International Journal of Chemistry; Vol. 8, No. 3; 2016
ISSN 1916-9698 E-ISSN 1916-9701
Published by Canadian Center of Science and Education

Determination of the Proximate Composition, Physicochemical Analysis and Characterization of Fatty Acid on the Seed and Oil of *Gossypium Hirsutum*

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Received: June 22, 2016 Accepted: July 4, 2016 Online Published: July 25, 2016

doi:10.5539/ijc.v8n3p57 URL: http://dx.doi.org/10.5539/ijc.v8n3p57

Abstract

The proximate composition, physicochemical analysis and characterization of *gossypium hirsutum* seed and its oil was carried out. The parameters that were studied in proximate analysis include (percentage) % moisture, % crude protein, % crude fiber, % ash content, % lipid. The total % carbohydrate content of the seed was 30.49±0.4 and the estimated % energy value was 435.58. The chemical parameters analyzed on the oil include iodine value, saponification value, free fatty acid (FFA) value and % volatile matter. In the characterization of fatty acid present in the oil with GC-MS Spectroscopy system, the major unsaturated fatty acid values were 14.53% for oleic acid and 55.38% for linoleic acid while the palmitic acid and stearic acid values which were saturated acid are 27.39% and 2.23% respectively. The percentage values of the rest of the fatty acid present in the oil were very low. The parameters determined were within the international and Nigerian industrial standard for vegetable oil.

Keywords: physicochemical, characterization, proximate and gossypium hirsutum

1. Introduction

Any nation that requires development must search inward to identify areas where it has comparative advantage over other nations and seek to develop the identified areas. Apart from hydrocarbons, Nigeria has advantage in agricultural sector where varieties of products are produced due to the favorable climatic condition, good soil and the fact that over 70% of the entire land mass of the country is arable. Oil seed is one of these major agricultural produce and serve as main source for production of edible oil that are normally called vegetable oil (Akubugwo et al; 2009). Cotton is a natural vegetable fiber obtained from the cotton plant of the genus *gossypium* and belongs to malvacae family. It is a shrub which grows to about 40 cm high (Shekhar, 2003) and grows in tropical and subtropical areas with its flowers either red or yellow. The capsules (seeds) of this plant are formed as soon as the petals fall off and burst open into four parts upon maturity thereby revealing the cottonseeds after which they are harvested mechanically. The most destructive pest that attacks cotton plants is the "boll weevil" (Bedigia, 2007). Cotton seed oil is use for cooking or frying at home, use for soap production, body cream and margarine. The cakes as by-products after extraction of oil are very useful in the production of animal feeds.

In some literature review concerning cotton seed oil, the % oil content of cotton seed is 31.33 and in the characterization of the oil, % of unsaturated fatty acid is more of oleic and linoleic acid (74%) and the results of physicochemical properties obtained, showed that the oil has high industrial value (Yan-Zhuan, 2010). Ojewola et al (2006) evaluated the physicochemical composition and nutritional value of cotton seed cake. The results showed that cottonseed cake can be used as substitute for soybean meal in broiler and these will go a long way to reduce the price of poultry meat products and the competition between man and animals for soybean as plant protein source thereby reducing the market price of soybean. Ikurior and Babatunde (1987) studied the proximate analysis, mineral and amino acid composition on recommended Nigeria commercial cottonseed varieties, Samara (S71), S72, and S77 collected from different locations, Funtua cottonseed (CSF), Malluumfashi cotton seed (CSM) and Kano cotton seed (CSK). In proximate analysis, results obtained did not vary between or within seed types while the location factors appear to influence the chemical components of the seeds. The effect of interestified palm oil and cottonseed oil blend in cookie in the ratio of 1:1 gave better results in proving the effectiveness of interesterified oil (Washeed et al 2010).

2. Material and Method

2.1 Material

Cotton seeds were obtained from the local market in Kano state. The seeds were dehulled with manual machine and after which they were wrapped in polyethene bag and kept in a desiccator until when needed.

