

Studies on the Characterization and Glyceride Composition of Safflower (*Carthamus tinctorius*) Seed Oil

M. Rafiquzzaman, M. Altaf Hossain and AJM Moynul Hasan

BCSIR Laboratories, Rajshahi-6206, Bangladesh

Abstract

Studies were carried out on the characterization and glyceride compositions of safflower (*Carthamus tinctorius*) seed oil. It was observed that safflower seed grown under the soil and climatic condition of Bangladesh contains 32 % of golden yellow coloured oil. The physicochemical characteristics of the oil were studied by the conventional methods and the fatty acid composition by GLC. From the results it is revealed that the oil can be used suitably in paint, varnishes and soap manufacturing industries on account of its high linoleic acid content (75 %), iodine value (148) and saponification value (190). The oil was fractionated into mono-, di-, and triglycerides by silicic acid column chromatography. The triglycerides were accounted for over 90 % of total weight of the oil. Fraction of lipids into three major lipid groups, neutral lipids, glycolipids and phospholipids was carried out by silicic acids column chromatography. The neutral lipids were averaged to 94 % of the total weight of the lipid applied. The percentage composition of individual fatty acids were found to be linoleic acid (75.0), oleic (12.6), palmitic acid (8.4), stearic acid (2.6) and myristic acid (1.4).

Introduction

Safflower (*Carthamus tinctorius*) is an erect annual herb with spinosely strate leaves. It grows more or less in all over Bangladesh. But it grows well in the northern part of Bangladesh especially in the districts of Rajshahi, Natore, Naogoan and Chapai-Nawabgonj. It likes to grow mixed with wheat, barley, gram or guar. The crop is drought and wind resistant. Its seed is reported to contain about 25-37 % fatty oils.¹ The oil can be used as an important industrial product.² The oil rich in linoleic acid and is somewhat similar to linseed oil.³ It is drying

oil of similar compounds of wal-nut oil.⁴ In the presence of dryer, safflower oil dries as fast as linseed oil.⁵ The oil lends itself satisfactorily to heat treatment, refining and bleaching.⁶ The safflower is replaced linseed oil in most organic coatings in some circumstances with advantage.⁷ The oil can be used for cooking, illuminating or soap manufacture.⁸ On accounting of its good drying properties, safflower oil is also used for the manufacture of paints, varnishes, linoleum, glass-cement etc.⁹ The oil is useful in rheumatism and hypertension.¹⁰

Bangladesh at present produces large amount of safflower seed annually. No attempt has yet been taken to evaluate the characteristics and glyceride compositions of locally available seeds oil which will have a great profound industrial importance.

Materials and Methods

Safflower seed was collected from local market / hut of Rajshahi. The seed was cleaned and dried in the oven at a temperature of 105^o C for an hour and then crushed into smaller particles in a iron mortar. The oil was then extracted in a soxhlet apparatus with n-Hexane for about 8 hours and by hydraulic pressing (2000 psi). The solvent was removed by a rotary vacuum evaporator and the percentage of oil contain was calculated. The crude oil thus obtained was purified in a column (neutral alumina in pet.-ether) using petroleum ether- diethyl ether (70 : 30) as the eluting solvent. The purity of the oil was checked by normal TLC. The specific gravity of the oil was calculated at 28^o C with the help of Pycnometer. Moisture and volatile matter, in the oil were determined by IUPAC method.¹¹ Refractive index of the clear oil free water and air bubbles was also determined at 25^o C by IUPAC method. The free fatty acid (FFA), saponification value, peroxide value and unsaponifiable matter in the oil were determined by the standard AOCS method.¹² Hanus method¹³ was followed to determined the iodine value of the oil.

Separation of glycerides

The whole oil was separated into mono-, di- and triglycerides by column chromatography

on silica gel 100/120 mesh. The silica gel was dried to constant weight at 200^o C and hydrated with 5 % water. 1g oil was dissolved in 15ml of chloroform and transferred to the column. The triglyceride was eluted with 200 ml of benzene, diglyceride with 200 ml of 1:9 (V/V) mixture of diethyl ether and benzene and monoglyceride with 200 ml of diethyl ether. The elution was controlled at a flow rate of 1.5 ml/min The elution of each fraction was collected in weighted flask and the eluted solvents were removed in a rotary vacuum evaporator under reduced pressure before being weighed. The purity of the separated fractions was further checked by TLC using silica gel developed with n-Hexane : diethyl ether 80/20(V/V) and visualization with chromic sulphuric acid at 180^o C.

Separation into lipid groups

Total lipid extracted by Bligh and Dyer method¹⁴ was fractionated into three major lipid groups neutral lipid, glycolipid and phospholipids by silicic acid chromatography on about 150 mg safflower seed oil lipids. Neutral lipids were eluted with chloroform, glycolipids with acetone and phospholipids with methanol. The elution was controlled at a flow rate of 0.5 ml - 1.0 ml./min. The complete elution of each fraction was collected in weighed vials. The percentage of these fraction was determined by gravimetric method.

