

Effect of Heat Treatment on the Characteristics and Oil Yield of Moringa Oleifera Seeds

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-----Abstract-----

The effect of heat treatment on the characteristics and oil yield of moringa oleifera seeds was investigated. The moringa oleifera seed samples was divided into four portions A, B, C and D. Samples A, B and C were heated at 100⁰C, 130⁰C, and 150⁰C respectively for 30 minutes, while sample D served as the control for the experiment. The oil extraction was done using soxhlet extraction method and the oil extracted characterized using standard methods. The results showed a percentage oil yield of 33.7%, 32.2%, 30.9% and 28.6% for samples A, B, C and D respectively. The specific gravity and density (kg/m³) for the extracted oil are 0.96, 0.89, 0.91, 0.91 and 0.99, 0.97, 0.95, 1.05 for samples A, B, C and D respectively. Saponification (mg/KOH/mol) and iodine (g/100) values are 230.81, 218.75, 177.70, 252.34 and 69.25, 66.70, 66.63, 72.40 for samples A, B, C and D respectively. The acid (mg/KOH/g) and peroxide (m/mol/kg) values are 1.37, 1.36, 1.35, 2.73 and 1.94, 1.90, 1.90, 3.10 for samples A, B, C and D respectively. The free fatty acid (mg/KOH/g) ranged between 2.70 to 5.80 for oil samples extracted. It can be concluded that the oil yield, saponification value, free fatty acid, acid value, peroxide value and iodine value decreases with increase in heating temperature, while heating temperature has no significant effect on the specific gravity of the oil. The properties evaluated indicate the long shelf life, resistance to rancidity and edibility of Moringa seed oil.

Keywords: Moringa Oleifera seeds, oil yield, characteristics, heat treatment

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I. Introduction

Moringa Oleifera originates from North West India but has spread to tropical and subtropical regions worldwide and is now the most widely known and utilized of the 14 species of *moringa* tree. This might be because of the species ability to grow on many different soil types as well as its drought tolerance and growth capacity. *Moringa Oleifera* is a pan-tropical species that is known by such regional names as benzolive, drumstick tree, *kolar*, *Marango*, *Mlinge*, *Mulangay*, *nebedey*, *Saijhan* (Jed, 2005, Anwar, *et al.* 2007). The tree itself is rather slender, with dropping branches that grow to approximately 10m in height. In cultivation, it is often cut back annually to one meter or less and allowed to re-grow so that pods and leaves remain within arm's reach. *Moringa Oleifera* has very high nutritional value, this positions the plant high in the table of those "healthy edible plants and vegetables" as an important source of vitamins and minerals essential for health. The leaves contain high level of crude protein (19.3% - 26.4%) as well as several vitamins and minerals (Linda and Elina, 2007).

An average-sized *Moringa Oleifera* tree of fifteen to twenty feet high can produce hundreds or even thousands of seed pods, yielding countless *Moringa* seeds each and every year. *Moringa* seed can be extracted and eaten as "peas" (boiled or fried) when still green. The dry seeds are apparently not used for human consumption, perhaps due to the bitter coating when it becomes hardened or dry. The matured seed contains 38-40% oil which has an excellent quality (73% oleic acid) similar to olive oil. The *Moringa* seed oil also known as "Ben oil" is used in cooking, perfumes and soap making and as well as a lubricants. *Moringa* oil is slow to become rancid (Martins, 2007).

There are different methods of oil extraction from agricultural produce apart from the traditional methods. There are basically three (3) major methods used in the extraction of oil from various agricultural seeds. These are the mechanical/hydraulic pressing method, solvent extraction method and rendering method. Mechanical/Hydraulic method or press method, involve the direct application of forces to the agricultural material in other to get the oil out of the seed. The rendering method, involve the slow heating of the seed which results in the separation of oil/fat from the seed. The solvent extraction method involve the leaching out of the insoluble solid structure of the oil seeds by the use of volatile organic solvent e.g. n-Hexane, Isopropanol, Butanol, Acetane e.t.c (Obikili, 2010).

Olaniyan, 2010, investigated the effect of moisture content, heating temperature and heating time on the yield of oil expressed from castor bean. Results showed that generally, the oil yield at any pressure is dependent on the moisture content of the sample after heating. Sirisomboon and Kitchaiya, 2008 reported that the total amount of oil extracted from jatropha kernels depend mainly on the extraction time and temperature, moisture content and particle size of the oil bearing material, with the oil extracted during the first 20 minutes of extraction. They also reported that oil recovery increases with decrease in moisture content.

