



## Physicochemical characterization, fatty acid in Corn Seed Oil using GC-FID method

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### Abstract

Physical properties like physical state at 25<sup>o</sup> C/ 1Atm, colour, odour, solubility, miscibility and physicochemical properties like density /specific gravity, viscosity, flash point, boiling point, saponification value, unsaponifiable value, iodine value, colour (Lovibond tintometer), peroxide value and refractive Index Corn oil were studied to evaluate the compositional quality of oils and also to investigate the effect on the use of same oil for repeated frying as it ultimately changes the nutritional, physicochemical, and sensory properties of the oil. The physicochemical analysis showed color values were 3.0 red maximum, density (g/mL)/specific gravity were 0.918 at 25<sup>o</sup>C, viscosity (millipoise) were 114.00, flash point were 305, boiling point were 148<sup>o</sup>C, saponification value (mg) were 153.6, unsaponifiable value ( gm ) were 2.2, iodine value (g) were 15.77, peroxide value (meq/kg) were 0.165, refractive index (40<sup>o</sup> C) were 1.466. The analyses of fatty acid profile showed higher linoleic acid, oleic acid and palmitic acid contents and saturated fatty acid were 15.282, unsaturated fatty acid were 84.717 present in corn oil.

**Keywords:** Corn seed, Corn seed oil, Fatty Acid Profile, Quality characteristics

### Introduction

Nuts are a good source of oil containing higher unsaturated fatty acids (PUFA and MUFA) to saturated fatty acids ratio the higher body weight is associated with higher percentage of fat in the diet [18, 19]. Fats and oils consist of a wide group of compounds that are soluble in organic solvents and insoluble in water. They have lower densities than water and at normal room temperature range inconsistently from liquids to solids depending on their structure and composition. The words oils, fats, and lipids are all used to refer to fat; oils are usually used to refer to fats that are liquids at room temperature, while fats are usually used to refer to that are solid at normal temperature [20, 21].

In quality control of oils, several parameters such as saponification value (average molecular weight), iodine value (degree of unsaturation), unsaponifiable value and peroxide value as well as the fatty acid profile are of interest as they determine the quality and hence the economic value of the product. Currently, the majority of these parameters are determined by using classical wet chemical methods [5]. Edible oils have been analyzed for different metals using atomic absorption spectrometer (FAAS and GFAAS), inductively coupled plasma optical emission spectrometer (ICP-OES), and inductively coupled plasma mass spectrometer (ICP-MS). Atomic absorption spectroscopy (flame, graphite furnace, hydride generation, and cold vapour) is still the most widely used technique [21-24].

Maize (*Zea mays* L.) has been a crop extended and an important food for the American societies since pre-hispanic times [1].

Maize (*Z. mays* L.) is the third most important food crop in the world and a major source of energy, protein, and other nutrients for both human and livestock. Maize contains 7-13 g/100 g proteins (d.m.). However, the quality of maize proteins is poor, because they are deficient in the essential amino acids lysine and tryptophan [2, 3].

Corn is successful cereal grass useful as food, feed and in industries. Corn is also source of corn oil which is tasteless odourless and pale yellow oil derived from the corn kernel. Normal corn has usually oil ranging about 3-4 per cent oil, especially in the embryo. Corn with oil content more than six per cent is called high oil corn (HOC) [4, 5, 6, 7, 8, and 9].

### Materials and Methods

#### Material and chemicals

Methanol, Hydrochloric acid, sodium hydroxide, potassium hydroxide, benzene, sodium thiosulphate, petroleum ether, potassium iodide (KI), hexane, acetic acid and chloroform were of A.R Grade and purchased from Merck Darmstadt Germany. Corn oils were extracted from their dried, dehulled and powdered seeds purchased from the local market in alwar, rajasthan (India).

#### Oil extraction

Corn is cultivated in the Alwar (Rajasthan). Corn oil sample was obtained from corn seeds using the cold pressed method. Oil was then stored at 4.0±2<sup>o</sup>C. Oil extraction was conducted using a Soxhlet apparatus for approximately 6 hrs with hexane as solvent, with a solid to solvent ratio of 1/7 m/v. After the extraction process, the flask contents were filtered, and the liquid fraction containing the lipid extract and solvent was poured into a 500 mL flask of a rotary film evaporator to remove the solvent. The obtained oil was collected, evaporated under nitrogen, weighed, and stored in sealed amber glass vials at -20<sup>o</sup>C until analysis [10]. Determination of physical properties such as physical state at 25<sup>o</sup> C/ 1Atm, colour, odour, solubility and miscibility was determined by the manual of methods of analysis of food, fssai, 2015 [13].

