



PRODUCTION AND REFINING OF CORN OIL FROM HOMINY FEED A BY-PRODUCT OF DEHULLING OPERATION

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ABSTRACT

The focus of this work is to produce and refine corn oil from the by-product of the local dehulling/degermination of corn with soxhlet apparatus using n-hexane as solvent. The extraction was done for duration of three and six hours using 12g each of unsieved sample and various fractions of the sample. Average oil yield were 14.28% and 14.50% for extraction time of three and six hours respectively. The extracted crude oil was refined to improve its quality by degumming, alkali refining and decolourization. Both the crude and refined corn oil was analyzed to determine some of their properties and subsequently, the results were compared with standard values. There were indications that the oil yield of the sample can be improved upon for it to serve as raw material for the production of corn oil.

Keywords: corn oil, hominy feed, dehulling, refining, by-product.

Nomenclature

C = concentration of sodium thiosulphate
 V_1 = Volume of sodium thiosulphate solution used for blank test
 V_2 = Volume of sodium thiosulphate solution used for determination
c = concentration of hydrochloric acid
 v_o = Volume of hydrochloric acid solution used for blank test
 v_1 = Volume of hydrochloric acid solution used for determination
M = M_0 = Mass of the test sample
 M_1 = is the mass of residue
 M_2 = mass of residue obtained with the blank
 M_3 = mass of free fatty acid and equal to 0.28VC
T = the normality of the sodium thiosulphate used
W = volume of 1 molar ethanol potassium hydroxide solution used for titration.
X = is the concentration in molar of the ethanolic potassium hydroxide solution

INTRODUCTION

Corn oil, a product which is obtained from corn, is an oil of premium quality and has long been preferred by discerning consumers. A pointer to this is the fact that annual production of crude corn oil currently exceeds 2.4 billion pounds in United States of America [5]. In addition, according to [4], 324 million gallons (1227 million L) of corn oil are estimated to be extracted in the US in 2009/ 2010.

Corn oil has many applications, including its use in pharmaceutical industry, manufacture of resins, plastics, lubricants and fuels. However, majority of oil produced is refined for direct consumption and for use by food industry. The reason for this is not far-fetched. Corn oil when refined has neutral flavour; relatively high smoking point and can therefore withstand heat. Also research has revealed that its consumption has health benefits [5]. In

developing countries, according to [9], most of the cultivation of corn is for human consumption.

Starch, nutritive sweeteners, feed products in addition to corn oil is among some of the products which can be derived from the industrial processing of corn. Common techniques of industrial processing include dry milling process and wet milling process. Processes have been developed to separate corn germ with increasingly higher purity: Quick germ [17], enzymatically milled germ [12] and MOR-Frac, a dry-milling followed by wet-mill refining [15] report germs with 30, 39, and 42% oil, respectively. According to [10], oil is currently removed from dry-milled germ or wet-milled germ by crushing for 35-45 \$/ tonne or by hexane extraction for 20-40 \$/ tonne. These costs, as well as the capital to build an oil recovery facility on site, are significant hurdles to separating corn germ, and then oil, from dry-grind ethanol plants. The unrefined product oil will be worth about \$450/ tonne. Oil yields of 65 wt % can be recovered from wet-milled or dry-milled germ by processing [3].

An approach that has been recently reported improves aqueous extraction yields of dry-fractionated germ by using enzyme(s) selected to weaken or degrade oil bodies present in the germ. The oil bodies may be degraded in wet-mill germ during steeping and or drying of the kernels of the germ [14].

The optimal pH of the wet-mill germ dispersion was about 4.0, prior to separation of the foam fraction [2]. In their later work, [1], found out that the procedure developed to extract oil from wet-mill germ with aqueous enzymes did not work with dry-fractionated germ; when the enzyme solution-to-germ mass ratio was raised to 3:80 the yield was 9%. They reported a description of an aqueous enzymatic extraction method to separate oil from dry-fractionated corn germ. The method they used is an extension of a method they previously developed for wet-mill germ. They used a microwave oven to cook the germ to its highest temperature, just short of burning. Thereafter they extracted the germ using the method developed for



wet-mill germ: mixed the heated germ with water and cooked it under pressure, followed by colloid milling and enzymatic digestion of the milled germ particles overnight. A foam fraction was then removed from the digested dispersion by bubbling nitrogen through a short column connected to a mixing tank. The foam fraction was then centrifuged to separate free oil.