Most oil seeds and nuts are heat-treated by roasting to liquefy the oil in the seed cells and facilitate its release during extraction. All oil seeds and nuts undergo this treatment except palm fruits for which "sterilization" replaces this operation. For large scale production, oil seeds are dried to moisture content of 10%. According to Olaniyan (2010) an increase in the heating temperature and heating time increases the free fatty acid value, peroxide value and the colour intensity of the oil expressed. The effects of heating temperature, heating time, applied pressure, and duration of pressing on the yield of oil expressed from castor bean was investigated by Olaniyan 2010. Result showed that, generally, the oil yield at any pressure was dependent on the moisture content of the sample after heating, temperature and heating time. High oil yields were obtained from samples with moisture contents between 8 and 10% after heating. The maximum oil yield of 39.6%, corresponding to an extraction efficiency of 66 %, was obtained when milled conophor nut conditioned to 11% moisture was heated at 65°C for 28 min and expressed at a pressure of 25 MPa. Oil expressed under the conditions stated above was of good quality with a free fatty acid value of 1.18% (Olaniyan, 2010).

Tunde-Akintunde *et al.* (2001) investigated the effect of moisture content, heating temperature, heating time, applied pressure and pressing time on mechanically expressed soybean oil. Results showed that oil yield increased as moisture content was varied from 7.3 to 10.2%, heating temperature from 70 to 80°C and heating time from 15 to 30 min. The highest oil yield of 10.4% was obtained when sample at 10.2% moisture content were heated for 30 min at a temperature of 80°C. The colour and FFA of the oil expressed at these processing parameters is within acceptable range for crude soybean oil. There are limited or no information on the processing parameters necessary for the optimum extraction of *moringa* seed oil. Hence the knowledge of the appropriate set of parameters for the extraction of *moringa* seed oil will enhance the production and quality attributes of *moringa* seed oil. The main objectives of this work are to evaluate the effect of heat treatment on the oil yield of *moringa* seed and to characterize the quality of the oil produced.

II. Materials And Methods

2.1 Sample Preparation

Moringa Oleifera seeds were obtained from Kure Market in Minna, Niger State, Nigeria. The seed samples were cleaned thoroughly to remove dirt, stones and deteriorated seed. The cleaned seeds were shelled and the initial moisture content of the kernel was determined using the AOAC, 1990 standard procedure with the sample heated for 6h at 100°C (Young, *et al.* 1998, Tunde-Akintunde, *et al.* 2001). The seed sample was then divided into samples A, B, C and D of 267.2 g each. Samples A, B and C were heated for 30 minutes at 100°C, 130°C and 150°C, while D was used as control for the experiments. A laboratory oven was used for heat-treating the *moringa* seed samples through the selected temperatures prior to oil expression.

2.2 Oil Extraction Using Soxhlet Method

The Soxhlet method is the most commonly used semi-continuous process for the extraction of lipids from foods (Obikili, 2010). According to Soxhlet procedure, oil and fat from solid material are extracted by repeated washing (percolation) with an organic solvent, usually hexane or petroleum ether. The n-hexane was used for the purpose of this work. The grounded *moringa* seed samples were placed in a porous cellulose thimble. The thimble is then placed in an extraction chamber which is being suspended above a flask containing the solvent and below a condenser. Heat is being applied to the flask and the solvent evaporates and moves to the condenser where it is converted into liquid that trickles into the extraction chamber containing the sample (Obikili, 2010). The extraction chamber is made in such a way that when the solvent surrounding the sample exceeds a certain level it overflows and trickles back down into the boiling flask. The flask containing solvent and lipid is removed at the end of the extraction process. The solvent in the flask is evaporated in an oven and the mass of the lipid remaining is measured. The percentage of the lipid in the initial sample is then calculated.

2.3 Determination of Percentage Oil Yield

The extraction of oil using soxhlet extractor was repeated on each of the sample and the oil was recovered by solvent evaporation. It was heated at a temperature higher than that of the solvent until the solvent finally evaporates leaving behind the extracted oil. The procedure was carried out for all samples. The average oil yield on each sample was obtained.