Analysis of fatty acid composition of safflower seed oil

The fatty acid composition of safflower seed oil was analysed as their methyl esters which was prepared by the Boron trifluoride

methanol ($\text{BF}_3\text{-CH}_3\text{OH}$) complex method.¹⁵ A GCD Pye-Unicum type gas chromatography equipped with a flame ionization detector was used to determine the fatty acid methyl esters. Nitrogen carried gas was used at a flow rate of 30 ml/min. Fatty acids were separated on a 1.8 x 1/8 i.d glass column packed with 6 % BDS (Butanediol succinate polyesters) on solid support Anakoram ABS(100/120) mesh. Analysis was carried out at isothermal column temperature 190^o C, injector and detector temperature for all GLC analysis were maintained at 230^o C. Gas chromatography peak were identified by comparison with standard methyl esters with respect to retention times against equivalent carbon length (ECL), peak areas were measured by a Pye-Unicum electronic integrator. The percentage of each peak was calculated as the percentage of total area of all the peaks.

Results and Discussion

The solvent and pressing extraction of safflower seed oil yielded 32 % and 20 % of golden yellow coloured oil respectively. The purity of the oil was carried out by column chromatography and was verified by normal TLC. The physico-chemical characteristics

in Table I. From the results it has been shown that the physico-chemical data of the oil are more or less agree with those given by Daniel Swern.¹ But the iodine value indicated that the amount of unsaturated acid is predominant in the oil. Specific gravity and refractive index are normal in comparison with other vegetable oils.

Table I. Physical and chemical characteristics of safflower seed oil

1. Percentage of oil (by solvent extraction)	32 %
Percentage of oil (by pressing 2000 psi)	20 %
2. Moisture and volatile matter	0.125
3. Specific gravity at 28 ^o C	0.9210
4. Refractive index at 25 ^o C	1.4735
5. Melting point	30-31 ^o C
6. Free fatty acid (% FFA) as oleic	1.3
7. Peroxide value (M.eqO ₂ /Kg.oil)	2.27
8. Saponification value	190
9. Unsaponifiable matter	1.25
10. Iodine value	148

The whole oil was fractionated into mono-, di- and triglycerides by means of column chromatography and the results are represented in Table II, it is evident that triglyceride of the oil was accounted for over 90 % of the total weight of the oil.

Table II. Glyceride composition of safflower seed oil (Weight %)

Name of the seed oil	Monoglyceride	Diglyceride	Triglyceride	Nonglyceride unsap+FFA
Safflower seed oil	3.1	2.7	90.5	1.3

of purified oil were determined by the conventional methods and the results are given

Lipid compositions of safflower seed oil are shown in Table III, it is observed that neutral

Table III. Lipid composition of safflower seed oil (Weight %)

Name of the seed oil	Neutral lipids	Glycolipids	Phospholipids
Safflower seed oil	94.0	4.5	1.2

lipids was found to be over 94 % of the total weight of the lipid.

The fatty acid compositions of the oil are presented in Table IV. From the results it is showed that unsaturated fatty acid in the oil mainly linoleic (75) and oleic (12.5) which altogether accounted for over 87 % of the total fatty acids.

Table IV. The fatty acid composition of safflower seed oil (Weight %)

Fatty acid	Weight percentage
Linoleic acid	75.0
Oleic acid	12.6
Palmitic acid	8.4
Stearic acid	2.6
Myristic acid	1.4

Conclusion

Safflower seed oil can be considered as an important industrial raw material. Except edible and illuminating purposes, many derivatives such as paint, varnishes, linoleum, glass cement, fixing agent, soap etc. may be prepared from it for its higher iodine and saponification value.

Acknowledgement

The authors express their sincere gratitude to Mr. M. Hazrat Ali, Director, BCSIR Laboratories, Rajshahi for giving kind permission and encouragement during the study.

Reference

- Daniel Swern (ED). Baily's industrial oil and fat products. 3rd Ed. Interscience Publishers. New York, (1951) 211.
- The Wealth of India, A Dictionary of Indian Raw Materials and Industrial Products, **Vol II** (1951) 85.
- Arthur and Elizabeth Rose. The condensed Chemical Dictionary, 7th Ed. (1966) 824.
- Joceln field Thorpe and M. A. Whiteley, Thorpes Dictionary of Applied Chemistry, 4th Ed. **Vol IV** (1940) 86.
- Pugsby and Winter, Rep. Dep. Muniton, Aust, No. **171** (1947) 287.
- Howard and Remington, *Bull Ogric Res. Ins*, No. **124** (1921) 211.
- Pugsby and Winter, *Indian Soap J, loc. Cit. II* (1956) 807.
- Gholam Mawla, N. M. Sheik and A. S. M. Kamal. A hand book of edible oils and fats, 1st Ed. July, (1990) 183.
- K. N. Ninan. Edible oil seeds, 1st Ed. (1987) 108 and 109.
- Kaliphodo Biswas and Shree Eq Kori Ghoes. Bharotiyo Bonushudhy, 2nd. Ed. 3rd part, (1950) 639.
- International Union of Pure and Applied Chemisty. Standard Methods for the analysis of Oils, 12. Fats and Derivatives, Pargamom Press, 6th Ed. (1979) 126.
- Official and Tentative Methods of the American Oil Chemist's Society (AOCS) **Vol-I** USA (1980).
- Association of Official Agriculture Chemists, Official Methods of Analysis, 8th Ed. Washington, (1955) 468.
- J. Jayrama. Laboratory Manual in Biochemistry, Wiley Eastern Ltd. 2nd Ed. (1985) 96.
- R. K. Das. Industrial Chemistry, Part-2. Kalyani Publishers, New Delhi, India, 4th Ed. (1987) 279.

