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Physical and Chemical Properties Analysis of Flax Seed Oil (FSO) for Industrial Applications

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ABSTRACT

***Abstract*—**A study on the physicochemical properties of Flax seed oil for industrial applications were carried out. Physicochemical properties of Flax seed oil (39% lipids) showed high content of iodine value (141mg/100g) and saponification value (198.45 mg/100g). The present study shows that, Flax seed oil is rich in linolenic and oleic acids. The Flax seed oil with the highest amount of polyunsaturated fatty acids (linolenic acid) can find an application in surface coating industries and biolubricant base oil applications, whereas the high amount of monounsaturated fatty acid can find an application as a biodiesel feed stock. Flax seed oil contains major TAG of monounsaturated OLL, POL,PPS, PLnLn , POLn, LnLnLn ,OLnLn , OLLn , LLLn and POLn. Flax seed oil can be classified as unsaturated oil with an unsaturated fat level of 89.5%. Hence the Flax seed oil has great potential for industrial applications such as in paint and surface coatings, production of biodiesel and biolubricant. Therefore, it is crucial to have more research on Flax seed oil in the future to explore its potential as a future industrial oilseed crop.

Keywords- Physical, Chemical, Industrial applications, Flax seed oil

I- INTRODUCTION

Flax seed, which is also called linseed, is important seed oil in the world. It is mainly grown in Canada, Argentina, America, China and India. Flax seed contains about 40%oil, 30% dietary fiber, 20% protein, 4% ash, and 6% moisture. The nutritional components of Flax seed are oil, protein, lignans, soluble fiber, minerals and vitamin, etc.

Flax (*Linum usitatissimum* L.) is a multi-purpose crop. Its production goes back to ancient history. Its remnants were found in Stone Age dwellings in Switzerland and ancient Egyptians made fine from flax fiber. Two types of flax are grown, seed Flax for the oil in its seed and fiber Flax for the fiber in its stem. It is mainly grown in Canada, Argentina, America, China and India (Wang, et al. 2007). The plants range in height from 30 to 100 cm and have narrow leaves flowers that are in different shades of blue. Its seeds containing about 36 to 40% of oil have long been used in human and animal diets and in industry as a source of oil and as the basic component or additive of various paints or polymers (El-Beltagi, 2007).

There has been considerable research on the physicochemical properties of Flax seed oil. The composition of Flax seed oil from Canada consists of mainly fatty acids such as palmitic acid (13%), stearic acid (2.53%), oleic acid (48.8%) and linoleic acid (34.6%) (Ooi, Jumat, 2006) . Flax seed oil contains a high percentage of unsaturated fatty acid, which is about 89.5- 90%. This makes the oils suitable for biodiesel production. However, the chemical compositions of the oil vary according to the climate and locality . Nowadays, the Flax plantation is receiving considerable attention in many parts of the world due to the advantages, including higher yield than other plant oils, such as soya, rapeseed, etc., easy to cultivate and reclamation of waste land (Lerma and Garcia. 2010) .The objective of this study is to evaluate the physicochemical characteristics of the seed oil extracted from the Flax seeds collected from Libya.

II. EXPERIMENTAL AND METHODOLOGY

A. The seed sample

The Flax seed (*Linum usitatissimum*) is obtained from a farm in the region of Misrata, east of Tripoli and the Flax is grown in winter and kept below-20 C^0 until being used.

B. Oil Extraction

Flax seed oil extraction was determined in accordance with [Abdullah, B.M. & Salimon, J. 2009]. About 600g milled Flax seeds were weighed and placed in a thimble. The thimble was then placed in the Soxhlet chamber, which was suspended above a boiling flask containing 2500 mL hexane. The hexane was heated under 60°C for 8 hours. The chamber containing the milled Flax seeds was slowly filled with warm hexane, until the warm hexane exceeded a certain temperature level when it overflowed and spilled over into the boiling flask. This cycle was repeated many times. After extraction for 8 hours, the hexane was evaporated by a rotary evaporator in water bath at 50°C for 60min.

C. Colour

Colorimetric measurements, according to [Siew, W.L., Tang, T.S. and Tan, Y.A. 1995], were carried out using a manual colorimeter Orbeco-Hellige, equipped with glass colour standards and a glass cuvette of 3.3-cm optical Path.

D . Lipid Content

The weight of the oil extracted from 600g of seed powder was determined to calculate the lipid content. The result was expressed as the percentage of lipids in the composition of the dry seed powder.

