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Studies on Seed Oil Characteristics of Trygonella Foenum (Methi Seeds)

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Abstract Oil Characteristics of the seeds of *Trigonella foenum graecum* Linn were studied. After purification physical chemical characteristics of the oil were determined and these values were compared with those of the standard oils. Fatty acid compounds of the oil were estimated by GLC. The ash, protein, phosphorus, moisture, crude fibre, starch, nitrogen, manganese contents of the cake were also determined.

Keywords GLC, TLC, Customary constant

1. Introduction

Trigonella foenum graecum Lin. fenugreek is one of the 70 species of the genus Trigonella belonging to the family papilonacea. It is an annual herb, a native of the mediterranean region, Europe, Asia, South Africa and Australia. It is also found to grow in the sub tropical region of India and Bangladesh. It is called as methi, methishak, methuka in our country. An annual herb 1-2 feet high, with an erect, slightly branched, cylindrical hallow smooth or slightly pubescent stem, root tapering. Leaves alternate on rather long stalks, trifoliolate, stipules 1/4 inch long. flowers sessile, solitary in the axils of the leaves calyx long and narrow. The seeds are used as a condiment and for flavouring food preaparation. they may be eaten raw or cooked in Egypt, ground seeds are mixed with wheat flour for making bread. The plant possesses insect-repellent properties. In Punjab Agriculturists mix the dried plants with stored grains to protect them from ravages of insects especially the rainy season. Present work mainly deals to carry out complete study of the physical and chemical characteristics of the oil to get information for industrial applications of the fatty oil.

2. Material and Methods

The seeds of Trigonella foenum graecum Linn. were collected from hervesting field from north district of Bangladesh. For seed and fodder, deshi methi is from mid September to November. The seed rate for deshi methi is 30-35kg/hector and for kasuri-methi is 23-27 kg/hector.

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1. Extraction of the oil from seeds and its purification.

Extraction; at first methi seed was crushed and then crushed seed was taken in a thimble prepared of filter paper. Definite amount of crushed seed (97g) was taken in the thimble and the thimble was placed in the soxhlet extraction unit. Petroleum ether (60-80℃) was used as solvent for extraction. Hot extraction was carried out in water bath for 24 hours at (80-90℃).

Purification: The methi fatty oil in the solvent obtained from soxhlet unit was filtered to remove impure materials. The solvent was removed from the mixture by using a condenser operating at 90℃. At this temperature pet ether vaporize and when this vaporized petether passes through condenser and this condensed petether was collected in a RB flask. The petroleum ether solvent was pumped out from the mixture by using a water pump. The trace amount of solvent remained in the fatty oil was eliminated by using high vaccum. Finally the oil is as dried over anhydrous sodium sulphate. By this process 4.88ml fatty oil was found. The percentage of the fatty oil 5% was calculated.

Physical parameter characteristics studies of the oil

Table 1. Physical characteristics parameters of methi seeds oil

SL NO	Characteristics	Methi seed oil
01	Taste	Bitter
02	Odor	Foetid
03	Color	Reddish yellow
04	Appearanch	Clear
05	Specific gravity at 30 degree Celcious	0.9167
06	Refractive index at 29 degree celcious	1.4815
07	Viscosity at 30 degree celcious	0.2725
08	Percent of oil	5%

All the physical characteristics of Trigonella foenum

graecum such as color, appearance, specific gravity, refractive index were determined by conventional methods under reference and the result are shown in Table-1.

Chemical characteristics of methi seeds fatty oil:

