

THE REACTION OF THE HYDROLYSIS LIGNIN
OF SUNFLOWER HUSKS WITH AMMONIA

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It has been shown previously [1, 2] that in the reaction of lignin with gaseous ammonia the maximum amount of ammonia introduced is about 1%. The action of liquid ammonia and its aqueous solutions on lignin has been described in detail by various workers [3-5] with the statement that the process of "amination" is irreversible.

In the present paper we have the results of a study of the influence of the temperature, the pressure, and the time on the maximum yield of nitrogen-containing derivatives of hydrolysis lignin (I) under the action of an aqueous solution of ammonia.

Hydrolysis lignin of sunflower husks from the Zaporozhe hydrolysis-yeast factory was treated with 25% aqueous ammonia solution in an autoclave at temperatures of 20, 50, 100, 150, 200, and 230 ± 2°C (ratio 1 : 3). Samples were taken every hour during the synthesis. The reaction products were washed with water to neutrality and dried to constant weight at 105°C. The nitrogen contents were determined by the Kjeldahl method. The maximum amount of nitrogen was added to the lignin molecule after 6 h at 230°C; it amounted to 2.4% (yield 84%).

The dependence of the amount of nitrogen introduced on the temperature and time is shown in Fig. 1a and b. In the experiments performed, the formation of two types of "aminated" lignin was observed: water-soluble (II) with a yield of 15-20%, and water-insoluble (IIa) with a yield of 80-85%.

Preliminary investigations by TLC [Al₂O₃, activity grade II, plate dimensions 20×9 cm, thickness of the layer of sorbent 0.5 mm, propanol-butanol-water (6 : 1 : 2) system] showed that substance (II) differed from (IIa). Thus, the examination of the chromatogram of (II) in UV light showed the formation of four substances with R_f 0.83, 0.76, 0.54, and 0.33, and two substances were revealed with iodine vapor (R_f 0.58 and 0.17, while in the case of (IIa) only one substance with R_f 0.31 was detected; the bulk of the product remained at the start.

No definite products were identified with the available reference materials.

The most effective conditions for the reaction of hydrolysis lignin from sunflower husks with ammonia have been found: temperature 230°C, time 6 h, 25% aqueous ammonia.

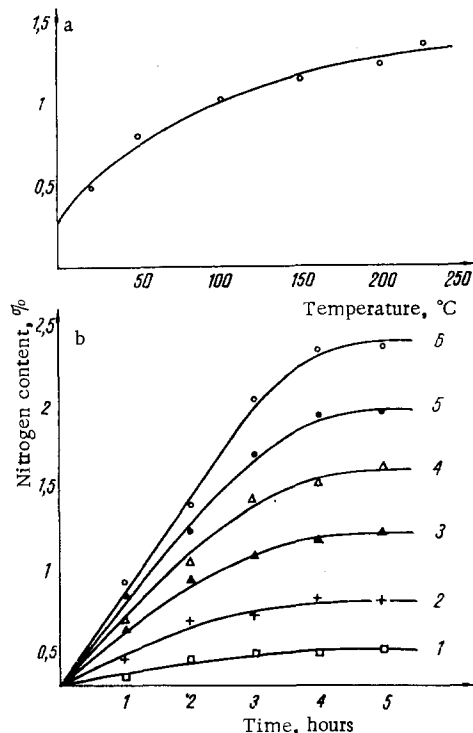


Fig. 1. Dependence of the amount of nitrogen introduced into hydrolysis lignin on the temperature (a) and the time of the reaction (b): 1) at 20°C; 2) at 50°C; 3) at 100°C; 4) at 150°C; 5) at 200°C; 6) at 230°C.

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