PREPARATION OF A NITROGEN-CONTAINING DERIVATIVE OF LIGNIN

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As is well known, some lignin derivatives are widely used in the well-drilling industry [1, 2], and others are recommended as plant-growth stimulators and herbicides [3-8]. To widen the field of use of hydrolysis lignin we have prepared a number of its derivatives, some of which are undergoing field trials in cotton-growing as biologically active substances.

The starting material – lignin from cotton pods of the Yangi-Yul' biochemical factory – after purification had the following indices (%): C 54.5; H 6.57; OH 4.52; OCH_3 5.4; N absent; moisture content 5.84; ash content 1.75; content of Komarov lignin 64.15.

In studying the reaction of hydrolysis lignin with urea, the following different solvents were tested: dichloroethane, ethanol, n-butanol, dioxane, and benzene. The reaction was performed at the boiling point of the particular solvent, but the initial lignin was recovered unchanged.

When dimethylformamide was used, a product of the interaction of lignin with urea (PLU) was obtained. The optimum conditions for the reaction are (Table 1) a ratio of the reactants of 1:1 and heating for 7 h.

The use of sulfuric and hydrochloric acids, and also of alkali, as catalysts did not lead to an increase in the amount of nitrogen in the PLU (the nitrogen content did not exceed 1.7%).

The reaction of lignin with urea formed a PLU, while when lignin was heated in dimethylformamide in the absence of urea (under similar conditions) no nitrogen-containing product was formed. The PLU was insoluble in water, but it was more soluble than the initial lignin in dilute solutions of caustic alkalis. Analytical results (%): C 54.82; H 6.75; N 3.25; OH 5.4; OCH_3 2.96.

The substance PLU is probably obtained by the addition of urea to the lignin through the carbonyl groups of the latter, since in the IR spectrum of the material the intensity of the carbonyl-group absorption band (1700 cm^{-1}) has greatly decreased in comparison with that of the initial lignin. The intensity of the absorption band of OH groups (3300 cm^{-1}) and that of the stretching vibrations of NH groups have also increased sharply. The presence of the latter in the PLU is confirmed by an absorption band in the 1640 cm^{-1} region. The fraction of the PLU that had dissolved in the dimethylformamide was freed from unchanged urea by treating the dry residue with hot water. This fraction is being studied.

TABLE 1

Ratio of lignin and urea	Time, hours	Product (% of the initial lignin)		Nitrogen content in the PLU, %
		insoluble	soluble	
2:1	1	90	7,5	1,74
2:1	2	85	12,0	2,28
2:1	4	90	9,0	2,16
2:1	6	90,5	9,3	3,12
1:1	5	83,5	20,0	3,04
1:1	7	100	20,0	3,25
1:1	7,5	100	17,0	3,11

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The results of a test of the PLU material as a growth stimulator for the cotton plant in vegetation experiments showed that the crop increased by 8%.

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