C) follows the trend: palm olein oil > groundnut oil > palm kernel oil > soybean oil as presented in Table 1.

The viscosity trend obtained before heating (seen in Table 1) was the same after heating (seen in Table 2). That is, palm olein oil showed the highest viscosity in the range of 810.635–875.412 while soybean oil showed the least viscosity reported in the range of 668.580–748.430. There was no definite trend on viscosity as heating time increased. In addition, there was no significant effect on heating time and temperature on the viscosity of oils as presented in Table 3. The increase in viscosity is likely due to saturation of the triglyceride chain by the process of hydrogenation while the decrease may be due to unsaturation (Santos et al., 2014). Increase in the viscosity of oil may also be due to polymerization of the oil and formation of dimer (Choe and Min 2007). A decrease on the other hand, as a result of increasing heating time can be attributed to increase in the kinetic energy which enhances the movement of molecules and enhances intermolecular forces.

3.2. Peroxide value

Peroxide is a useful indicator on the extent of oxidation the oil has undergone. In Table 1 and Table 2, the range of peroxide values of palm olein and soybean oil indicate that they are below 10 meq/kg oil specified by NIS (1992) and SON (2000). Perhaps, the reason for the wide variation in peroxide values (of palm olein and soybean oil) in comparison with the standards can be attributed to their dissimilar production processes. On account of their very low peroxide values, the two

Table 1 Physicochemical properties of oil before frying.

Properties	Palm olein oil	Soybean oil	Groundnut oil	Palm kernel oil
Viscosity (millipoise)	865.065	640.395	863.576	747.491
Specific gravity (g/ml)	0.911	0.918	0.911	0.916
Peroxide value (meq/kg)	0.014	0.005	18.000	29.000
Saponification value (mg)	61.897	61.897	65.917	67.600
Free fatty acid	5.772	7.517	11.226	5.610
Iodine value (g)	1.670	0.921	76.307	70.302

Table 2 Physicochemical properties of oil after frying at different time.

Trials	Viscosity (milipoise)	Specific gravity (g/mL)	Peroxide value (meq/kg)	Saponification value (mg)	Free fatty acid	Iodine value (g)
<i>Palm olein oil</i>						
1 h	840.415	0.910	0.018	61.336	5.610	1.728
3 h	810.635	0.910	0.020	63.019	6.956	1.785
5 h	896.895	0.909	0.021	60.214	3.142	1.612
7 h	875.412	0.912	0.023	59.653	2.805	1.382
<i>Soybean oil</i>						
1 h	691.485	0.919	0.016	60.962	3.029	0.691
3 h	668.580	0.921	0.008	61.897	5.386	1.209
5 h	678.870	0.921	0.011	62.645	8.303	0.864
7 h	748.430	0.922	0.017	61.523	6.900	1.611
<i>Groundnut oil</i>						
1 h	875.657	0.910	16.000	64.515	10.084	74.424
3 h	831.708	0.909	14.000	70.125	8.062	73.855
5 h	827.389	0.901	10.000	70.276	10.261	73.094
7 h	862.300	0.911	8.000	67.320	7.854	69.541
<i>Palmkernel oil</i>						
1 h	839.933	0.918	20.000	69.003	4.448	73.855
3 h	829.578	0.920	22.000	69.206	2.224	76.901
5 h	812.057	0.920	14.000	71.808	3.366	79.947
7 h	767.459	0.921	10.000	67.039	1.122	81.977

Table 3 ANOVA.

Independent variables	Dependent variables					
	Viscosity	Specific gravity	Peroxide value	Saponification value	Free fatty acid	Iodine value
Heating time	0.9547	0.8904	0.9044	0.7984	0.9044	0.9999
Heating temperature	0.7210	0.9227	0.8809	0.7920	0.6185	0.9857
Oil types	0.0001*	0.0000*	0.0000*	0.0001*	0.0024*	0.0000*

* = Significant.

oils will display the tendency to be slow to oxidation or polymerization unlike groundnut oil and palm kernel oil as evident from their peroxide values after heating as presented in Table 2. According to Decker et al. (2010), the addition of antioxidants to oils can lead to low peroxide values. Antioxidants are added so as to prevent rancidity and increase shelf-life. Aside the antioxidants, the low peroxide values also suggest that the oils (palm olein oil and soybean oil) contain low molecular weight fatty acids of low unsaturation as a result of hydrolytic rancidity. Of all the oils, only palm olein oil showed an increase in the peroxide value with increasing heating time while groundnut

oil showed a decrease in peroxide value with increasing heating time. Soybean oil and palm kernel oil showed no regular pattern in the peroxide values obtained. For soybean oil, a decrease is observed from 1- 3h and an increase from 5-7 h. As for palm kernel oil, there is an increase from 1 - 3h and a decrease from 5 - 7h. The fluctuation in the peroxide values of soybean oil and palm kernel oil can be attributed to decomposition of the peroxides that are formed during primary oxidation to secondary oxidation (Guillén and Cabo, 2002). A decrease in peroxide value with increasing heating time has been observed by several researchers (White, 1991; Vieira

and Regitano-D'arce, 1999; Zhang et al., 2007). The decrease can be attributed to the fact that the peroxides formed on heating are unstable compounds towards high temperatures and frying time, transforming them to carbonyl and aldehyde compounds (Serjouie et al., 2010). The results further reveal that groundnut oil and palm kernel oil have gone rancid due to their high peroxide values and there is need for further refining in order to meet specific purpose. The trend in peroxide value before heating and after heating can be reported as palm kernel oil > groundnut oil > palm olein oil > soybean oil.

