CHEMICAL EXAMINATION OF THE FATTY OIL FROM THE SEEDS OF *MALLOTUS PHILIPPINENSIS* MUEL.-ARG.

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In 1936 Krishna[1], *et al.*, drew attention to the drying properties of the fatty oil from the seed kernels of *Mallotus philippinensis* Muel.-Arg. (Fam. Euphorbeaceæ) commonly known as "Kamala" or "Kamela" in Hindi and as "Rohini" or "Raini" in Kumaon and Dehra Dun Districts of Uttar Pradesh. In 1941 and 1942 the results of the preliminary chemical examination of the fatty oil were published in the Annual Reports[2] of the Forest Research Institute and Colleges. This was soon followed by the notes of Singh and Saran[3] and Puntambekar,[4] the latter worker pointing to the highly drying nature of the oil comparable to that of tung Oil. In this laboratory further work was suspended during the remaining period of the World War II but the study of the component acids of the oil was again resumed in early 1946 and the results reported in the subsequent Annual Reports[5] of this Institute. In 1948 Aggarwal,[6] *et al.*, published their first paper on the chemical examination of the fatty oil giving a different picture of its chemical composition. This was followed in 1949 by a paper from Bhushan and Om Prakash[7] on the chemical and physical characteristics of the oil as well as its performance as a fast drying oil when used alone or in combination with double boiled linseed oil. The recent letter by Aggarwal,[8] *et al.*, gives the possible structural formulæ for Kamlonic acid, the active component conjugated double bond acid, isolated by them from the oil. In view of the different findings of the author to that reported by Aggarwal, *et al.*, it is now thought desirable to put together the findings from this laboratory.

The black seeds from the fruit of Kamala were found to contain 22 to 24 per cent. of a pale bland viscous oil having medicinal properties but they were mostly treated as a waste product in the preparation of Kamala powder of trade. The oil could not be expressed from the kernel in a hydraulic press probably on account of its viscous nature and tendency to polymerise owing to the effect of heat, pressure, air and light. It was, therefore,

extracted from the fresh seed kernels with petroleum ether, sulphuric ether and benzene with the following results:

Solvent	Colour	Consistency	Yield in kernels	Behaviour on standing
1 Petroleum ether ..	Reddish brown	Thick liquid	50%	
2 Sulphuric ether ..	do	Viscous liquid or solid	50%	
3 Benzene ..	Dark brown	Very viscoas	61%	Becomes solid & hard and insoluble in the solvents for fatty oils.

Samples of the fat extracted with sulphuric ether and kept in sealed bottles in a refrigerator were found to be practically unaffected after one year.

It has been found that the oil exposed to atmosphere in a thin film changes to a rubbery mass at room temperature (30°) within 48 hours. The effect is quite rapid if the oil or its mixed fatty acids are heated either alone or with chemicals like acetic anhydride and lead acetate. Thus in the determination of acetyl value and separation of solid and liquid acids by the Twitchell's method the oil and its fatty acids have been found to get altered partially to leathery products. The change appears to be a progressive one particularly in case of the mixed fatty acids which in contact with air get altered to a hard insoluble product from the exposed surface to the interior. During conversion of the acids to their lead salts and separation of the latter from alcohol into alcohol-soluble and alcohol-insoluble salts as much as 20% of the acids when liberated from them were found to have become rubbery. Since the oil shows a high refractory index (1·5105), a high iodine value (183), a significant diene value (42) and an appreciable carbonyl value (26) and is four times as fast jellying as tung oil not only the presence of a conjugated system of double bonds in the component acids is indicated but also an additional factor of activity in the structure of the fatty oil and acids. It has been noted that mixtures of the oil in other drying, semi-drying and non-drying oils like linseed, olive, castor, coconut, cocobutter, etc., as well as with solvents like benzene, toluene, xylene, turpentine, etc., when heated react and set to gels, indicating addition of the conjugated system of double bonds of the Kamala oil to the double bonds of the glycerides, benzene, toluene, xylene, turpentine, etc. This is a very significant property of the oil suggesting many practical applications.