The chemical test were performed to evaluate the nature of the Methi seeds fatty oil. The value were expressed in terms of some customary constsntst. The acid value of the methi seeds fatty oil was found to be 2.18. Acid value indicates the proportions of free fatty acid in the oil. The free fatty acid is produced by the hydrolytic decomposition of the oil. The acid value 2.18 indicates that the oil is not suitable for edible purpose. Iodine value gives an estimanation of the degree unsaturation and thus allows its classification into nondrying, drying, semidrying oils. The iodine value of methi seeds indicates that the seed sample contain moderate proportion of unsaturated fatty acids and are of semidrying types. The saponification value of the methi seeds fatty oil was found to be 182. Saponification value is inversely proportional to the avearage molecular weight or chain length of the fatty acid present in the fat or oil. The result clearly indicates that the sample contain higher proportion of high molecular weight fatty acid. The unsaponifiable matter in the methi seeds oil was found to be 3.6%. In general, if a fixed oil or fat contains unsapoifiable matter in excess of about 2% there is reason to support adulteration. The result indicates that the methi seeds oil sample may contain a much amount of unsaponifiable matter such as sterols, vitamins, A&D, hydrocarbons etc. The ester value of the methi seeds oil was found to be 100.15. This value indicates the fair amount of ester present in the sample. The henher value of the sample was found to be 48.55. This result indicates the higher percentage of water insoluble nonvolatile fatty acid present in the sample. The Reichert-Meissl value also indicates the higher percentage of water insoluble fatty acid present in the sample The Peroxide value is an indication of unsaturation present in fats and oil. The more unsaturated fats or oil absorb more oxygen, from greater amounts unstable hydroperoxide and show higher peroxide value. The result indicates that the methi seeds oil sample may contain moderate amount of unsaturated fatty acid. The results of various chemical tests are shown in the following Table-2.

Table 2. Chemical characteristics of methi seeds fatty oil

SL NO	Characteristics	Results
01	Acid value	2.18
02	Iodine value	115.36
03	Saponification value	182
04	Unsaponifiable matter	3.6%
05	Ester value	100.15
06	Hanus value	48.55
07	Reichert-meissle value	1.073
08	Peroxide value(milliequivalent/L)	8.25

Estimation of the fatty acids by GLC

Estimation of the purified oil: The estification 0f the purified oil was done with methanol by using standard method. Methyl ester thus prepared was then purified. A portion of the purified methylester was injected into one end of the column of a gas liquid chromatography using a flame ionization detector and a chart recorder. Standard fatty acid, methl esters (E. merck) were used for the identification of the peaks.

The standard comprised of the methl esters of caprilic acid, lauric acid, myristic acid, palmatic acid, linoleinic acid, oleic acid, stearic acid, arachidic acid and behenic acid. The fatty acids present the experimental sample were identified by comparing the relative retention time and peak positions of the sample. At the same time, studies on the fatty acid composition of the methi fatty oil was done. The RF value of the ester was determined on TLC. The RF value of the five spots on the TLC plate coincided favourably with five standard acid. Chromatography with those of the standard and the corresponding amount of oil were determined from the areas under the peaks (Table-3).

Table 3

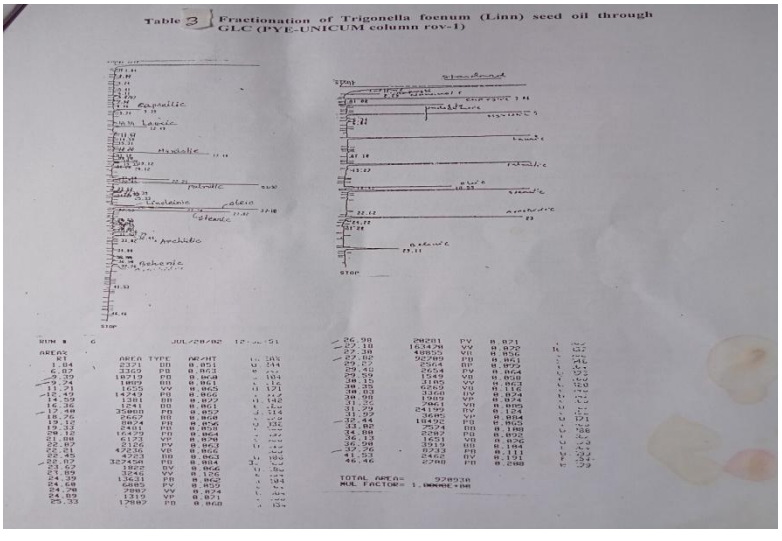

Table 4. Analysis of Fatty acid mixture of Trigonella foenum-gracecum oil

Retention time	Area	Name of the acids	Relative
9.74	107	Caprilic acid	1.5
12.49	147	Lauric acid	2.1
17.40	350	Myristic acid	5.0
22.87	3274	Palmitic acid	47.4
26.98	202	Linoleinic acid	2.4
27.18	1634	Oleic acid	24.0
27.82	927	Stearic acid	13.4
32.44	184	Arachidic acid	3.0
37.76	87	Behenic acid	1.2