The percentage oil yield was calculated as follow;

$$\% \text{ oil yield} = \frac{\text{Weight before extraction} - \text{Weight of sample after extraction}}{\text{Weight of sample before extraction}} \times 100$$

2.4 Determination of Specific Gravity and Density

Hundred millilitres (100ml) of specific gravity bottle was cleaned and dried in a dry-air oven. Then it was cooled in a desiccator. The weight of the specific gravity bottle was obtained as W_1 , then the specific gravity bottle was filled with clean water, and the weight of the bottle plus water obtained as W_2 . The water was poured out and the bottle allowed to dry (Ibrahim and Onwualu, 2005). The specific gravity bottle was again filled with oil and the weight of the specific gravity bottle oil obtained as W_3 . The specific gravity and relative density of the oil is calculated using the formula below;

$$\text{Density} = \frac{\text{Weight of oil}}{\text{Volume of oil}} \quad \text{i.e.} \quad \frac{W_3 - W_1}{100}$$

$$\text{Specific gravity,} = \frac{\text{Weight of oil}}{\text{Weight of equal Volume of H}_2\text{O}}$$

2.5 Determination of Saponification Value

Two grams (2g) of sample weighed into a 250 cc. conical flask and added 25cc of approximately normal alcoholic potash (prepared by dissolving 56g KOH in 1 litre of alcohol) and a few glass beads. The mixture was then boiled gently under reflux on a water bath for 1hr. while the process is on (refluxing), titrate 25ml of the alcohol potash against 0.5M HCl, using phenolphthalein as indicator. After boiling, the mixture was titrated with 0.5M HCl, immediately. The end point will be a faint pink (Obikili, 2010).

$$\text{Saponification Value} = \frac{(B-A) \times 28.05}{\text{Weight of oil sample}} \text{ mg. KOH/gm.}$$

Where; B=Blank titre, A= sample titre

2.6 Determination of Free Fatty Acid

Acid value is expressed as % free fatty acid calculated as oleic acid. The dish was accurately weighed containing about 5g of the oil sample, poured into a conical flask and re-weighed, thus obtaining the actual weight of the oil taken. Fifty milliliters (50ml) of hot neutral alcohol was added with a few drops of phenolphthalein and shaken vigorously. The solution was titrated with 0.5 M sodium hydroxide (NaOH) solution with constant shaking until the pink colour remains constant. From the quantity of 0.5M alkali used, the percentage of acid present was calculated, stating the result in terms of oleic acid (Farooq *et al.* 2006).

2.7 Determination of Peroxide Value

One gram (1g) of oil sample was added with 1g of potassium iodide and 20ml glacial acetic acid chloroform 2:1. It was then boiled for 1 min. The hot solution was transferred into a flask containing 20ml of 5% potassium iodide solution. And few drops of starch solution was added and titrated with 0.025 N $\text{Na}_2\text{S}_2\text{O}_3$ to a faint yellow colour. One millilitre (1ml) of starch indicator was added and the titration continued until the blue colour disappears. Determine using AOAC 1990 method (Eromosele, *et al.* 1994).

$$\text{Peroxide Value} = \frac{\text{Molar equivalent}}{\text{Weight of oil Sample}} = \frac{S \times N \times 100}{\text{Weight of oil Sample}}$$

Where; S = Weight of $\text{N}_2\text{S}_2\text{O}_3$ used, N = Normality of $\text{N}_2\text{S}_2\text{O}_3$

2.8 Determination of Iodine Value

Five millilitre (5ml) of chloroform solution was taken and 5ml of Dan's reagent (acetic acid + CHCl_3) was added, the solution was kept in fume cupboard for 10min. Five millilitre (5ml) of 10% potassium iodite was added with 20ml of distilled H_2O , stirred several times to mix solution and Titrate to a colourless end point with 0.025N $\text{N}_2\text{S}_2\text{O}_3$ (Eromosele, *et al.* 1994).

$$\% \text{ Iodine Number} = \frac{(B-A) 0.00317 \times 0.001269 \times 100}{\text{Weight of sample}}$$

Where; B = Blank Titre, A= Sample Titre, 1ml 0.025 $\text{N}_2\text{S}_2\text{O}_3$ = 0.0 0317g

2.9 Statistical Analysis

The data obtained were analysed statistically using the software package SPSS 15.0 (Statistical package for social science).

