Composition of Wax from Seed Flax Straw

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ABSTRACT AND SUMMARY

Wax from leaves and stems of ripe seed flax contains hydrocarbons (14%), esters (35%), aldehydes (12%), free acids (2%), free alcohols (17%), and unidentified material (20%). The chain length range of the hydrocarbons is C_{25}-C_{33} (major component C_{29}); of the esters is C_{40}-C_{52} (major component C_{46}); of the combined acids is C_{14}-C_{30} (major component C_{18}); and of the combined and free alcohols, the aldehydes, and the free acids is C_{22}-C_{32} (major component C_{28}).

INTRODUCTION

Flax, *Linum usitatissimum* L., belongs to the family *Linaceae* which has 12 genera and 290 species. Two distinct types of the flax plant have been developed: those known as fiber flax, which produce fiber for linen, and those known as seed flax, which produce oilseeds for linseed oil. Isolation of wax in yields of 2% or more, by solvent extraction of fiber flax or by-product dust, has been reported several times (1,2). The ester value and amount of unsaponifiable matter indicated that it was mainly a nonglyceride wax, but no reliable chemical composition was obtained.

Wax extracted from straw of seed flax has apparently been investigated only once (3). A 3.5% yield of wax was reported, but was probably contaminated by low melting lipid-like material from the interior of the stem since it had relatively high saponification and iodine values and a low amount of unsaponifiable matter.

In this study the composition of the surface or epicuticular wax of leaves and stems of ripe seed flax plants has been determined. As a potential agricultural by-product, it may be useful to know the type of composition to be expected. Also, no waxes had been previously examined from species of the family *Linaceae* (4).

EXPERIMENTAL PROCEDURES

Flax, variety Dufferin, was grown outside; when ripe, the seed bolls were removed and wax extracted from stems and leaves by 10 sec immersion in redistilled hexane (4). The wax was a brown solid with melting point 62-69 C. Gas liquid chromatographic (GLC) analyses were performed as previously described (5). Proton magnetic resonance (PMR) spectra were obtained with a Varian HA-100 spectrometer using CCl_4 as solvent.

Wax (7.53 g) was applied to a silicic acid column, 4 cm x 35 cm, (Biosil A, 200 g, Bio Rad, Richmond, CA) and components eluted with hexane and hexane containing increasing proportions of ether. Hydrocarbons (1.07 g) were eluted with hexane, and a mixture of esters and aldehydes

TABLE I

Composition of Leaf and Stem Wax from Seed Flax[a]

Component	%
Hydrocarbons	14
Esters	35
Aldehydes	12
Free acids	2
Free alcohols	17
Unidentified fractions	
Eluted after esters	3
Eluted after alcohols	7
Lost on column	10
Yield (% of dry weight)	0.2

[a]Calculated from weight of components obtained by silicic acid chromatography.

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TABLE II

Composition of Fractions from Leaf and Stem Wax of Seed Flax

Carbon chain length	Hydrocarbons %	Esters %	Hydrolysis products of esters: Acids %	Hydrolysis products of esters: Alcohols %	Aldehydes %	Free acids %	Free alcohols %
14	-	-	2	-	-	- -	-
16	-	-	24	-	-	-	-
18	-	-	45	-	-	-	-
20	-	-	10	-	-	-	-
22	-	-	5	1	3	-	1
24	-	-	3	2	6	5	5
25	4	-	-	-	-	-	-
26	-	-	3	16	26	15	33
27	13	-	-	-	-	-	-
28	-	-	4	54	40	40	44
29	61	-	-	-	-	-	-
30	-	-	4	25	22	35	16
31	14	-	-	-	-	-	-
32	-	-	-	2	3	5	1
33	1	-	-	-	-	-	-
40	-	1	-	-	-	-	-
42	-	4	-	-	-	-	-
44	-	25	-	-	-	-	-
46	-	49	-	-	-	-	-
48	-	18	-	-	-	-	-
50	-	2	-	-	-	-	-
52	-	1	-	-	-	-	-
Unidentified	7	-	-	-	-	-	-

(3.55 g) was eluted with hexane-ether (99.5:0.5, v/v). Unidentified material (0.23 g) followed by a mixture of acids and alcohols (0.37 g) was eluted with hexane-ether (99:1 to 99:3, v/v). Hexane-ether (94:6 to 90:10, v/v) eluted the remaining alcohols (1.07 g), and unidentified gum (0.53 g) was eluted with hexane-ether (80:20, v/v). Fractions were examined by thin layer chromatography (TLC) (with $CHCl_3$ containing 1% of ethanol v/v) and GLC (4). Components of the various fractions were identified by reanalysis by GLC after addition of suitable authentic compounds (5).

The mixture of esters and aldehydes was separated by preparative TLC with $CHCl_3$-hexane (1:1) solvent. Esters were then converted by acid methanolysis (6) to methyl esters and alcohols and the mixture acetylated (5) and analyzed by GLC. Aldehydes were then examined by PMR and chain length established by reduction to alcohols with $NaBH_4$ in ethanol and analysis (GLC) as acetates. The mixture of free acids and alcohols was treated with diazomethane and acetylated (5), and the proportions of methyl esters and acetates were determined by GLC.

RESULTS AND DISCUSSION

The yield of wax was quite low, only 0.2% of dry weight (Table I), though this might be partly because the leaves were small and the stems relatively heavy. More wax would probably have been obtained if plants had been extracted while still green instead of when the seed bolls were ripe. Also, other flax varieties might have given better yields of wax.

Table I shows wax composition and indicates that esters form the major components, but there are also appreciable percentages of free alcohols, hydrocarbons, and aldehydes. Carbon chain lengths of wax components are shown in Table II. Hydrocarbons have a common composition with nonacosane as major component, and major esters are C_{44} to C_{48}. The composition of the constituent acids and alcohols shows that they are probably formed randomly from C_{16} to C_{20} acids and C_{26} to C_{30} alcohols. Aldehydes, free alcohols, and free acids all have compositions quite similar to those of combined alcohols suggesting a common biosynthetic origin. No unusual components such as diols or hydroxy acids were detected.

Ester composition is similar to that of esters from wax of *Portulaca oleraceae* (which also has esters as principal components) (6) but differs from the latter in that the chain length range is obtained by combination of relatively long chain alcohols with relatively short chain acids. In esters of *P. oleraceae* wax, C_{22} to C_{26} alcohols are esterified by C_{20} to C_{24} acids (6). The major C_{18} acid component of the combined acids is also unusual in wax esters, though not many have been analyzed (4), since C_{16} generally exceeds C_{18}. Acids from esters of wax from the grass *Lolium perenne,* however, have a similar composition to those of the flax esters (7).

The chain length range of the free alcohols, mainly C_{26} to C_{30}, resembles that reported for waxes of other dicotyledonous plants (4), and the presence of triacontanol is of interest in view of a recent report suggesting that this compound is a plant growth regulator (8). Aldehydes, though less frequently reported in plant waxes, are often similar to the free alcohols in chain length. However, free acids, which here also have the same chain length range as the free alcohols, more usually have a much wider distribution of chain length.

Thus, wax from seed flax straw generally resembles other waxes in composition and would not be expected to possess any unusual properties. However, the low yield of wax obtainable from seed flax straw makes it unlikely that it could become a useful by-product.

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