In the study of the component fatty acids of this highly reactive oil, the problem has been now actually to isolate them without altering their

nature and structure. The fatty oil was kept in bottles tightly corked and sealed with paraffin wax and stored in a refrigerator. The following procedures for the separation and identification of individual acids were adopted:

(*a*) The mixed acids were separated mainly into "solid acids" and "liquid acids" by the modified Twitchell's method.[9] The solid acids which contained almost all the polythenoid conjugated acids were isolated and identified by fractional crystallization from different solvents and catalytic hydrogenation of the different fractions. The permanganate oxidation products of solid acids in acetone were also studied.

The liquid acids were identified by their permanganate oxidation and bromination products.

(*b*) The mixed acids were reacted with maleic anhydride to form adducts with the conjugated polyethenoid acids present in them; after the removal of the adducts the components of the residual acids were studied.

This study has indicated that the component acids are mostly unsaturated acids mainly conjugated polyethenoid C_{18} (probably isomeric-eleostearic and isomeric keto-linoleic acids) oleic and linoleic acids and to a small extent saturated acids, namely, stearic and lower acids. Although valeric and undecylic acids have been identified in the products of permanganate oxidation of the solid unsaturated acids, the position of conjugated double bonds yet remains to be finalized and that of the keto group in the isomeric keto linoleic or isomeric keto-eleostearic acid still remains indefinite. In this connection attention might be drawn to the different picture with respect to the component acids presented by Aggarwal, *et al.*[6,8] They have found the principal unsaturated acid to be Kamlonic acid (isomeric oxy-eleostearic acid) and that the oxy group is not a keto but a hydroxyl one occupying the (ω) position and having only two double bonds conjugated. Further, instead of stearic, oleic and linoleic acids they have reported the presence of palmitic and myristic acids but have not supplied confirmatory data to prove their occurrence.

Experimental

The air-dry fruit consisted of the pericarp with the outer thin layer of Kamala powder (70·9%) and the black seed (29·1%). The seed in its turn consists of 53% testa and 47% kernel, the latter of which yields 50% of a viscous or solid fatty oil when extracted with solvents like petroleum ether, benzene, sulphuric ether, etc.

The characteristics of the fat extracted with petroleum ether are recorded below along with the values obtained by other workers:

	Author	Singh & Saran	Aggarwal, *et al.*	Bhushan & Om Prakash
Sp. gr. at 30° C.	0·9347	0·9933	0·9409	0·9444
Ref. index at 30° C.	1·5105	1·5155	1·5052	..
Unsaponifiable matter	1·75	1·9	1·7	2·3
Viscosity	..	..	386·3 @ 40° C.	..
Acid value	5·7	11·3	6·4	5·2
Saponification value	178·3	207·6	195·0	190·7
Iodine value	183·2 (Hanus)	157·3	179·8 (Woburn "B")	166·4 (Wijs)
Acetyl value	49·24	46·8	15·7–44	..
Carbonyl value	26·0	..	..	..
Diene value (Maleic anhydride value)	42·9	..	40·4	48·5
Browne heat test	2′	..	9′ 30″	..

COMPOSITION OF THE MIXED ACIDS

200 g. of the fat was saponified in the usual manner with alcoholic sodium hydroxide. On distilling off the alcohol at 70° under vacuum the resultant soap was dissolved in 2 litres of water and the solution extracted for the unsaponifiable matter with sulphuric ether. The ether extracted soap solution was then carefully treated with con. hydrochloric acid, warmed up to 70° and the liberated mixed acid separated from the lower watery layer. The mixed acids were taken up in sulphuric ether, washed with water dried over anhydrous magnesium sulphate and the ether distilled off.

CHARACTERISTICS OF THE MIXED ACIDS

	1	2	3	4
Melting point	51–52° C.	..	45·5° C.	49·5° C.
Mean molecular wt.	300·6	..	281·2 Calc. from neutralisation value (199·5) obtained by these authors.	..
Iodine value	168·0 (Hanus)	..	182·7 (Woburn "B")	..
Diene value (Ellis & Jones)	58·0	..	56·6	..
Carbonyl value	60·0	..	..	..

100 g. of the acids of the above characteristics were subjected to Twitchells lead-salt-alcohol separated with the following results:

"Liquid" acids .. 31%; i.v. 87–97.

"Solid" acids .. 49%; I.V. 120–140.

Polymerised acids .. 20%.

Liquid Acids

Oxidation.—A portion of the liquid acids was converted into potassium soaps and oxidised in cold alkaline solution with dilute potassium permanganate according to the modified method of Hazura.[10] From the precipitated oxidized acids only dihydroxy stearic acid M.P. 131–32°, m.w. 314, has been isolated.