III. Results and Discussion

3.1 Presentation of Results

The results shows that the seeds sample A, B and C heated at 100⁰C, 130⁰C and 150⁰C has 5.25%, 3.40% and 2.50% moisture contents respectively before the extraction process. The control sample had 10% moisture content before extraction. The results of the effect of heat treatment on the physical characteristics of *moringa* seed oil are as presented in Table 1. The chemical characteristics of the *moringa* seed oil are as presented in Table 2.

Table 1: Physical Properties of *Moringa Oleifera* oil

Parameters	Sample A	Sample B	Sample C	Sample D	FAO/WHO Standard
Oil Yield (%)	33.7%	32.2%	30.9%	28.6%	38 - 40%
Specific Gravity	0.964	0.891	0.911	0.909	0.9 - 1.16
Density (kg/m ³)	0.99	0.97	0.95	1.05	-

*F.A.O/W.H.O International Standard for Edible Oils

Table 2: Chemical Properties of *Moringa Oleifera* Oil Samples

Parameters	Sample A	Sample B	Sample C	Sample D	FAO/WHO Standard
Saponification Value (mg/g)	230.81	218.75	177.70	252.34	181.4 ± 2.60
Free Fatty Acid (mg/KOH/g)	2.74	2.71	2.70	5.80	5.78 - 7.28
Acids Value (mg/KOH/g)	1.37	1.36	1.35	2.73	4
Peroxide Value (m/mol/kg)	1.94	1.90	1.90	3.10	10
Iodine Value (g/100g)	69.25	66.70	66.63	72.40	80 - 106

*F.A.O/W.H.O International Standard for Edible Oils

3.2 Discussion of Results

The percentage oil yield was compared with the results obtained by Martins (2007) on the oil contents of *moringa* seed, while the physical and chemical properties were compared to FAO/WHO (2009) international standard for edible oil.

3.2.1 The Effect of Heat Treatment on the Oil Yield of *Moringa* Seeds.

The oil yields for *moringa* seeds heated at 100⁰C, 130⁰C and 150⁰C were 33.7%, 32.2% and 30.9% respectively. The unheated seeds (control) had an oil yield of 28.9%. This results shows that the oil yield for sample A (100⁰C) has the highest percentage yield of 33.7%, while the control has the lowest percentage yield of 28.9%. The results also show that there is a reduction in percentage oil yield when the seed is heated above 100⁰C (Fig 1). The oil yield for all the pre-treated samples were lower than the 38-40% oil content for *moringa* seed as reported by Martins (2007). This variation is probably due to the processing methods, varieties of seeds and soil conditions.

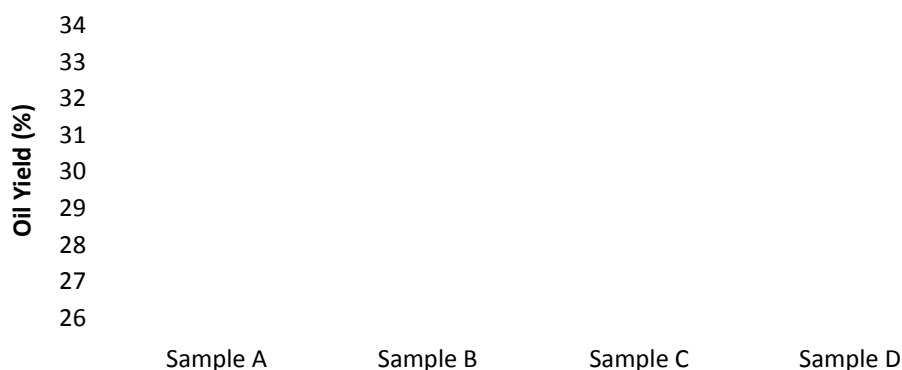


Figure 1: The Effect of Heat Treatments on the Oil Yield of *Moringa* Seeds

3.2.2 The Effect of Heat Treatment on the Specific Gravity of *Moringa* Seed Oil

The specific gravity of samples A, B, C and D are 0.92, 0.91, 0.90 and 0.96 respectively. The values are within the range given by FAO/WHO (2009) for edible oil which is 0.9-1.16. The specific gravity increases in a non-uniform linear manner, as the temperature increases the specific gravity decreases (Fig 2).