E. Acidity

Free fatty acid (FFA %) and acid value (as oleic) were determined according to [Abdullah, B.M. & Salimon, J. 2009]. Approximately 50mL of isopropanol was placed into the flask, and about 0.5mL phenolphthalein was added and was neutralized by addition of sodium hydroxide (NaOH, 0.02 N) until a permanent pink colour was obtained. The neutralized isopropanol was added to the 5g of Flax seed oil, which was placed into an Erlenmeyer flask, and about 0.5mL of phenolphthalein was added. After shaking the mixture gently, the mixture was neutralized by the addition of (NaOH, 0.02 N) until the first permanent pink colour was obtained.

E. Iodine Value

The iodine value was determined according to [Abdullah, B.M. and Salimon, J
. 2009]. About 0.3g of Flax seed oil was placed in a 500mL flask. Then 15mL of carbon tetrachloride (CCl_4) was added to dissolve the oil, and 25mL of the wijs solution was added into the flask and the stopper was inserted. After shaking the mixture gently, the flask was placed in the dark for 1 hour. After standing for 1 hour, 20mL of potassium iodide (KI, 10% v/v) solution and 150mL of water were added, the mixture was titrated with the sodium thiosulphate ($Na_2S_2O_3$, 0.1 N) solution until the yellow colour due to the iodine had almost disappeared, 1mL of the (starch, 1% v/v) indicator solution was added, and the titration was continued until the blue colour just disappeared after very vigorous shaking. The blank test was carried out under the same conditions.

G. Saponification Value

The saponification value (SV) was determined according to [Abdullah, B.M. and Salimon, J. 2009]. About 2g of Flax seed oil was placed into conical flask, and 25mL ethanolic potassium hydroxide (KOH, 0.5 N) was added with some boiling stones. The boiling flask was connected to the condenser and the mixture was boiled gently for 1 hour. After the boiling, the mixture was cooled and 1ml of phenolphthalein, (1% v/v) was added, the mixture was titrated with hydrochloride acid (HCl, 0.5N) until the pink colour of the indicator just disappeared. The blank test was carried out under the same conditions.

H. Unsaponifiable Matter

Unsaponifiable matter was determined according to [Abdullah, B.M. & Salimon, J. 2009]. About 10g of Flax seed oil was placed into a round bottomed flask and 30mL ethanol and 5mL of aqueous KOH solution were added with some boiling stones into the round bottomed flask. The round-bottomed flask

was connected to a condenser, and the mixture was boiled gently for 1 hour. After the boiling, the heating was stopped and the reaction mixture was transferred into the separating funnel. The flask was rinsed with 10mL ethanol followed by 20mL warm distilled water and then 20mL cold distilled water, and all the washings were transferred into the separating funnel. The contents of the separating funnel were left to cool at room temperature, after that 50mL of hexane was added into the separating funnel. After shaking the mixture vigorously for 1min, the mixture was left a few minutes to get two phases. The soap solution phase was converted completely into the second separation funnel, and 50mL of hexane was added into the separating funnel. After shaking the mixture vigorously for 1 minute, the mixture was left a few minutes to get two phases. The extractions using 50mL of hexane were repeated five times.

The combined extracts in the separating funnel were washed three times with 25mL of 10% (v/v) ethanol, after shaking the separating funnel vigorously; the ethanol layer was drawn off after each wash. The hexane was evaporated to dryness under the vacuum using a rotary evaporator, the drying was completed in a vacuum oven at 75-80°C, and was cooled in a desiccator and was weighed (Wr). The residue was dissolved in 50mL 95% ethanol, and was titrated with 0.02 N NaOH solution using phenolphthalein indicators to a faint pink colour.

I .Viscosity

The viscosity of the Flax seed oil was measured using chemistry Labs. Inc. Viscometer [Siew, W.L., Tang, T.S. and Tan, Y.A. 1995]. The spindle size $S0_5$ was used at 100 rpm for 1min in room temperature.

J. Gas Chromatography Method

Gas chromatography method (GC) analysis was performed using Shimadzu Gas chromatography (GC) equipped with a flame ionisation detector and capillary column (30m × 0.25mm × 0.25mm film). The parameters of GC have been carried out according to [Abdullah, B.M. and Salimon, J. 2009]. The fatty acids were determined using their fatty acid methyl esters and were injected into gas-chromatography for analysis. The identification of the peaks was carried out by retention times by means of comparing them with genuine standards analyzed under the same conditions.

***J.* High Performance Liquid Chromatography Method**

High performance liquid chromatography (HPLC) analysis was performed using a Waters model 1515 equipped with refractive index detector and Spherisorb C18 column (250mm × 4.8mm × 3mm), which was used for analysis of the TAG. The parameters of HPLC were carried out according to [Abdullah,

B.M. and Salimon, J. 2009]. Triacylglycerol of Flax seed oil and FFA were determined by using HPLC. Flax seed oil and the FFA were dissolved in 10mL of the mixture acetone: acetonitrile before 20mL of the sample being into HPLC.