Corn has several means of preparation for human consumption, locally is the dehulling/degermination of the corn and subsequent milling into flour. The by-product which results is sometimes called hominy feed and is used as an animal feed.

When used as animal feed, corn and by-products of its processing serve principally as source of energy in the feed formulation. However, poor harvest yield of corn and increasing demand for animal feed by the expanding livestock production industry has led to use of alternatives such as sorghum, cassava and sweet potato to replace corn's function in the feed formulation. In addition, feed produced from corn and its derivatives have other limitation for usage as feed for animals [7]. Worthy of mention here is cassava, whose availability and subsequent usage in Nigeria is set to increase with recent favourable policies by government on it.

Currently, corn oil is produced industrially using germ separated from the maize kernel as raw material. Locally, several research works have been done in production of oil from maize kernel. An example is the study carried out by [11]. Though, industrial processing of corn would produce a wide array of products, the availability of corn to satisfy local production is uncertain. This is due to the fact that much of production of corn is for human consumption and comparatively lower harvest yields would likely have an impact on its supply. However, hominy feed contains maize germ, which has a significant amount of oil [5]. The hominy feed therefore has potential as local source for the production of corn oil which is not readily available despite its economic importance. The use of hominy feed for the production of corn oil is a way of obtaining great value from the feed whose traditional uses are increasingly threatened by substitutes. It is against this background that the present work will aim at production and refining of corn oil from hominy feed.

MATERIALS AND METHODS

Materials

The equipment and reagents used in this work are presented below

Procedure

The stages involved in the extraction and refining of corn oil from the by-product of dehulled/degermed corn (sample) include

Pre-treatment

- i) Extraction
- ii) Purification

Pre-treatment of sample

After collection of the sample, the first step was to carry out size reduction. This was done by arranging the sieves in descending order of pore aperture. A pan was then placed under the last sieve to collect residue. The sample was then poured on the topmost sieve and the sieves shaken to enable separation occur. The fractions of the sample retained on the various sieves were collected, dried in an oven set at temperature of 50°C for 10 minutes and placed in different labeled containers.

Extraction of oil using soxhlet apparatus

An empty and clean thimble was weighed and its weight recorded. 12g of the dried unsieved sample was weighed and put into the empty thimble. The thimble containing the sample was then weighed again and the value obtained recorded. The sample in the thimble was subsequently covered with cotton wool and put in a soxhlet apparatus. 150ml of n-Hexane was measured and poured into a round-bottomed flask. The soxhlet apparatus was mounted on this flask and fixed under a condenser, which was already clamped to a retort stand. In addition, the condenser was already connected to 2 pipes, one pipe connected to a water supply (tap) and the other removes water from the condenser. The electro thermal heating mantle was switched on and temperature set at 40°C with the time this was done being noted. Also, the tap was put on.

After three hours, the heating mantle was switched off. The soxhlet apparatus was disconnected from the condenser and the thimble removed and dried in an oven for 5 minutes at 100°C. Any solvent in the soxhlet apparatus was poured back into the flask. The set-up was reassembled as earlier described and the heating mantle switched on with a set temperature of 40°C. The pure solvent began to evaporate and condense back into the soxhlet apparatus. The solvent was collected when the liquid level reached about three-quarters of the attached inverted u-tube to prevent reflux. The heating mantle was switched off and the round bottomed flask allowed to cool when no appreciable quantity of solvent could be gotten anymore from the flask. Cold oil was then poured into a glass container and the round bottomed flask rinsed with small quantity of solvent. The cotton wool covering the sample was removed carefully and the thimble weighed when cooled after removal from oven. The weight was recorded.

The residue was removed from the thimble and the procedure repeated for the various separate fractions. Also, extraction time was increased to six hours and the whole procedure repeated. On completion of extraction, the oil was heated for 20 minutes at 50°C to enable residual solvent escape.

Refining processes

According to [13], during the refining of vegetable oils almost all the compounds that negatively affect the quality of the product, in terms of turbidity,



colour and oxidation are eliminated, including phospholipids, waxes and free fatty acids.

Degumming

According to [6] degumming is the first step in the refining process. It removes phospholipids and mucilaginous gums: the presence of substantial amounts of phospholipids can lead to dark-coloured oils, and they can also serve as precursors of off-flavours. Hence, the removal of nearly all the phospholipids is very important for the finished oil quality.

