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LIPIDS OF THE PRODUCTS OF PROCESSING OF COTTON SEEDS

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The free, bound, and strongly bound lipids of the crushed seeds, pulp, husks, and meal have been characterized. It has been shown that the bound and strongly-bound lipids differ from the free lipids by a higher level of saturated acids. The acid numbers of the bound lipids are 5-6 times higher than those of the free lipids.

In the production of vegetable oils by the current technology from oil seeds, their lipid composition changes [1]. We have investigated the free (F), bound (B), and strongly-bound (SB) lipids in the processing of cotton seeds: the crushed seeds (I), the pulp (II), the husks (III), and the meal (IV), samples of which were obtained in the Tashkent Oils and Fats Combine. Below we give some indices of the products of the processing of cotton seeds:

Index	Amount, %			
	I	II	III	IV
Free lipids	28.02	28.40	18.00	0.91
Bound lipids	3.24	3.40	3.60	3.71
Strongly bound lipids	0.28	0.32	0.33	0.36
Moisture content	8.12	4.70	3.90	10.29
Free gossypol	1.24	0.31	0.24	0.08

The free lipids made up 88.9% of the mass of the lipids in the crushed seeds, the proportions of bound and strongly-bound lipids being 10.2 and 0.9%, respectively.

In the course of the industrial process for the extraction of the oil, the amount of free lipids in the seed-processing products naturally falls sharply, while the amount of bound and strongly-bound lipids changes only slightly. Some increase in the amount of free lipids is explained by the fact that in the moist heat treatment of (I) the lipids interact with the protein molecules [2].

The decrease in the amount of free gossypol is also explained by its capacity for taking part in a chemical interaction with many components of cotton seeds.

The free and bound lipids isolated from (I)-(IV) were analyzed as described in [3]. The neutral lipids from all the samples contained the following set of classes: traces of hydrocarbons, sterol esters, triacylglycerols, epoxyacylglycerols, free fatty acids (FFAs), free sterols, diacylglycerols, and traces of monoacylglycerols. The polar lipids isolated from the free lipids of all the samples investigated contained glyco- and phospholipids. These were monogalactosyldiglycerides, digalactosyldiglycerides, sterol glycosides, phosphatidylcholine, phosphatidylinositol, phosphatidylethanolamine, and

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TABLE 1. Fatty Acid Compositions of the Lipids of Cottonseed Processing Products

Sample	Lipids	Fatty acid, %								
		C _{14:0}	C _{16:0}	C _{16:1}	C _{18:0}	C _{18:1}	C _{18:2}	C _{18:3}	S	U
I	F	0,5	22,7	1,4	3,4	18,5	53,5	Tr.	26,6	73,4
	B	0,4	22,8	0,2	3,8	26,8	45,9	Tr.	27,0	72,9
	SB	1,2	20,8	1,8	4,8	20,8	39,7	1,9	35,8	64,2
II	F	0,9	24,5	1,7	2,4	18,3	52,2	Tr.	27,8	72,2
	B	0,3	26,8	2,6	3,1	25,5	41,7	Tr.	30,2	69,8
	SB	1,1	30,0	2,4	4,6	18,0	42,2	1,7	35,7	64,3
III	F	0,6	23,2	1,3	2,8	20,7	51,4	Tr.	26,6	73,4
	B	0,8	25,4	0,8	5,6	20,1	47,3	Tr.	31,8	68,2
	SB	1,5	30,4	2,1	5,0	15,0	43,2	2,8	33,9	63,1
IV	F	0,6	24,5	1,9	2,8	19,0	51,2	Tr.	27,9	72,1
	B	0,6	27,3	0,5	5,2	18,6	47,8	Tr.	33,1	66,9
	SB	1,2	30,8	2,3	4,2	14,2	43,8	3,5	36,2	63,8

TABLE 2. Acid Numbers and Phospholipid Content in Free and Bound Lipid Samples I-IV

Sample	Acid number of lipids, mg KOH/g		Phospholipids, % P ₂ O ₃	
	free	bound	free	bound
I	5,2	30,2	0,16	4,57
II	7,4	38,6	0,10	4,44
III	7,6	41,5	0,15	3,85
IV	10,5	49,9	0,27	5,48

lysophosphatides. The individual classes of lipids were identified by specific reagents for the corresponding groups of substances, as in [3].