III. RESULTS AND DISCUSSION

In determining the colour of the Flax seed oil, Cielab coordinates were used. In this coordinate system the **R*** value is a measure of redness. The **Y*** value is a measure of yellowness Flax seed oil colour comes from the presence of highly coloured material and carotene extracted from the seed, however, most of the colour is due to a low residual level of gossypol, which is a yellow pigment, and its derivatives [O'Brien, R.D.2009]. Some of the pigments can be removed by adsorption bleaching; gossypol can only be removed by alkalire fining. The higher **Y*** is a measure of 5, which makes Flax seed oil darker in colour. At room temperature, the colour of the Libyan Flax seed oil appeared as golden yellow oil **(Table I).**

Table I … shows the physicochemical properties of the Flax seed oil compared to Canadian Flax seed oil . The Flax seed oil in this study contained a relatively high percentage of total lipid content 39**%** compared to the canadian seed oil, which was 37**%** . The high lipid content of Flax seed indicates that Flax seeds are suitable as non-edible plant oil feed stock in oleochemical industrie (biolubricants, biodiesel, fatty acids, soap, fatty nitrogenous derivatives, surfactants and detergents). The FFA% and acid value of flax seed oil are 4.37(mg KOH/g), 2.2**%** respectively. Otherwise, a high free fatty acid content would be nutritionally desirable by its enhancement of the availability of fatty acids (especially the unsaturated ones), which normally esterifies to the glycerol moiety of the triacylglycerol [Ukhun, M.E. and Uwatse, G.M. 1988]. The iodine value is a measure of the unsaturation of fats and oils. A higher iodine value indicates higher unsaturation of fats and oils [Ukhun, M.E. and Uwatse, G.M. 1988]. The iodine value of Flax seed oil in this study is 141 mg/g , which is lower than Canadian flax seed oil 170 mg/g . The present saponification value of the Flax seed oil 198 mg/g is higher than the Canadian Flax seed oil 187 mg/g.The average molecula weights of TAG of Flax seed oil is 848 g/mol .The unsaponifiable matter is important in determining the total quantity of substances present in oil or fat, and after saponification with an alkaline hydroxide, it is insoluble in water but soluble in the solvent used. The Flax seed oil was saponified by an ethanolic alkaline hydroxide solution, followed by dilution. The unsaponifiable matter was extracted with hexane. The total quantity of unsaponifiable matter of Flax seed oil is 1.8% as shown in Table I.

TABLE I: PHYSICOCHEMICAL PROPERTIES OF FLAX SEED OIL

Properties	Libya	Canada
Color	Y* = 5	/
Oil content (%)	39 %	37 %
Free fatty acid as (oleic acid) (%)	2.2 %	0.1 – 2 %
Acid value (mg KOH/g)	4.37(mg KOH/g)	3.3 (mg KOH/g)
Iodine value (wijs)	141mg/g	170 mg/g
Saponification value(mg/g)	198 mg/g	187 mg/g
Average molecular weight	848 g/mol	875 g/mol
Unsaponification matter (%)	1.8 %	2.0 %
Viscosity	104cp	/

Source: Akintayo. 2004

At room temperature, kinematic viscosity of the Flax seed oil was detected at 104cp. This is a comparable value with that reported elsewhere (Akintayo,2004). The viscosities of Flax seed oil must be increase for biodiesel application since the kinematic viscosity of biodiesel is very high compared to plant oils. The determination of fatty acid composition of the flax seed oil reveals important characteristics, as shown in **Table II**. Three major long chain fatty acids were detected in the Flax seed oil, which are oleic 17.8%, linolenic 55.7%, and linoleic 15.9 % acids. Other fatty acid compositions were less than 10% and comprised stearic 4.4% and Palmitc 5.3% Beheric 0.2% acids.

TABLE II FATTY ACIDS COMPOSITION OF *FLAX* SEED OIL

Fatty acid composition	Libya (%)	Canada (%)
Palmitic	5.3	5.3
Stearic	4.4	3.3
Beheric	0.2	/
Lignoceric	0.1	/
Arachidic	0.1	/
Oleic	17.8	17.9
Linoleic	15.9	14.7
Linolenic	55.7	58.7
Σ Saturated Fatty acid	10.1	8.6
Σ Unsaturated Fatty acid	89.5	91.3

Source: Salimon, et al.2011

In general, the Flax seed oil in this study contained unsaturated fatty acids 89.5% less than Canadian Flax seed oil 91.3% (Salimon, J, Abdullah, B.M. and Salih, N. 2011) . Medium fatty acids such as capric, lauric and myristic were not detected. As a comparison, the Flax seed oil in this study and Canadian Flax seed oil contain less palmitic. Plant oils that are rich in polyunsaturated fatty acids such as linoleic acid, include soybean 53.2% and sunflower 66.2%, which tend to give methyl ester fuels with oxidation stability. Plant oils with high degree unsaturation tend to have a high freezing point. Due to its industrial potential, it is crucial to determine the triacylglycerol (TAG) profile for the Flax seed oil. The results from the reversed phase HPLC show that the oil is composed of at less ten important TAGs **(Fig. 1)** in which the mechanism of separating the TAGs involves the chain length and degree of unsaturation of the fatty acids [Gutierrez, V.R. and Barron, L.J.R. 1995.].