Bromination.—Freshly prepared liquid acids were brominated according to the method of Eibner and Muggantheler[11] but no insoluble hexa or higher bromides were obtained. The brominated acids (Br, 37·56%) were then crystallised from petroleum ether (b.p. 40°–60° C.) when white needles (m.p. 113°–14° C.) were obtained. These were evidently tetrabromostearic acid and were confirmed as such by their mixed melting point with an authentic sample of the tetrabromide.

Formation of dihydroxy stearic and tetra bromostearic acids and the iodine value of the liquid acids show that they consist of mainly oleic acid (92%) and a small quantity of linoleic acid (8%).

Solid Acids

A freshly prepared sample of solid acids (i.v. 140) on exposure to air soon became resilient and insoluble in the organic solvents indicating their great tendency to polymerize due to high unsaturation. The usual process of converting them into methyl esters and separating the latter into pure components by fractional distillation under vacuum could not be employed as heat readily polymerised them. Therefore, they were successively extracted and crystallized at room temperature in an atmosphere of CO_2 from petroleum ether (b.p. 40°–60° C.) and benzene and separated into the following fractions:

Fractions	Solvents	Yield
(*a*)	Crystals from petroleum ether m.p. 77–78° C. (m.w. 281) ..	25%
(*b*)	Crystals from benzene m.p. 77–78° C. (m.w. 286)	55%
(*c*)	Polymerised product insoluble in benzene	10%
(*d*)	Viscous oil from mother liquor of (*a*)	5%
(*e*)	Viscous oil from mother liquor of (*b*)	5%

The fractions (*a*) and (*b*) were mixed together, dissolved in 95% alcohol and reduced with hydrogen at room temperature in the presence of active platinum oxide catalyst[12] for three hours. After the removal of the catalyst by filtration the products of reduction were isolated on concentration of the alcoholic solution as follows;

(1) Crystals (40%) m.p. 93–94° C., i.v. 9, m.w. 305, acetyl derivative m.p. 79–80° C. (Found: C, 71·5%; H, 11·7%; calculated for hydroxy saturated C_{18} acids $C_{18}H_{36}O_3$, C, 72·0; H, 12·0%).

(2) Crystals from mother liquor (15%), m.p. 69–70°, mixed melting with an authentic sample of stearic acid remained unchanged.

(3) Dark reddish brown liquid acids (45%) from the mother liquor of (2) i.v. 80, m.v. 280, indicating most of the material to be an isomer of oleic acid obtained as the result of partial reduction of conjugated system of double bonds of isomeric eleostearic acid.

Fraction (*c*) being insoluble in organic solvents was not worked up further; fractions (*d*) and (*e*) being almost similar were mixed together and the mixture on further hydrogenation gave a small quantity of acids (i.v. 8, m.w. 267) melting at 55° C. to 56° C. indicating mixtures of stearic acid with some lower saturated acids.

It will thus be seen from the above data that the mixed acids consist of 31% of liquid acids (oleic and linoleic acids) and 69% of solid acids mostly C_{18} polyethenoid acids (i.v. over 120) which as a result of undergoing Twitchell's separation gets converted to an extent of 20% to a hard polymerised product insoluble in organic solvents. The remaining 49% besides the multiple double bonds contain oxygen in the molecule and during its fractional separation from solvents gets further polymerised to the extent of 10%. The unpolymerised polyethenoid and oxygen containing acids on catalytic reduction yield an oxy-stearic acid isomer, m.p. 93–94° C., stearic acid, m.p. 69–70° C., an iso-oleic acid (i.v. 80, m.d. 280) and small quantities of saturated acids lower than stearic acid. The above data on calculation gives the following composition of the mixed acids:

Oleic acid	28·6%
Linoleic acid	2·4%
Polyethenoid keto C_{18} acid	15·7%
Isomeric eleostearic acid	23·5%
Saturated acids	4·9%
Polymerised acid	24·9%

Since the liquid acids have not been found to polymerise at any stage and since the solid acids have actually yielded 10% of polymerised acids during their fractional crystallization the polymerisation undergoing constituents evidently are located in the solid acids and no doubt are the conjugated polyethenoid keto C_{18} and isomeric eleostearic acids found in them. The polymerised acids can, therefore, be taken to have the same proportionate composition in the unpolymerised state as is represented by poly-ethenoid

keto C_{18} and isomeric eleostearic acids in the above table for the composition of the mixed acids. Taking these facts into consideration the composition of the total mixed acids in the unpolymerised state works out as follows:

