

**Chemical Examination of *Cuscuta Reflexa*, Roxb.
Part III. The Constituents of the Oil from
the Seeds.**

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In two previous parts of this series the stem of the plant *Cuscuta reflexa*, which is commonly known as *Amarbel*, has been put to a thorough chemical examination (Agarwal and Dutt, *J. Indian Chem. Soc.*, 1935, 12, 384, 586). The seeds of *Cuscuta reflexa*, which are commonly known as *Khasus* in vernacular, are also reputed to possess anthelmintic properties. In the present communication they have been put to a thorough chemical examination. The oil obtained by the benzene extract of the seeds has been worked up in details. The isolation and constitution of a yellow colouring matter belonging to flavone group is described in a subsequent paper. The oil consists of linolenic acid (9.92%), linolic acid (17.26%), oleic acid (25.58%), stearic acid (27.2%), palmitic acid (11.5%) and unsaponifiable matter (1.8%).

E X P E R I M E N T A L .

5.3 Kg. of the authentic dried sample were obtained from Amritsar (Punjab) and were crushed in a powerful crushing machine. The crushed material was then repeatedly extracted with boiling benzene in a big extraction flask. The combined benzene extracts were then concentrated in order to remove the solvent when it was obtained as a greenish yellow mobile liquid. On keeping it for about a week some white crystalline matter began to separate, which was filtered on a filter pump. It was recrystallised from boiling ethyl alcohol when practically colourless flakes were obtained (2.65 g.) which were identified as cuscutalin (Agarwal and Dutt, *J. Indian Chem. Soc.*, 1935, 12, 384).

The oil was freed from traces of the solvent and purified by heating with animal charcoal and Fullers' earth on a water-bath. On filtration the oil was obtained as a transparent light greenish yellow liquid.

Examination of the Oil.—The oil contained no nitrogen or sulphur. It burnt with a sooty flame. On examination it was found to be a

semi-drying oil since it became viscous on exposure to aerial oxidation. Table I contains the usual physical and chemical constants of the oil.

TABLE I.

<i>Oil.</i>	
Sp. gr.	0.9352 at 23°
Refrac. Index	1.4820 at 25°
Solidifying pt.	-10°
Acid value	3.25
Sap. value	189.5
Acetyl value	17.4
Unapon. matter	1.5-1.8%
Hehners' value	93.0
Iodine value	96.9

TABLE II.

<i>Fatty acids.</i>	
Consistency	Solid
Sp. gr.	0.9026 at 25°.
Refrac. index	1.4639 at 25°.
Neutral value	192.9
Mean M. W	290.9
Iodine value	111.3

90 G. of the oil were then saponified in the usual manner and the unsaponifiable matter extracted with ether. The fatty acids were then liberated and purified (yield 80 g.). Table II gives the physical and chemical constants of the liberated mixed fatty acids.

The mixture of the fatty acids was then separated into the saturated and unsaturated acids by the Twitchells' lead salt-alcohol method (*Ind. Eng. Chem.*, 1921, 13, 806) and Table III gives the percentage, iodine value and the mean molecular weight of the saturated and unsaturated acids.

TABLE III.

Acids.	Percentage in mixed acids:	Iodine value.	Mean M. W.
Saturated	41.32	3.02	265.6
Unsaturated	58.68	150.52	280.9

Examination of Unsaturated Acids.—10 G. of the unsaturated acids were dissolved in excess of caustic potash and oxidised by 2% aqueous solution of potassium permanganate in small instalments at the ordinary temperature. After the oxidation was complete, a current of sulphur dioxide was passed through the mixture. A white substance in flakes remained undissolved, which was filtered and extracted with

ether. The ethereal extracts after the removal of ether deposited a crystalline substance as white flakes, m. p. 136° , and was identified as dihydroxystearic acid. The ether-insoluble portion was next extracted with boiling water and the extracts on cooling deposited white crystals, m. p. 172° , which proved to be tetrahydroxystearic acid. Traces of hexahydroxystearic acid were also present (m. p. 201°) in the original filtrate. The formation of these substances establishes the presence of linolenic, linolic and oleic acids in the unsaturated acids.

The quantitative estimation was next done by the bromine addition products by the method adopted by Jamieson and Baughman (*J. Amer. Chem. Soc.*, 1920, **42**, 1198; cf. also Agarwal and Dutt, *Proc. Acad. Sci. U. P.*, 1935, **5**, 227). A known weight of the unsaturated acids was dissolved in 150 c.c. of dry ether and treated with excess of bromine at -10° to -5° . On standing at this temperature for 2 hours a white precipitate of the linolenic hexabromide (m.p. 181°) separated which was filtered, washed and weighed. The excess of bromine was removed from the filtrate by thiosulphate, and the ether evaporated away. The residue was taken up with about 200 c.c. of dry petroleum ether and kept overnight. Linolic tetrabromide separated as fine glistening star-shaped needles (m. p. 112°) which was filtered, washed and dried. On concentrating the mother liquor a further crop of the tetrabromide was obtained which was added to the first and weighed. Finally the petroleum ether filtrate was evaporated to dryness and the bromine content of the residue determined.

TABLE IV.

Percentage of the various components.

Acids.	In unsaturated acids.	In mixed acids.	In the original oil.
Linolenic	18.62%	10.92%	9.92%
Linolic	32.51	19.07	17.26
Oleic	48.42	28.41	25.58

Examination of the Saturated Acids.—The mixture of the saturated acids (16 g.) was converted into their methyl esters and fractionally distilled under reduced pressure. The saponification values of the different fractions were determined and the mean molecular weights

calculated. The acids were liberated from different fractions and melting points determined. The following table contains the results of these experiments.

TABLE V.

B. P. at 0.5 mm pressure.	Wt.	Sap. value.	Mean M.W.	Acids		M.p. of liberated acid.
				Palmitic.	Stearic.	
160-170° rose to 180°	2.12 g.	204.1	274.0	1.68 g.	0.34 g.	63-64°
180-182° rose to 190°	1.80	200.0	280.5	1.09	0.64	
190-192°	4.63	192.5	291.5	1.08	3.32	64-65°
192-195° rose to 200°	4.80	190.1	295.2	0.52	4.05	67-68°
Residue.	2.21	189.5	296.0	0.18	1.92	66-67°
				4.55 g.	10.27 g.	

TABLE VI.

Acids.	In saturated acid.	In mixed acids.	In the oil.
Palmitic	30.71%	12.69%	11.5%
Stearic	69.29	28.63	27.2

Examination of the Unsaponifiable Matter.—The unsaponifiable matter was freed from the solvent when yellowish white waxy flakes were obtained. It was dissolved in water and repeatedly extracted with ether. The ethereal extracts were dehydrated with fused calcium chloride and the ether evaporated. The residue was once crystallised from acetone when colourless flakes were obtained, m. p. 126-128°. On recrystallisation from alcohol a phytosterol was obtained in fine silky needles, m. p. 134-35°. The acetyl derivative was prepared in the usual manner, m. p. 124-25°. The optical rotation of the sterol in chloroform solution was $[\alpha]_D^{25} = -30.9^\circ$.

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