In this work, distilled water was heated to 100°C and left to boil for several minutes. The crude corn oil was poured into a beaker and equal volume of hot distilled water added and stirred vigorously to remove the gums. The mixture was allowed to settle for 5 minutes, the oil - water mixture separated into layers with the oil layer on top. The oil was decanted and the process repeated again.

Alkali refining

The degummed corn oil was poured into an empty clean beaker and put into an oven set at 80°C for 30 minutes. 0.5 ml of 40% NaOH was then added to it after removal from the oven. The oil was then stirred and left for 20 minutes. The soap formed settled at the bottom of the beaker. The oil was decanted and washed with distilled water thrice to remove residual soap from the oil.

Decolourization

2g of unactivated clay was weighed and poured into the oil. The oil was then stirred and placed in an oven set at 100°C. The oil was kept in the oven for 30 minutes and stirred gently every 10 minutes. It was then filtered hot using a filter paper.

Analytical tests

A number of analytical testes were carried out on both the crude and refined corn oil in order to determine some of their physical and chemical properties and compare these values with standard values.

Refractive index determination

The refractive index was determined using a refractometer. This was done by placing a few drops of the oil sample on the face of the refractometer. The refractive index was read after a few minutes

Specific Gravity determination

An empty beaker was weighed. A specific volume of distilled water was added to the beaker and weighed. The beaker was emptied and dried. An equal quantity of oil was put in the beaker and weighed.

The specific gravity is calculated as shown below
 Specific gravity =
 Weight of oil sample/ weight of equal volume of water

Iodine value determination

Procedure

The method specified by International Standard Organisation (ISO) 3961 1989 was employed [11]: 0.4g of crude corn oil sample was weighed into a conical flask, 20ml of carbon tetrachloride and 25ml of DAM reagent were added to the flask. A stopper was fixed and the content of the flask was vigorously swirled. The flask was then placed in the dark for 1 hour 30 minutes. At the end of the time, 20ml of potassium iodide solution and 150ml of water were added. The contents of the flask were titrated with 0.1mol sodium thiosulphate solution until the yellow colour due to iodine has almost disappeared. Few drops of starch solution were then added and titration continued until the blue colour disappeared after vigorous shaking. The same procedure was repeated for the refined corn oil and blank test.

Two determinations were carried out on each sample.

The iodine value is given by the expression

$$\text{Iodine value (I.V)} = \frac{12.69 \times C \times (V_1 - V_2)}{M}$$

Saponification value determination

Procedure

The method specified by International Standard Organisation (ISO) 3657, 1988 was employed [11]: 0.2g of the crude corn oil was weighed into a conical flask; 25ml of ethanolic potassium hydroxide was then added with the aid of a pipette. The flask was attached to a reflux condenser and placed on an electric heater. The contents of the flask were allowed to boil gently for 60 minutes with shaking from time to time. 1ml of phenolphthalein indicator was then added to the flask and the content of the flask titrated with 0.5mol hydrochloric acid until the pink colour of the indicator just disappeared. The procedure was repeated for the refined corn oil sample and for blank test. Two determinations were carried out on each test sample

The saponification value is given by.

$$\text{Saponification value} = \frac{56.1 \times c \times (v_o - v_1)}{M}$$

Peroxide value determination

Procedure

The method specified by international standard organization (ISO) 3960 1975 was employed [11]: The experiment was carried out in diffused daylight. 2g of sample was weighed into a 500ml conical flask, 10ml of chloroform was added to dissolve the sample quickly by stirring, then 15ml of acetic acid was added and 1ml of freshly prepared saturated potassium iodide solution was added. The flask was then closed immediately, stirred for 1 minute and kept for exactly 5 minutes away from light at



room temperature. 75ml of water was added as indicator. The liberated iodine was then titrated against 0.01n sodium thiosulphate solution. The same procedure was carried out for other samples and the blank test was carried out by the same procedure by omitting test sample. Two determinations were carried out on each sample.