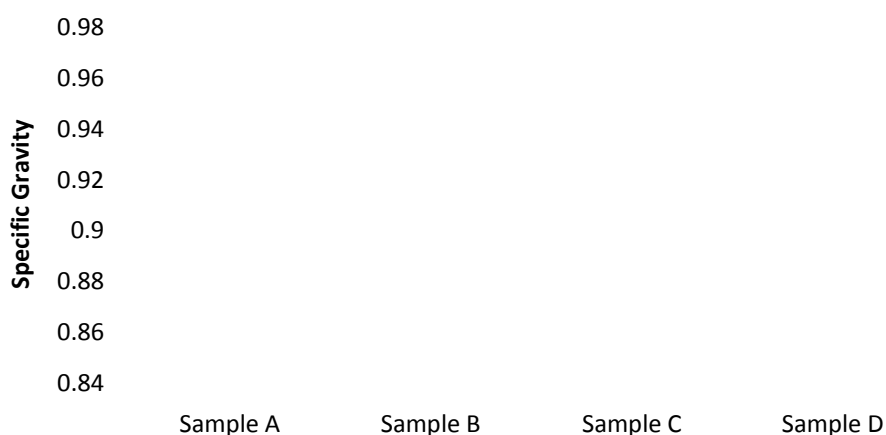


Figure 2: The Effect of Heat Treatment on the Specific Gravity of *Moringa* Seed Oil

3.2.3 The Effect of Heat Treatment on the Density of *Moringa* Seed Oil

The densities of the samples A, B, C and D are 0.99, 0.97, 0.95 and 1.05 respectively. This shows that the higher the temperature, the lower the density of oil. The control has higher density value compared to the other samples (Fig. 3). This is probably due to the fact that an oil bearing seed tends to lose some of its properties such as weight when heated hence resulting in lower density.

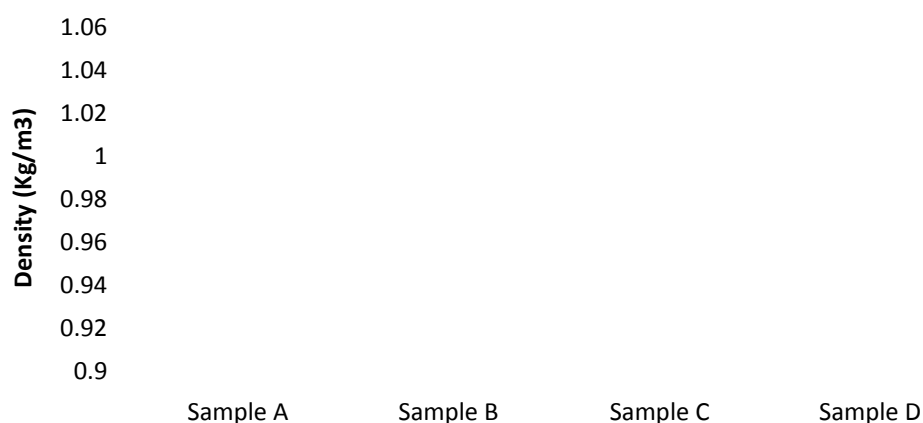


Figure 3: The Effect of Heat Treatment on the Density of *Moringa* Seed Oil

3.2.4 The Effect of Heat Treatment on the Saponification Value on *Moringa* Seed Oil

The saponification value of samples A, B, C and D are 230.81, 218.75, 177.70 and 252.34 respectively. These values are within the range recommended by FAO/WHO (2009) international standard for edible oil which is 181 ± 2.60 . The saponification value decreases with an increase in temperature. The control (not heated) had the highest saponification value of 252.34 (Fig. 4).

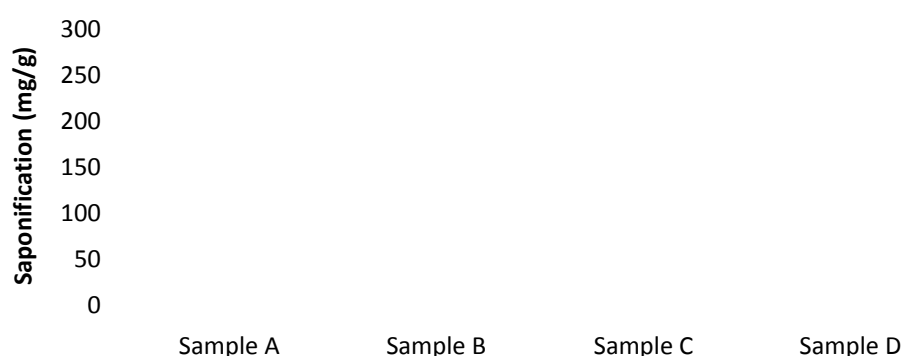


Figure 4: The Effect of Heat Treatment on the Saponification Value on *Moringa* Seed Oil