Oleic acid	28·6%
Linoleic acid	2·4%
Polyethenoid keto C_{18} acid ..	25·7%
Isomeric Eleostearic acid ..	38·4%
Saturated acids	4·9%

Oxidation of the solid unsaturated acids in acetone solution with potassium permanganate, removal of acetone by distillation and reduction of the excess permanganate and manganese dioxide with sodium sulphite and sulphuric acid gave a mixture of fatty acids, on steam distillation of which a volatile acid was obtained and was identified as valeric acid (m.w. 99). The residual mixture of fatty acids on extraction with sulphuric ether and crystallization of extract from dilute acetone in cold yielded a product (m.w. 187·5) which was identified as undecylic acid (m.w. 186). No other compound could be isolated in a pure condition.

In the light of the above findings the polyethenoid keto C_{18} acid and the isomeric eleostearic acid present in the solid acids would respectively appear to have the following possible structural formulæ:

either

(1) (*a*) $CH_3-(CH_2)_9-CH=CH-CH=CH-CH_2-\underset{\underset{O}{\|}}{C}-CH_2-\underset{\underset{O}{\|}}{C}-OH$

(isomeric keto-linoleic acid)

(*b*) $CH_3-(CH_2)_3-CH=CH-CH=CH-CH=CH-(CH_2)_7\underset{\underset{O}{\|}}{C}-OH$

(eleostearic acid)

or

(2) (*a*) $CH_3-(CH_2)_3-CH=CH-CH=CH-CH=CH-\underset{\underset{O}{=}}{C}-(CH_2)_6-\underset{\underset{O}{\|}}{C}-OH$

(keto-eleostearic acid)

(*b*) $CH_3-(CH_2)_9-CH=CH-CH=CH-CH=CH-CH_2-\underset{\underset{O}{\|}}{C}-OH$

(isomeric eleostearic acid)

The components of the mixed acids have also been found from the procedure adopted for determination of the diene value by the following way:

The mixed acids were treated with maleic anhydride in toluene solution according to the method of Ellis and Jones.[13] The toluene ether solution of the adduct after the removal of the unreacted maleic anhydride with hot water in a separatory funnel was evaporated on a hot water-bath to remove the solvents. The residual adduct was extracted with petroleum ether to remove all the fatty acids (i.v. 82·0) unreacted with maleic anhydride. Then knowing the diene value (58) the composition of the mixed acids works out and compares with the above recorded composition (Twitchells method) as follows:

		Twitchells method
Conjugated double bond acids ..	66·0%	66·5%
Oleic acid	30·0%	28·6%
Saturated acids	4·0%	4·9%

DISCUSSIONS

1. It has been found that the conjugated polyethenoid glycerides and acids dissolved in solvents containing double bonds in the molecule such as benzene, toluene, xylene and turpentine react with the solvents on prolonged standing or heating. The significantly high yield of the fatty oil extracted with benzene, *viz.*, 61% as compared with 50% obtained by petroleum or sulphuric ether extracted oil is due to this effect. Benzene has simply reacted with the conjugated polyethenoid glycerides of the oil and the resulting adduct remains behind on removal of benzene and thereby increases the weight of the oil. Petroleum ether or sulphuric ether extracted oil does not behave in this way.

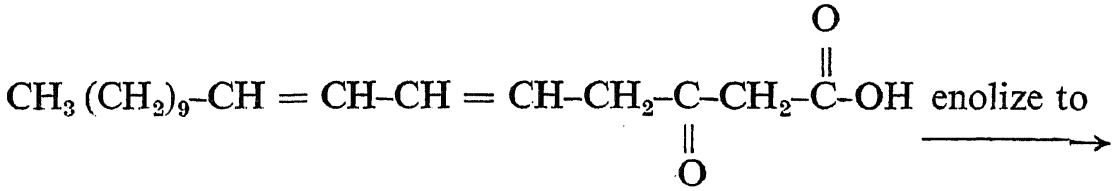
2. The family relationship and the similarity of properties between Kamala and tung oils, which indicates similarity of their chemical composition, is worth noting. Both the oils are derived from the seed kernels of small trees belonging to the same botanical family, *Euphorbeaceæ*. Both have high refractive indices. Both gelatinize rapidly at high temperatures (Browne heat test). Within 48 hours at room temperature in the absence of driers both give films that are opaque, matt and dull producing the typical wrinkled effect. Bodied with linseed oil or with resins both give waterproof varnishes of high gloss and covering power. Both contain about 70% glycerides of conjugated polyethenoid C_{18} acids.