2.2 Extraction Method

Solvent extraction method was used to extract oil from the processed *gossypium hirsutum* with soxhlet reflux apparatus according to Akpan et al., (2006). 300ml of solvent extractant (ethanol, 60^0C – 80^0C) was poured into round bottom flask containing 10g of the sample. The soxhlet was heated at 60^0C and as the solvent boils, the vapour rises through the vertical tube with the condenser at the top. The liquid condensate drips into the thimble containing the solid sample to be extracted. The extract seeps through the pores of the thimble and fills the siphon tube, from where it flows back down into the round bottom flask. This process was allowed to continue until all the oil was extracted from the sample.

2.3 Proximate Analysis

2.3.1 Determination of Percentage (%) Moisture Content

The moisture content of the processed samples was determined by vacuum oven method. 3g of sample were weighed into crucibles of known weight. The crucibles with the samples were placed into an oven operated at 105^0C for 3 hours. They were carefully removed from the oven and allowed to cool in desiccators before re-weighing. The crucible with the sample were returned to the oven and dried further and re-weighed. The process of drying, cooling and re-weighing was continued until a constant weight was obtained.

2.3.2 Determination of Ash Content

2g of the sample were weighed into crucible of known weight. The crucible with its content was placed in a furnace set at 550^0C for 6 hours after which it was carefully removed from the furnace and allowed to cool before being re-weighed. The process was continued until a constant weight was obtained.

2.3.3 Determination of Crude Protein

The crude protein was determined by Kjeldahl method. 2g of processed sample were taken and placed into 100ml Kjeldahl digestion flasks. Few grams of Kjeldahl catalyst mixture (Na_2SO_4 +$CuSO_4$) and 15ml of conc.H_2SO_4 were added. The mixtures were mixed thoroughly and heated in a fume cupboard for about 2 hours until complete digestion was reached. This was identified when a clear solution was obtained. The cool digest was diluted to 100ml and only 10ml of the digest was mixed with equal volume of 10mol/dm^3 NaOH. The mixture was placed in micro- Kjeldahl distillation apparatus which was distilled by steam and the distillate was collected into a conical flask containing 10ml of 4%Boric acid. Few drops of mixed indicator (5g bromocrysol green and 1g methyl red in 100ml of ethanol) were added into 50ml of the distillate and titrated against 0.1M H_2SO_4 solution. A blank was conducted simultaneously under similar experimental condition.

2.3.4 Determination of the % oil Content

3g of the processed samples were weighed into a known weight of thimble. A clean dry extraction flask (250ml capacity) was weighed and sufficient quantity of 40^0C – 60^0c acetone was poured into it. The thimble with the sample was placed into the flask and oil was extracted as already described above. At the end of extraction, the resulting mixture containing oil was heated to recover the solvent and the weight of the round bottom flask with oil was noted after cooling.

2.3.5 Determination of Energy Value

The energy value (kcal) of the samples was estimated by multiplying percentage crude protein, crude lipid and carbohydrate by the recommended factor (3.44, 8.37 and 3.57 respectively) used in vegetable and seed analysis (Akubugwo et al., 2008).

2.4 Chemical Analysis

2.4.1 Determination of Iodine Value

The iodine values of the oils were determined using Wijs's iodine method. 20ml of carbon tetrachloride (solvent) was added into 0.2g sample that was placed into 500ml flask and the mixture was swirled. 25ml of Wijs's iodine solution was pipette into flask containing the sample and covered with glass stopper and swirled to ensure an imitate mixture. A blank was prepared simultaneously under similar experimental condition. The flasks were stored in the dark for 30 minutes at a temperature of 25^0C $\pm$ 5^0C after which 15ml of potassium iodide (KI) solution was added, followed by 100ml of distilled water. The solutions were titrated with 0.1M sodium thiosulphate solution using starch as an indicator with constant and vigorous shaking.