3.2.5 The Effect of Heat Treatment on the Free Fatty Acid of *Moringa* Seed Oil

The free fatty acid (mg/KOH/g) of samples A, B, C and D are 2.74, 2.71, 2.70 and 5.80 respectively. The values of the free fatty acid for the heated seeds samples are lower than the FAO/WHO (2009) standard for edible oil, while the unheated is within the recommended standard. The higher the temperature the lower the free fatty acid, the control (not heated) had the highest free fatty acid value (Fig. 5). The free fatty acid value measures the extent to which the glycerides in the oil have been decomposed by lipase action. This decomposition is accelerated by light and heat, hence, rancidity is usually accompanied by free fatty acid formation.

3.2.6 The Effect of Heat Treatment on the Acid Value of *Moringa* Seed Oil

The acid value for samples A, B, C and D are 1.37, 1.36, 1.35 and 2.76 respectively. These are within the range specified for edible oil as given by FAO/WHO (2009) which is 2.89 ± 0.01 . The acid value decreases in a uniform linear manner with an increase in temperature (Fig. 6) hence, the higher the temperature, the lower the acidity value, indicating the edibility of *moringa* oil.

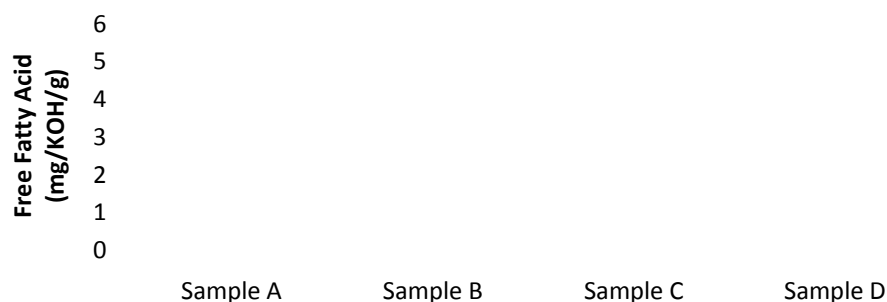


Figure 5: The Effect of Heat Treatment on the Free Fatty Acid of *Moringa* Seed Oil

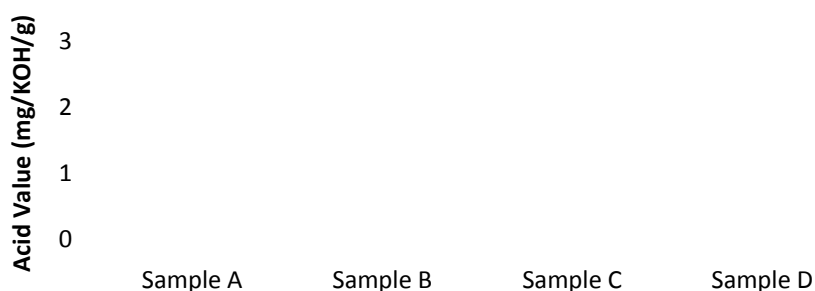


Figure 6: The Effect of Heat Treatment on the Acid Value of *Moringa* Seed Oil

3.2.7 The Effect of Heat Treatment on the Peroxide Value of the *Moringa* Seed Oil

The Peroxide value of samples A, B, C and D are 1.94, 1.90, 1.90 and 3.10 respectively. These are in line with the standard specified by FAO/WHO (2009) for fresh edible oil which is below 10m/mol/kg. Peroxide value is used to monitor the development of rancidity through the evaluation of the quantity of peroxide generated in the product (initiation product of oxidation). The peroxide value is usually less than 10 per gram of a fat sample when the sample is fresh. The peroxide value decreases with an increase in temperature (Fig. 7). The lower peroxide value of *moringa* oil indicates its resistance to rancidity. The best oil samples are B and C in terms of resistance to rancidity.

