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STUDY OF THE LIGNIN OF THE COTTON PLANT OF VARIETY C-4880 BY THE METHOD OF ACIDOLYTIC CLEAVAGE

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Continuing a study of the lignin of cotton plants of the variety C-4880 [1], we have used the method of acidolytic cleavage of the lignin. The comminuted (0.25 mm) mature stems of the cotton plant were extracted with ethanol–benzene (1:2) and with hot water. Then the residue was subjected to the acidolysis reaction by Adler's method [2]. After the end of the reaction, the solution was brought to pH 3 with a 0.4 N solution of $NaHCO_3$ and the low-molecular-mass fraction of the acidolysis products was extracted with chloroform. The yield of the products of acidolytic cleavage was 7.6% of the initial material (30% on the Komarov lignin). In order to facilitate the identification of the components of the low-molecular-mass products with the aid of GLC, the products of the acidolysis reaction were reduced with the use of Raney nickel and lithium tetrahydroaluminate.

The reduction of the total acidolysis products with Raney nickel was carried out in the following way: Raney nickel catalyst freshly prepared from 5 g of nickel–aluminum alloy was added to a solution of 2.28 g of the total acidolysis products in 30 ml of 8% NaOH. Then, with constant stirring, the reaction mixture was boiled for 8 h under a current of nitrogen, after which it was acidified to pH 8, and the low-molecular-mass products were extracted with ether; the extract was evaporated and the residue was dried in vacuum over P_2O_5. The yield of reduced products was 0.66% of the initial raw material (3.19% on the Komarov lignin).

The reduction of the low-molecular-mass acidolysis products by lithium tetrahydroaluminate was performed in the following way: 4.5 g of lithium tetrahydroaluminate was suspended in absolute dioxane in a three-necked flask fitted with a condenser plus calcium chloride tube, a dropping funnel, and a stirrer, and, with constant stirring, 1 g of the total acidolysis products, dissolved in 10 ml of absolute dioxane, was added over 1 h. Stirring was continued for 1.5 h, and then, with ice cooling, a mixture of 10 ml of distilled water and 15 ml of glacial acetic acid was added. The solid matter was separated off by centrifugation, and the pH of the reaction mixture was brought to 6-7 with 5% NaOH solution, and it was extracted with ether. The extract was evaporated, and the residue was dried in vacuum over P_2O_5. The yield of lithium-tetrahydroaluminate-reduced material from acidolytic cleavage was 2.0% of the initial raw material (9.2% on the Komarov lignin).

The compositions of the products obtained were investigated by the GLC method on a Chrom 41 instrument with a stainless steel column (0.3 × 370 cm) filled with 15% of Apiezon on Chromaton NAW-DMCS (0.200-0.250 mm) at a rate of flow of carrier gas (helium) of 40 ml/min and a thermostat temperature of 205°C.

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The following substances were identified in the GLC investigation of the products obtained:

Substance	Percentage of the total after reduction with Raney nickel	Percentage of the total after reduction with $LiAlH_4$
1. Phenol	1.80	5.24
2. Guaiacol	31.10	5.03
3. p-Hydroxyphenylethane	2.50	5.11
4. p-Hydroxypropane	--	2.62
5. Guaiacylethane	10.98	5.55
6. Guaiacylpropane	1.40	4.40
7. 1-Guaiacylethanol	--	33.50
8. 1-Guaiacylpropan-1-ol	21.70	4.19
9. Syringylpropane	30.40	32.70
10. 1-Guaiacylpropan-3-ol	2--	1.57
Ratio of p-coumaryl	0.06	0.24
guaiacyl	1.00	1.00
syringyl	0.47	0.60

The results of acidolytic cleavage confirmed that the structural units of the lignin of cotton variety C-4880, as of other cotton plant varieties, are guaiacyl, syringyl, and p-coumaryl.

Judging from the yield of reduced products of acidolytic cleavage, it may be assumed that on the reduction of these products with Raney nickel it is possible that, together with the reduction of the low-molecular-mass products, under the influence of the aqueous medium secondary condensation processes took place, because of which the yield of the products of reduction with Raney nickel is low in comparison with the yield after reduction by lithium tetrahydroaluminate.

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