3. The facts that the conjugated polyethenoid acids occurring in "solid" acids are reduced with catalytic hydrogen to hydroxy stearic acid isomer and to an isooleic acid and the latter on further catalytic reduction is reduced to stearic acid indicate that they contain polyethenoid keto C_{18}

and isomeric eleostearic acids. The presence of the former acid is further corraborated by the carbonyl value (60) of the mixed acids.

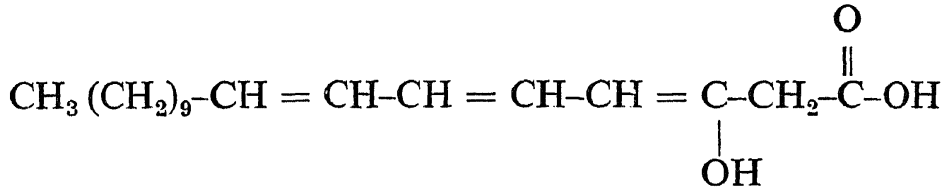
4. Browne's heat test has shown that Kamala oil becomes solid in 2 minutes as compared with $8\frac{1}{2}$ minutes taken by tung oil. This fact considered with the component glycerides of the two oils point to the higher reactive nature of the component glycerides of kamala oil than those of tung oil. Whereas tung oil has been reported to contain 70% glycerides of eleostearic acid, kamala oil has been found to contain nearly the same total percentage of the glycerides of polyethenoid keto C_{18} and isomeric eleostearic acids. In other words a mixture of the glycerides of these two conjugated polyethenoid acids is found to be more reactive than the same concentration of glycerides of eleostearic acid alone. This behaviour leads to the possible inference that the mixture is more reactive because the intermolecular reaction between the two different conjugated polyethenoid glycerides of Kamala oil is catalysed by the oxygen of one of the glycerides. This effect is naturally absent in tung oil which provides no such catalytic component.

5. The structures (1) *a* (isomeric keto-linoleic acid) and (1) *b* (eleostearic acid) proposed for the polyethenoid keto C_{18} acid and isomeric eleostearic acid appear to be more likely than (2) *a* and (2) *b* since the common family relationship of kamala and tung oils would indicate that what has been considered as isomeric eleostearic acid is really eleostearic acid. This inference is further supported by the identification of n-valeric acid in the products of permanganate oxidation. The keto acid having two active methylene groups adjacent to the carbonyl group is readily susceptible to enolizing in presence of catalyst or heat and to produce highly reactive conjugated acids of following configurations:



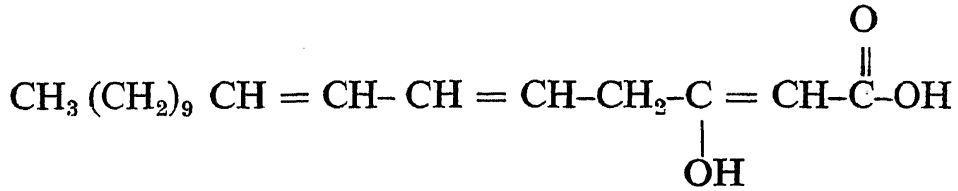
(isomeric keto linoleic acid)

either



(P) (isomeric hydroxy eleostearic acid)

Or



(R) (Isomeric hydroxy eleostearic acid)

The (P) form is expected to be the more reactive than the (R) form as it contains the 3rd double bond in conjugated position. This structure (1 *a*) therefore is in keeping with the finding of undecylic acid in the products of permanganate oxidation of " solid " acids.