The compositions of the fatty acids of the free, bound, and strongly-bound lipids were determined by the GLC method (Table 1).

It follows from the analytical results that the qualitative sets of acids of the lipids of all the samples were the same but the quantitative amounts of the individual acids showed an increase in the level of saturated acids from the free lipids to the strongly bound lipids, mainly due to palmitic acid, and the proportion of unsaturated acids decreased correspondingly. In contrast to other lipids, the strongly bound lipids contained linolenic acid, the amount of which increased from the crushed seeds to the meal.

The acid numbers of the free and bound lipids rose from (I) to (IV) (Table 2), i.e., the amount of FAAs in the free lipids was 5-6 times greater than in the bound lipids.

On extraction with chloroform-methanol, the phospholipids passed almost completely into the bound lipids.

EXPERIMENTAL

The free lipids were extracted with hexane and the bound lipids with chloroform-methanol in a Soxhlet apparatus. After the elimination of the free and bound lipids, the strongly-bound lipids were extracted by a standard method [4]. The compositions of the fatty acids were determined by the GLC of their methyl esters on a Chrom-4 instrument using a column filled with Chromaton N-AW DMS impregnated with 15% of Reoplex 400, at a temperature of 192°C and a rate of flow of helium of 100 ml/min.

SUMMARY

The compositions of the free, bound, and strongly-bound lipids in cotton seed processing products - crushed seeds, pulp, husks, and meal - have been studied.

It has been shown that the bound and strongly-bound lipids differ from the free lipids by a higher level of unsaturated acids. The acid numbers of the bound lipids were 5-6 times higher than those of the free lipids.

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X-RAY STRUCTURAL INVESTIGATION OF SESQUITERPENE ESTERS FROM PLANTS OF THE GENUS *Ferula*.

V. STRUCTURE AND STEREOCHEMISTRY OF FERTICIN

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An x-ray structural investigation has been made of the sesquiterpene ester ferticin: diffractometer, $\text{CuK}\alpha$ radiation, 1037 reflections, direct method, R factor 0.105. The spatial structure of the molecule has been determined.

The chromatographic separation on silica gel of the total neutral components of the roots of *Ferula tenuisecta* Korov. has yielded a compound $\text{C}_{20}\text{H}_{32}\text{O}_4$, mp 109-110°C (decomp.) which has been called ferticin (I).

An analysis of spectral characteristics permitted the conclusion that ferticin was an ester of angelic acid and a carotane alcohol containing a carbonyl group in a six-membered ring and two tertiary hydroxy groups (one of them being esterified).

The dehydration of ferticin with thionyl chloride led to a compound $\text{C}_{20}\text{H}_{30}\text{O}_3$ (II) in the PMR spectrum of which there were only slight changes in the chemical shift of the signals present in the spectrum of the initial compound, while in the IR spectrum the absorption band of the hydroxy group had disappeared. The absence from the PMR spectrum of (II) of the signals of olefinic protons showed that the double bond formed was fully substituted. This was possible only if the hydroxy group were located at C_5 or C_4 of the carotane skeleton. The latter position was excluded on the basis of the mass spectrum of ferticin which lacked the peak of an ion with m/z ($M - 43$)⁺ [1, 2]. Consequently, the second hydroxy group in ferticin, esterified with angelic acid, may be located at C_8 .

The value of the chemical shift of the angular methyl group and also the absence of conjugation with the carbonyl group in the dehydro derivative of ferticin showed the possibility of only two positions in the six-membered ring for the keto group - C_7 or C_9 . The choice of the alternative position for the carbonyl group and the establishment of the stereochemistry was made with the aid of x-ray structural analysis. The results of the investigation performed showed that ferticin has the following structure and stereochemistry (I) (see scheme on following page).

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