Determination of physicochemical properties such as colour by using lovibond tintometer, flash point, unsaponifiable value was

determined by hand book of phytochemical, pharmacognostical, water and edible oil analysis (A lab manual) [17].

### Density measurement

Densities of oil samples before and after frying were measured by an R.D bottle with a capacity of 10 mL [17].

### Boiling point measurement

The boiling point of oil samples were measured by a thermometer. The boiling point depends upon the degree of unsaturation of fatty acids.

### Viscosity measurement

The viscosity of oil samples before and after frying was measured by Viscometer techniconominal constant 0.05 Cs/c. The flow time of corn oil sample were recorded with a stop watch least count  $\pm 0.01$  s [17].

### Frying process

Potatoes were peeled and cut into pieces (approx 18) and were fried in the frying oil at constant temperature at four frying times. Frying experiments were conducted in duplicate in each frying medium.

### Saponification value

At first, 2 g oil was taken in 250 ml round bottom flask and 25 ml, 0.5 N alcoholic potassium hydroxide solution added in same flask. The flask was fitted with a long air condenser and heated solution at reflux temperature about 30 minutes. Finally, the flask was cooled and added 1 ml of 1 percent phenolphthalein solution and titrate the excess of the alkali against standard 0.5 N HCl. At the same time and under similar conditions carry out a blank titration without fat (25 ml, 0.5 N same alcoholic KOH solution was taken in another round bottom flask and heated in a similar way and titrated, against 0.5 N acid) [14-17].

### Iodine value

5 g of oil was taken into 200 ml a glass stoppard bottle. 5 ml of CCl<sub>4</sub> was added to dissolve this oil after 25 ml of Wij's solution was added and to allow it at least 1 hour in a dark place. Then 5 ml of 10% potassium iodide solution and 50 ml water were added to each bottle and titrated against 0.1N Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> solution using starch solution as the indicator, near the end point of titration the color of solution becomes pale yellow. Blue color disappears which indicates the end point. At the same time and under similar conditions carry out a blank titration without oil [14-17].

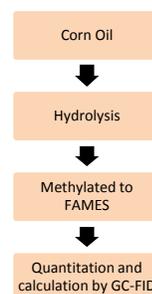
### Peroxide Value

The peroxid value is a measure of the concentration of substances that oxidize potassium iodide to iodine. 5 g weight of oil samples is dissolved in acetic acid then chloroform and saturated KI mixture are added to the sample and the amount of iodine liberated from KI by the oxidative action of peroxides present in the oil is determined by titration with 0.1 N sodium thiosulphate using starch solution as an indicator. Titration was also performed for blanks [14-17].

### Preparation of fatty acid methyl esters (FAMES):

For conversion of corn oil into FAME the following methods was adopted: around 0.2 gm corn oil was taken into 50 mL glass vial

then mix with 5 mL of 0.50 N methanolic NaOH, the mixture was heated for 4 mins at 60 C. The mixture was allowed to cool at room temperature, then 6 mL of 14% BF<sub>3</sub> solution was added [11] to the mixture and again heated for 3 mins at 60 C. The mixture was again cool at room temperature then added 10 mL , isooctane and shake it well, then keep it to settling down. After settling the mixture, the upper layer of the mixture was transferred to the tube containing sodium sulfate to remove the moisture. The extract was analyzed with GC-FID (Nuckon - 5700), the flow chart of the corn oil analysis method condition shown in Scheme 1 [12].



Scheme 1. Flow chart of the corn oil fatty acid analysis.

### Chromatography

Nuckon – 5700 gas chromatograph equipped with a flame-ionization detector (FID) and glass column, digital controls, separate independent proportional temperature controllers for oven, detector, Injector, Detector temperature 390 0 C in 10 C steps set by thumbwheel switches, oven temperature 399 0 C in 1 C step set by thumbwheel switches. Temperature programming: - microprocessor based programmer with rate 0.1 0 C – 29.9 0 C per minute with initial time hold up to 99 minute. Dual channel data station, 2 detectors can be operated simultaneously. Use Nitrogen gas cylinder -60 psi. Hydrogen gas cylinder -20 psi. Zero air gas cylinder -20 psi.

### Certified Reference Materials (CRM)

Sigma, Catalog no 46961, Lot no - LC 26752V, Exp. May 2020.

### Results

Corn oil show physical properties liquid foam, yellow colour, characteristic odour, slightly soluble in alcohol and miscible in ether, chloroform, petroleum ether, benzene, hexane and other chemical solvents (table -1).