Calculations

The peroxide value expressed in milliequivalent of active oxygen per kilogram of sample is equal to

$$\frac{(V_1 - V_0) \times T \times 1000}{M}$$

Determination of unsaponifiable matter

The method specified by International Standard (ISO 3596 - 1, 1988) was employed [11]: This method uses diethyl ether extraction 2g of sample was weighed into a conical flask, 50ml of 1M solution of ethanolic potassium hydroxide was added with a few anti-bumping granules. The flask was attached to reflux condenser and the content was allowed to boil for 1 hour. 100ml of water was added through the top of the condenser with continuous swirling of the flask. The flask was allowed to cool and the contents now transferred into 500ml separating funnel. The conical flask was rinsed by using 100ml of diethyl ether. Stopper was inserted into the separating funnel and then inverted after vigorous shaking. The content was allowed to stand until there was complete separation of the two phases. The lower layer was then run off completely as possible into a second separating funnel. The ethanolic soap solution was extracted twice more each time in the same way with 25ml of diethyl ether and the contents of the extract was collected in separating funnel containing 40ml of distilled water. The separating funnel containing the combined extract and 40ml of water was rotated gently. The layers were allowed to separate completely and the lower aqueous layer was drawn off. The ethereal solution was washed successively with 40ml of potassium hydroxide solution and 40ml of water. The washing continued until the solution no longer gave a pink colour on addition of a drop of the phenolphthalein solution.

The ethereal solution was transferred through the top of separating funnel into 250ml conical flask previously dried and weighed. The solvent was added and the content completely evaporated in a gentle current of air. The residue was dried in an oven maintained at 103°C for 15 minutes. This was allowed to cool in a desiccator and then weighed to the nearest 0.1mg. The same procedure was used for the other samples and the blank.

Calculations

The unsaponifiable matter content expressed as a percentage by mass of the sample is

$$= \frac{100(M_1 - M_2 - M_3)}{M_0}$$

RESULTS AND DISCUSSIONS

From the results obtained during extraction as presented in Tables 3 and 4, it is clear that the yield of oil increased though not linearly, as the particle size of the sample decreased. However, on comparing yields for the same particle size but different extraction times, the variance noted is not wide. This is despite the fact that the latter extraction time is double that of the former. In addition, the sample contains maize germ, known to have an oil content of about fifty percent [5]. However, the oil yields gotten from the experiment do not necessarily reflect this. One of the reasons for this apparently low yield can be traced to the composition of the sample, a byproduct of dehulling and degermination of maize. The operation of this process is not perfect. This means there is the possibility of by-product appearing in main product stream and vice versa. In addition, apart from the germ, other components of the feed such as the hull contain relatively insignificant amount of oil. This means the oil producing portion of the separate fraction of the sample is quite small. Perhaps, this could also be the reason why the difference in yields at different extraction times for particular fractions is narrow. However, the yield for the unsieved sample presents a deviation from this norm. At extraction time of six hours, the oil yield is almost double that of three hours. This is an indication that the unsieved sample contains some amount of germ which was not retained in any of the separated fractions. This claim is further boosted by the fact that the oil yield increase as the particle size decreased. [11] Got average oil yields of 14.11 and 20.65% for extraction times of three and six hours respectively.

Also, there is appreciable difference in the oil yields gotten at different extraction times for the same fraction of samples. The average oil yields observed in this study are 14.28 and 14.50% for extraction times of three and six hours respectively.

Obviously, [11] got a better average oil yield from his sample especially on comparison with his result for extraction time of six hours. But he used maize germ separated from the kernel. The dried germ was crushed and separated into several particle sizes. It is highly probable he will get a better oil yield as the germ has a significant oil content and even if not completely pure as a result of presence of impurities, appreciable oil yields, would still be gotten due to the effect of the centrifuge in obtaining the desired germ.

During degumming, which is the first stage in the refining process, the hot distilled water removed the 'gums; as expected but this removal was not complete. The next stage, Alkali refining completely removed the gums among other undesirables and subsequent washing of the oil with distilled water removed residual soap. Decolouration with clay achieved total removal of soap and resulted in clear golden yellow oil.

For the physical tests performed on both the crude and refined corn oil, the difference in the refractive index and specific gravity was insignificant. However, the chemical tests reveal the importance of refining vegetable



oils. For instance, the peroxide value, a measure of the degree of rancidity which has occurred in oil though quite low, dropped even lower after refining. The saponification value followed the same trend. It is interesting to note that the iodine values for both the crude and refined corn oil were close and within the range of the standard. The implication of this is that had the oils been adulterated, the iodine value would not have fallen in this range as it is specific for particular oil. The iodine value is also a measure of the degree of the unsaturation of oil [16]. The high iodine values of the crude and refined corn oil agree with the fact that corn oil is polyunsaturated oil.