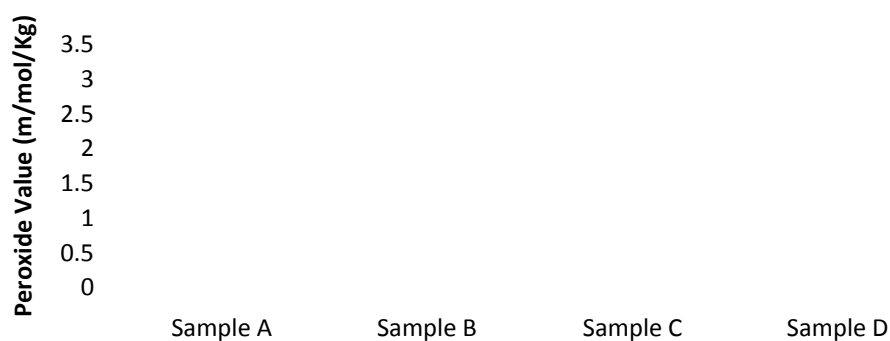


Figure 7: The Effect of Heat Treatment on the Peroxide Value of the *Moringa* Seed Oil

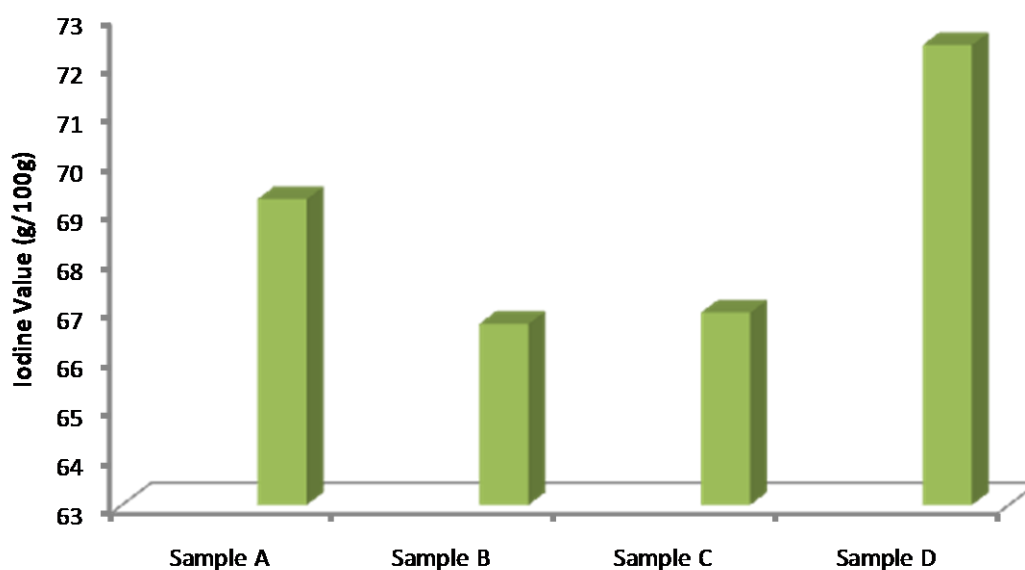


Figure 8: The Effect of Heat Treatment on the Iodine Value of *Moringa* Seed Oil

3.2.8 The Effect of Heat Treatment on the Iodine Value of *Moringa* Seed Oil

The iodine value (100/g) of samples A, B, C and D are 69.25, 66.70, 66.63 and 72.40. These are lower than the range (80-106) specified by FAO/WHO (2009) for edible oil. Sample D has the highest value which is the sample that is not heated, although they all fall below the standard for edible oil. The iodine value decreases in a uniform manner with an increase in temperature (Fig. 8). The iodine value is a measure of the unsaturated acid present in the oil. Therefore, the test measures the amount of iodine consumed by the acid. The lower value indicates a lower degree of unsaturation. Thus sample C (heated at 150°C) has the lowest iodine value, indicating that the higher the temperature, the lower the iodine value.

IV. Conclusion

The following conclusions can be made from this work:

- i. *Moringa* seed oil yield decreases with increase in heating temperature.
- ii. Heat treatment has no significant effect on the specific gravity and density of *moringa* seed oil.
- iii. Heat treatment decreases the saponification value, free fatty acid, acid value, peroxide value and iodine values of *moringa* seed oil.
- iv. Heating *moringa* seeds at 100⁰C for 30 minutes, gave the highest oil yield compared to the control and samples heated above 100⁰C.
- v. The physical and chemical properties of the *moringa* seed oil evaluated indicate its edibility and longer shelf life.

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