Table 1: Physical properties of corn oil

| Property                                  | Characteristic/ Value                                           |
|-------------------------------------------|-----------------------------------------------------------------|
| Physical state at 25 <sup>0</sup> C/ 1Atm | Liquid                                                          |
| Colour                                    | Yellow                                                          |
| Odor                                      | Characteristic Corn                                             |
| Solubility                                | Slightly soluble in alcohol                                     |
| Miscibility                               | Miscible in ether, chloroform, petroleum ether, benzene, hexane |

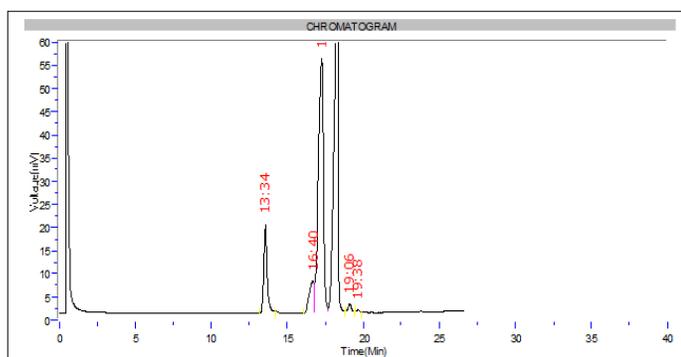
The quality of Corn oil was analyzed by evaluating physicochemical properties such as density /specific gravity, viscosity, flash point, boiling point, saponification value, unsaponifiable value, iodine value, colour (Lovibond tintometer),

peroxide value and refractive Index. Results are presented in Table 2.

**Table 2:** Physicochemical properties of corn oil

| Property                        | Characteristic/ Value |
|---------------------------------|-----------------------|
| Density (g/mL)/specific gravity | 0.918 at 25°C         |
| Viscosity (millipoise)          | 114.00                |
| Flash point                     | 305                   |
| Boiling Point (°C)              | 148.00                |
| Saponification Value (mg)       | 153.6                 |
| Unsaponifiable value in gm      | 2.2                   |
| Iodine Value (g)                | 15.77                 |
| Colour (Lovibond tintometer)    | 3.0 red max.          |
| Peroxide value (meq/kg)         | 0.165                 |
| Refractive Index (40° C)        | 1.466                 |

Corn seed samples collected from the local market alwar, rajasthan were analyzed on the composition of fatty acids using gas chromatographic FAMES method. The content of following saturated and unsaturated fatty acids was tested in the samples: palmitic acid, stearic acid, oleic acid, lenoleic acid, linolenic acid and arachidic acid. The fatty acid percent compositions of corn oil are shown in table 3 to 4 and figure 1 respectively.



**Fig 1:** Gas chromatogram of corn oil

**Table 3:** Presenting the different parameters of the fatty acid of the corn oil.

| Peak No. | RT (Min) | Area (mV-Sec) | Height (mVolt) | RF    | Amount (ML) | Amount% | Component Name |
|----------|----------|---------------|----------------|-------|-------------|---------|----------------|
| 1        | 13:34    | 317.555       | 19.153         | 1.000 | 317.555     | 10.386  | Palmitic       |
| 2        | 16:40    | 145.137       | 6.903          | 1.000 | 145.137     | 4.747   | Stearic        |
| 3        | 17:16    | 1,256.088     | 54.738         | 1.000 | 1,256.088   | 41.081  | Oleic          |
| 4        | 18:15    | 1,309.805     | 69.792         | 1.000 | 1,309.805   | 42.838  | Lenoleic       |
| 5        | 19:6     | 24.402        | 1.668          | 1.000 | 24.402      | 0.798   | Linolenic      |
| 6        | 19:39    | 4.568         | 0.365          | 1.000 | 4.568       | 0.149   | Arachidic      |

**Table 4:** The content of SFA, MUFA, PUFA (% w/w)

| Saturate d fatty acid | Monounsaturate d fatty acid | Polyunsaturate d fatty acid | unsaturate d fatty acid |
|-----------------------|-----------------------------|-----------------------------|-------------------------|
| 15.282                | 41.081                      | 43.636                      | 84.717                  |

Unsaturated fatty acids are classified as either monounsaturated fatty acids, because they have only one double bond (e.g., omega-7 and -9 fats), and polyunsaturated fatty acids, since they have more than one double bond in their backbone (e.g., omega-3 and -6 acids) [25, 26]. Saturated fatty acids are in solid state at room

temperature. The main sources include ghee, butter, tallow coconut, lard, palm, and palm kernel oil. Saturated fatty acids are also called nonessential fats (not to be confused as unimportant) [27-31].

## Conclusion

The observation results concluded that we should consider the physical, physicochemical properties and fatty acid profile for selecting good type corn oil.

## Competing Interests

Authors have declared that no competing interests exist.

## Acknowledgement

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