6. The main points of difference between the findings of Aggarwal, *et al.*, and the author are as follows:

	Aggarwal, *et al.*	Author
I.	No carbonyl value exhibited by the oil or its mixed acids.	Carbonyl values[14] exhibited by the oil and its mixed acids.
II.	Occurrence of Kamlonic acid or (w) hydroxy isomeric eleostearic acid, α-form, m.p. 77–78°, β-form m.p. 86–87°, m.w. 293·5, which on catalytic reduction gives (w) hydroxy stearic acid, m.p. 100·5° C., m.w. 305, acetyl derivative m.p. 72° C.	Occurrence of a mixture of (x) keto polyethenoid C_{18} acid and isomeric-elecstearic acid (crystals from the petroleum-ether; m.p. 77–78°, m.w. 281, and crystals from benzene, m.p. 77–78°, m.w. 286) which on catalytic reduction and crystallization from alcoholic solution give (x) hydroxy isomeric stearic acid (m.p. 93–94° C., m.w, 305, acetyl derivative 79–80° C.) and stearic acid (m.p. 68–69° C, m.w. 285) respectively.
III.	Browne heat test 9′ 30″.	2′.
IV.	Out of the three double bonds of Kamlonic acid only two in conjugated position.	All the three double bonds of (x) keto polyethenoid C_{11} acid and isomeric elecstearic acid in conjugated position.

7. Aggarwal, *et al.*, have so far advanced no direct proof for the presence of OH group in the (ω) position in " Kamlonic " acid, *i.e.* (ω) hydroxy isomeric-eleostearic acid although its presence is just indicated by the erratic acetyl values (15–44) reported by them for the oil. The only proof they offer is its reduction to hydroxystearic acid by catalytic reduction. On the other hand carbonyl values shown both by the oil and its mixed acids strongly indicate the presence of a keto group in (x) position in one of the conjugated acids actually present initially in the mixed acids. It might be pointed out that Aggarwal, *et al.*, converted the original α-Kamlonic acid (m.p. 77–78° C.) which corresponds to the author's (x) keto polyethenoid C_{18} acid to β-Kamlonic acid (m.p. 86–87; m.w. 293·5) by irradication under ultra-

violet light. That is they have used a well-known isomerisation process to alter the constitution of the original acid and later supplied the proof of its structure by catalytic reduction of the isomerised acid. Why could not the α-form be reduced directly to the hydroxy stearic acid stage? In the present case it is likely that the isomerisation, after the ketol enol tautomerisation analogy, has also tautomerised the original carbonyl group to the hydroxyl.

8. From the data presented by Aggarwal, *et al.*, it will be seen that the mixed acids have the following composition:

Kamlonic acid	..	50%, i.v. 249·6.
Other unsaturated acids	..	38%, i.v. (X), m.m.w. (Y).
Saturated acids	..	12%, i.v., nil, m.m.w., 247.
The total mixed acids	..	100%, i.v. 182·7, m.m.w., 281·2.

Since the constants (iodine value and m.m.w.) for the Kamlonic acid and the saturated acids have been determined by them the same for the other unsaturated acids can be calculated and have been found to be i.v. (X) = 152 and m.m.w. (Y) = 267·7. The acids with these values would indicate a mixture of C_{16} and C_{18} multiple double bond acids. These, however, have so far not been reported. As a matter of fact no data has been so far published by them for the 38% of these unsaturated acids.

9. In view of the above different findings by Aggarwal, *et al.*, and the author it now becomes necessary to study the absorption spectra of the individual as well as the mixed acids of kamala oil to clarify their structure.

Unsaponifiable Matter

The unsaponifiable matter isolated from the saponified oil (sodium soap) by extraction with sulphuric ether before liberation of the mixed acids was crystallized thrice from concentrated alcohol yielding crystal melting at 131–32° C. (acetate m.p., 129–30° C.). The sterol has a specific rotation of −35·99° C. (Conc. 0·139%) in chloroform and appears to be the common phytosterol, sitosterol, found in fatty oils of vegetable origin.

Summary

1. Kamala oil is one of the lesser known indigenous oils of forest origin and perhaps the most rapid drying oil, even faster than tung oil, among those so far known to science.

2. In contact with air and at laboratory temperature it rapidly polymerises to a hard mass insoluble in organic solvents,

3. It consists of the glycerides of isomeric-eleostearic, (x) keto polyethenoid C_{18}, oleic, linoleic, stearic and lower saturated acids.

4. The rapid drying nature of the oil is due to the presence of the glycerides of (x) keto polyethenoid C_{18} and isomeric-eleostearic acids.

5. The unsaponifiable matter of the fatty oil has yielded the common phytosterol, sitosterol, m.p. 131–32° C. (acetate, m.p. 129–30° C.).

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