CONCLUSIONS

The yields of oil obtained by using of hexane as solvent are quite encouraging but there is much room for improvement. Average oil yield was 14.28% and 14.50% for extraction time of three and six hours respectively.

Also the properties of the refined corn oil especially the chemical ones highlighted the importance of the refining process in the production of refined corn oil and some of their determined values include peroxide value 0.25meq/ kg, iodine value, 119.92gIodine/100g of sample. In addition, these values confirmed the high quality of the oil.

On a general note, the usage of the sample as raw material in place of maize for corn oil production holds promise but further work needs to be done in improving oil yield of the sample.

RECOMMENDATIONS

- Solvents like petroleum ether, ethanol other than Hexane, should be used as solvent in the extraction;
- Research should be carried out with respect to enriching the germ content of the sample; and
- After collection of the sample, it should be processed as soon as possible to minimize the deteriorating effects of rancidity.

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**Appendix- A**

Tables 1 and 2 present the list of equipment and instruments and the list of chemicals used in the experiments.

Table-1. List of equipment and instruments.

Equipment/instrument	Manufacturer
Sieves (2.00mm, 1.00mm, 850µm, 600µm, 250µm)	Endecotts Ltd, England
Weighing balance	Mettler, Germany
Measuring cylinder (100ml)	Assistant, Germany
Beakers (100ml)	Pyrex, England
Electro thermal heating mantle	Unknown
Soxhlet apparatus	Quick fit, England
Thimble	Unknown
Funnel	Pyrex, England
Filter paper	Unknown
Stirring rod	Pyrex, England
Digital oven	Gallenkamp, Germany

Table-2. List of chemicals.

Chemicals	Manufacturers/Source
n-Hexane	May and baker limited, England
Distilled Water	Fisheries Lab., FUT Minna, Niger State, Nigeria
Sodium hydroxide	May and baker limited, England
Dam's reagent	Biochemistry lab, FUT, Minna.
Phenolphthalein	May and baker limited, England
Potassium iodide	May and baker limited, England
Starch solution	Biochemistry lab, F.U.T Minna
Ethanol	May and baker limited, England
Potassium hydroxide	May and baker limited, England
Hydrochloric acid	May and baker limited, England

Appendix- B

Tables 3 to 7 present experimental results, properties, and comparison of experimental data with standard values.

Table-3. Results of extraction for 3 hours.

Particles size	Weight of oil (g)	% yield
Unsieved sample	1.27	10.58
1.00mm	1.40	11.67
850µm	1.59	13.25
600µm	1.87	15.58
250µm	1.94	16.62

Table-4. Results of extraction for 6 hours.

Particles size	Weight of oil (g)	% yield
Unsieved sample	2.16	18.00
1.00mm	1.42	11.83
850µm	1.65	13.75
600µm	1.86	15.75
250µm	2.00	16.67

Solvent used: n-Hexane

**Table-5.** Properties of crude corn oil.

Property	Value
Specific gravity	0.9132
Refractive index	1.461
Saponification value (mgKOH/g)	224.40
Iodine value (g iodine/100g of sample)	116.27
Peroxide value (meq/kg)	0.75
Unsaponifiable matter content (%)	0.83

Table-6. Properties of refined corn oil.

Property	Value
Specific gravity	0.92
Refractive index	1.470
Saponification value (mgKOH/g)	195.30
Iodine value (g iodine/100g of sample)	119.92
Peroxide value (meq/kg)	0.25
Unsaponifiable matter content (%)	0.55

Table-7. Comparison of the characteristic values of crude and refined corn oil with standard values.

Property	Standard value	Refined corn oil value	Crude corn oil value
Specific gravity	0.922-0.928	0.920	0.9132
Refractive index	1.470-1.474	1.470	1.461
Saponification value (mgKOH/g)	189-195	195.30	224.40
Iodine value (g iodine/100g of sample)	107-135	119.92	116.27
Peroxide value (meq/kg)	≤ 10	0.25	0.75
Unsaponifiable matter content (%)	≤ 1.5	0.55	0.83