2.4.2 Determination of Saponification Value

2g of the samples were taken into conical flask and 25ml of 0.1M alcoholic potassium hydroxide was added. The contents which were constantly stirred were allowed to boil gently for 1 hour with reflux condenser placed on the flask containing the mixture. Few drops of phenolphthalein indicator was added to the warm solution and then titrated with 0.5M HCI to end point (until color of the indicator disappeared). The same procedure was used for the blank.

2.4.3 Determination of Free Fatty Acid (FFA)

25ml of diethyl ether and 25ml of ethanol were mixed in a 250ml beaker. The resulting mixture was added to 10g of oil in a 250ml conical flask and few drops of phenolphthalein were added into it. The mixture was titrated with 0.1M NaOH to end point with constant shaking; the volume of 0.1M NaOH was noted.

2.4.4 Determination of Acid Value (A.V)

10g of oil sample was weighed in a conical flask. $50cm^3$ of ethyl alcohol and few drops of phenolphthalein indicator solution were added to the conical flask with the oil sample. The mixture was heated for 5 minutes and then titrated with 0.05M KOH to a faint pink color end point with constant shaking.

2.4.5 Determination of Moisture Content and Volatile Matter

The moisture content and the volatile matter of oil was determined using air oven method. 15g of oil sample was weighed into a known weight of tarred Petri dish. The Petri dish was placed in the oven for approximately 2 hours. The dish was carefully removed from the air oven, cooled and was re-weighted. The process of drying, cooling and reweighing was continued until a constant weight was obtained.

2.4.6 Determination of Percentage Fatty Acid Content

0.2g of oil sample were weighed into 250ml conical flask and methylated with 6ml of sodium methyloxide. The mixture was refluxed for 10 minutes on steam bath, thereafter 10ml of chloride was added and refluxed for another 10 minutes.10ml of hexane was added and refluxed for more 2 minutes after which the solution was allowed to cool. 10ml of distilled water was added and poured into a separating funnel, organic layers collected and dried over $CaCl_2$. The samples were injected into the GC-MS (QP2010 PLUS, SHIMADZU JAPAN) to identify the fatty acid present in the oil samples.

3. Results and Discussion

Table 3.1. Proximate composition of *gossypium hirsutum*

Seed Parameter	% Composition
Moisture	9.87 $\pm$ 0.10
Crude protein	27.27 $\pm$ 0.03
Crude fibre	22.94 $\pm$ 0.28
Ash	4.55 $\pm$ 0.06
Lipid	27.83 $\pm$ 0.35
Total carbohydrate	30.49 $\pm$ 0.41
Nitrogen free extract (N F E)	7.55 $\pm$ 0.56
Energy value (Kcal/100g)	435.58

Each data is mean of three replicates $\pm$ standard deviation (SD)

Table 3.1 shows the result of proximate analysis of *gossypium hirsutum.* The moisture content of *gossypium hirsutum* seeds is a bit low when compared with legumes which range between 7.0-11.0% (Arkroyed and Dought, 1984). This shows that the seeds are very high in dry matter content which is an advantage because it reduces microbial activities, prevent oxidation-reduction reaction, algae and fungi growth and increase their shelf life when properly stored. The % protein obtained is similar to the values of seeds rich in protein (soybeans, cowpeas, pigeon peas etc) which were between 23.1 - 33.0% (Olaofe et al., 1994). Their high % protein will make them serve as a proper source of amino acids and protein for both man and animal and this value exceeded FAO recommended vale (19.8%) (FAO/WHO, 2004). *Gossypium hirsutum* seed is high in crude fibre when observe from the result, it has enough fibre for dietary nutrition which will help to maintain intestinal distention, reduce constipation, colon diseases and cancer (Njoku et al., 2007). The % ash indicates high inorganic matter that could be retained in the body while the % oil (27.8 $\pm$ 0.35%) reveals that the seed is rich in oil and fat and is very important in diet as it promotes absorption of fat, soluble vitamin and provide high energy nutrient. The value of this seed oil is quite promising and suggests obtaining commercial quantities for industries, pharmaceutical, cooking and other purposes. Their total calculated carbohydrate is considered sufficient for energy the body required.