3.3. Saponification value

Saponification value (S.V) is an index of the molecular weights of triglycerides in the oil. It is inversely proportional to the average molecular weight or chain length of the fatty acids (Muhammad et al., 2011). Therefore, the shorter the chain length, the higher the S.V. The S.V obtained for the oil samples in Table 2 showed 59.69–61.34 mg/KOH/g for palm olein oil, 60.96–62.65 mg/KOH/g for soybean oil, 64.52–70.28 mg/KOH/g for groundnut oil, and 67.04–71.81 mg/KOH/g for palm kernel oil. Thus, S.V obtained for palm kernel oil and groundnut oil were found higher than S.V obtained for palm olein oil and soybean oil. All measured saponification values were below the expected range of 195–205 mg/KOH/g for edible oil as specified by SON (2000) and NIS (1992). The low value of S.V. suggest that the mean molecular weight of triglycerides is low or that the number of ester bonds are low. Therefore, these oils need refining in order to meet the required standard and would be better fitted for industrial purpose. On subjecting to statistical analysis, there was no significant effect of temperature and heating time on the saponification values of the oils as can be found in Table 3. However, there was significant effect of the physicochemical properties on the oil types, indicating large variations in their production.

3.4. Iodine value

Iodine value is a measure of the amount of unsaturation in fatty acids. The iodine values obtained for the oil samples are shown in Table 2. For palm olein oil, the iodine value (1.382–1.785), increased from 0 to 3 h and then decreased steadily up to 7 h, due to breakage and saturation of bonds. In soybean oil, the trend is less defined with iodine value of 0.691–1.611. However, in groundnut oil, the iodine value (69.541–76.307) decreased steadily. This may be due to decrease in unsaturation. In contrast, the iodine value (70.302–81.977) of palm kernel oil, increased steadily. This is likely attributed to increase in saturation. Despite the observed fluctuations in the iodine values of the oils, no significant effect was found on the iodine values from heating at different times (ranging 1–7 h) and temperatures (between 27 °C and 185 °C). The low iodine values observed for both palm olein oil and soybean oil at different frying times when heated at 185 °C is likely to have contributed to their greater oxidative stability than palm kernel oil and groundnut oil.

3.5. Free fatty acid

The oxidation and chemical changes in oils during heating are characterized by an increase in free fatty acid content and

decrease in the total unsaturation of oils (Perkin, 1992). Oxidative stability of oil is influenced by the fatty acid which is the chief composition of oil. From Table 2, the lowest FFA of 1.122–4.448 was recorded in palm kernel oil while the highest recorded FFA of 7.854–10.261 was found in groundnut oil. Only palm olein oil and palm kernel oil were within the maximum permissible FFA of 3.5mg/KOH/g for edible oils even after 7 h of heating. Before heating, (seen in Table 1), none of the oils were within the maximum permissible FFA as specified by SON (2000). Irregular high values of free fatty acid content as observed, might be due to impurities and variation during production, refining processes and also to the moisture content. Furthermore, high levels of FFA is an indication of hydrolytic rancidity which indicate the need for further refining before it can be used for specific purpose. According to Table 3, there was no significant effect of heating time and temperature on the fatty acid values of the oils, despite seeming that all the oils showed decrease in FFA after 7 h of heating. The decrease in FFA values of oils can be attributed to the esterification reaction between the FFA and the glycerol in the oil structure leading to the formation of triglyceride, and thus a reduction in FFA. There is also the possibility that low molecular weight acids in the oil may be lost through volatilization, hence causing a reduction in FFA (Hau et al., 1986).

4. Conclusion

The physicochemical properties of vegetable oil measured at room temperature were found to be outside permissible range except for the specific gravity. This indicate that the oils would need refining in order to meet recommended standard. When subjected to heat changes at different time, it was observed that palm olein oil and soybean oil were more stable to oxidation than palm kernel oil and groundnut oil. It was also observed that heating (frying) time and temperature had no significant effect on physicochemical properties of the oil. The findings, however, showed there was significant differences in the physicochemical properties when comparing the oil types which is inferred to be the result of variation in the oil production. The oxidative stability of the oils were found in the order: soybean oil > palmolein oil > groundnut oil > palm kernel oil.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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