Analysis of the seed cake

Seed cake obtained after oil extraction was dried and subjected for determination of percentage of moisture, ash crude fibre, starch, protein and the value obtained were 14.33%, 3%, 8.6%, 24.65%, 4.7%, 29.38%, respectively which shows in Table-5. Result of analysis of methi seeds cake found from literature (Nutritive value of Indian foods) were as follows, moisture 13.7%, protein 22.6%, ash 3%, crude fibre 7.2%. Oil seed cake is the residues would act as valuable feeds for livestock as it is rich in protein. This agricultural residues serve as a major valuable feedstock for cattle or fish.

Table 5. Constituents of methi seeds cake

SL. NO.	Analysis	Result
01	Moisture Content	14.33%
02	Ash Content	3%
03	Crude Fibre	8.6%
04	Starch Content	24.65%
05	Nitrogen Content	4.7%
06	Protein Content	29.38%

3. Results and Discussion

The physio-chemical characteristics of Trigonella foenum oil was estimated. Its oil content was to be 5% which is higher percentage quantity than super critical CO_2 extraction ($SC\text{-}CO_2$) of method carried by Renming et al. They got the percentage of oil was 3.78%. Table-1 report the analysis of physio-chemical characteristics of the oil. From the data, it has been seen that the refractive index of the oil was 1.4815 at 29 c which indicate that the oil contains long chain fatty acids. The specific gravity of the oil was determined and found to be 0.9167 at 30℃. The viscosity of the oil was found to be 0.2725 at 30℃ which indicates that the oil contain low molecular weight of fatty acids. The acid value and the percentage of FFA (as oileic) was calculated and it was found to be 2.18 and 1.10% respectively which indicates that the oil is not suitable for edible purpose. Another researcher (Ling –Biao et. al.) found the refractive index was 1.479+_0.233, relative density 0.922+0.021 which are nearable same the getting value. We found The safonification value, iodine value and Peroxide value were found to be 182 and 115.36 g oil and 8.25 mili equivalent 0_2 per Kg of oil but another researcher (Khaled Hamden et. al) found that 189, 110 and 12 mili equivalent O_2 per Kg respectively, which were very close to the result. The safonification value indicates the presence of high proportion of higher fatty acid and iodine value also indicates the oil to be unsaturated one which was very samely revealed with GLC result. Again the peroxide value indicates that the oil is unsaturated and it is confirmly indicate by the oil characterization. The fatty acid composition was determined by GLC. It was found that (as shown in Table-4) the oil contained the highest proportion of palmitic acid 47.4%. Other fatty acid e. g. oleic acid 24%, stearic acid 13.4%, myristic acid 5% and arachidic acid 3% were also found in significant amounts. The acid constituent also revealed the result of $SC\text{-}CO_2$ method which composed of 28.3% c 18:3, 33.45% C 18:2, 9.89% C 16, 8.1% C18:1, 3.7% C18, 0.71% C20 and 0.61% C22 (Renming et al.) The fenngreek oil is rich in unsaturated fatty acids and polyunsaturated fatty acids accounted for 61.42% of the total amount. Some standard fatty acid methyl ester samples were used in the GLC analysis. There are still unknown peaks which could not be identified for want of reference samples. It is evident from the Table-4 that the seed oil contains considerable amount palmitic acid and this may be trapped as a source of palmitic acid. From Table-5 the qualitative and quantitative composition of the de-oiled portion of oil cake showed about 29.38% protein and 24.65% starch content followed by about 8.6% crude fibre. Another researcher (Ahamad Dilbhad, J. for medicinal chemistry) showed the content of moisture (5.47+0.66)% and oil content (7.04+021)% whereas we found in 14.33% moisture in the form of oil cake. We get protein in oil seed cake 29.38% but other researcher (Nasim Khorbhidian et al., Nutrition and food science research) found 25.4% and get excellent percentage of minerals 3%.

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