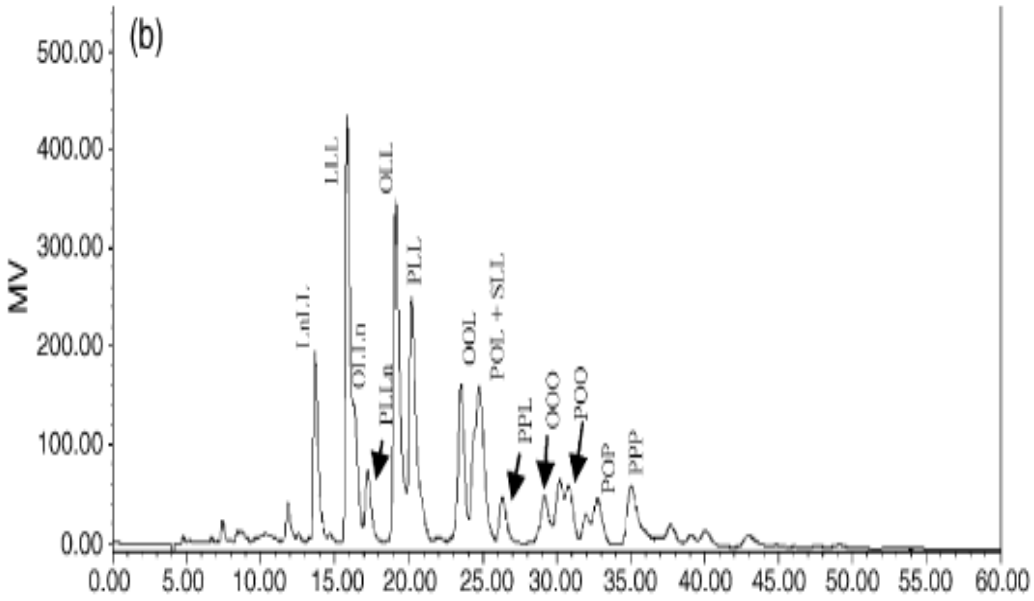

Fig. 1 .HPLC chromatogram of TAGs composition of Flax seed oil

The **TAGs** composition in Flax seed oil was identified according to the equivalent carbon number **(ECN)** compared with standard (**Table III).**

TABLE III TAG COMPOSITION OF FLAX SEED OIL

TAGs	ECNs	Libya(%)	Canada (%)
PLnLn	42	7.9	7.9
PLLn	44	6.7	6.7
LnLnLn	36	20- 21.4	20.9
OLnLn	44	8- 8.8	8.4
POLn	46	4.3	4.0
POL	48	1.9	1.5
OLL	38	17.4	/
LLLn	40	0.6	/
PPS	50	0.5	/
POL	46	0.17	/
OOL	38	0.1	3.4

Source: Schorno et al.2003& Ku, et al.2007

The Flax seed oil is rich in triacylglycerol, containing (98.8%), depending on the FFA% [Ku, C.S. & Mun, S.P. 2007]. The TAG exists in the solid or liquid form depending on the nature of the constituent fatty acids. Most plant triacylglycerols have low melting points and are liquid at room temperature. They contain a large proportion of unsaturated fatty acids, such as oleic, linoleic, and linolenic.

IV. **CONCLUSION**

The present study shows that , Flax seed oil is rich in oleic, linoleic and linolenic acids. The Flax seed oil with the highest amount of polyunsaturated fatty acids (linolenic acid) can find an application in surface coating industries and biolubricant base oil applications, whereas the high amount of monounsaturated fatty acid can find an application as a biodiesel feed stock.

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الفهـــــــــــرس

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- يرفق بالبحث تزكية لغوية وفق أنموذج معد .
- تعدل البحوث المقبولة وتصحح وفق ما يراه المحكمون .
- التزام الباحث بالضوابط التي وضعتها المجلة من عدد الصفحات ، ونوع الخط ورقمه ، والفترات الزمنية الممنوحة للتعديل ، وما يستجد من ضوابط تضعها المجلة مستقبلا .

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