Table 3.2. Physical properties of oil extracted from *gossypium hirsutum* seed

Seed properties	***gossypium hirsutum***
Percentage (%) oil yield	27.83 ± 0.35%
State at 25^0C	Semi liquid
Colour	Dark brown
Odour	Pleasant

From table 3.2, *gossypium hirsutum* oil is dark oil when in its crude state. It is semi-liquid at normal room temperature and has pleasant smell. The percentage oil extracted was 27.83 ± 0.35%.

Table 3.3. Chemical properties of oil extracted from *gossyspium hirsutum*

Chemical properties	***Gossypium hirsutum***
Iodine value (g iodine/100g)	111 ± 0.72
Saponification value ($mgKOHg^{-1}$)	193.31 ± 0.31
Free fatty acid (%)	1.88 ± 0.20
Acid value ($mg\ KOHg^{-1}$)	3.76 ± 0.10
Volatile matter (%)	0.60 ± 0.04

Chemical properties of oil are among the most important properties that determines the present condition of oil (Nzikou et al., 2009). The iodine value which indicates the degree of oil saturation shows that the oil of *gossypium hirsutum* is semi-drying and unsaturated and this makes it suitable for utilization in certain industrial applications (Ibiyemi et al., 1992). The low acid value and free fatty acids shows the ability of the oil to resist hydrolytic rancidity (Akubor 2008). The saponification value of cotton seeds is 193.31 ± 0.31mgKOH/g and it fall within the range of values obtained for some vegetable oil 188 - 235mgKOH/g (Aremu et al., 2006).

The major unsaturated fatty acids in *gossypium hirsutum* oil were oleic and linoleic and that of saturated fatty acids were palmitic and stearic acids which is similar with the report of Qing Liu et al., (2002). The oil contains high % of linoleic as seen in table 3.4 below and it is one of the most important essential polyunsaturated fatty acid in human food because of the inability of the body to synthesize it. It's present in the body prevents distinct heart vascular diseases in the body (Boelhouwer, 1983).

Table 3.4. Results of fatty acids composition of oil extracted from *gossypium hirsutum*

Fatty acids names present in the oil	**Fatty acids with their number of carbons and double bonds present.**	**% value of fatty acid in the oil**
Hepta2,3-dienoic acid	C7:2	0.02
Myristic acid	C14:0	0.23
Palmitoleic acid	C16:1	0.04
Palmitic acid	C16:0	27.39
Oleic acid	C18:I	14.53
Stearic acid	C18:0	2.28
Linoleic acid	C18:2	55.38
Methyl Ricinoleic acid	C19:1	0.01

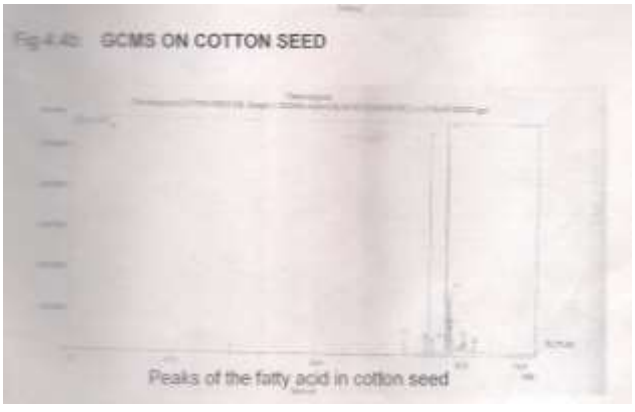

Figure 1. GCMS on Cotton Seed

4. Conclusion

From the results of the analysis carried out, oil of *gossypium hirsutum* collected within Kano metropolis could be within the specification of international and Nigeria industrial standard for vegetable. The seed and oil extracts of *gossypium* exhibited good physiochemical properties and could be useful for industrial application.

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