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ISOLATION AND STRUCTURE DETERMINATION OF BANANA PSEUDOSTEM BASED URO-LIGNIN

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ABSTRACT

Lignin was isolated from wet Banana pseudostem with 2% NaOH solution by boiling. The isolated lignin was treated with urea and called uro-lignin. IR, Mass and NMR spectra of urolignin were taken to determine its molecular structure.

INTRODUCTION

Lignin is the principal constituent of the woody substance of plant materials (1) . The specific nature of lignin polymers vary with wood source. The properties and composition of each lignin depend on the source and method of isolation. About 30% of woody materials consist of lignin (2). There are mainly two methods, acid and alkali, are used for the isolation of lignin form different sources. In both the cases, lignin obtained as a condensed form. Water insoluble lignin can be obtained by treatment with 72% H_2SO_4 or by saturating the powder wood with a mixture of acetone and ethyl alcohol and treating the product with conc. HCl (3). Water soluble lignin are obtained by digestion of wood in aqueous alkali and alkaline bisulphates (4) and are separated from cellulose by digesting wood chips at 130 – 150^oC with an aqueous solution containing calcium and magnesium hydrogen sulphates (5). Hence, the object of this work is to isolate lignin from wet banana pseudostem and also to determine its probable molecular structure.

EXPERIMENTAL

Isolation of lignin

One kg banana pseudostem was taken in 5 ltr stainless steel beaker containing 3 ltr of 2% of NaOH solution and boiled for 4 hours with continuous stirring. During boiling the volume of the mixture was kept constant by adding soft water and kept at rest for 24 hours. The residual banana pseudostem (cellulosic fibre) then separated with cloth filter. The volume of solution was reduced to 2 ltr by heating and then cooled. It was then acidified by adding 50 ml conc. HCl and the lignin emulsion was obtained. Then 250 g urea was added with it and boiled. It was then cooled for 24 hours and then needle shaped crystals were formed and those were separated with cloth filter and washed with rectified spirit. The crystal uro-lignin was dried in an oven at 40ºC and the yield was 500 g.

Melting point of uro-lignin

A Richard micro melting point apparatus was used for recording the melting point. Care was taken to ensure steady heating. The melting point of uro-lignin was 210-212ºC.

Functional group test of uro-lignin

The functional groups of the uro-lignin were tested and found to be phenolic, tertiary and primary amines, carbonyl and ether.

IR spectra

IR spectra of the sample were taken at HEJ Research Institute, Karachi, Pakistan. Infrared spectra were recorded on a PYE-UNICAM SP3 IR spectrophotometer. Spectra were taken in KBr pellets. The absorption bands in the spectra were found to be due to the presence of aromatic rings, saturated aliphatic chains, phenolic groups and small amounts of carbonyl groups in the sample.

Proton NMR Spectra

The proton NMR spectra were recorded on a Bruker AM 300 FT NMR, AM 400 FT NMR and AM 500 FT NMR spectrometers using TMS (Trimethylsilane) as internal standard. In the spectra of the sample a broad absorption band at 7-7.5 ppm was found. This was probably due to the presence of aromatic protons. A band was observed at 2.54 ppm due to the presence of saturated aliphatic chains. The absorption band 9.5 ppm was probably due to the presence of hydroxyl protons and another pick at 3.5 ppm was probably due to the presence of alcoholic protons.

Mass Spectra

The mass spectra were registered on a Varian-MAT 112S and Finnigan MAT-112 and 312A double-focusing Mass spectrometer connected to DEC PDP 11/34 and IBM-AT compatible PC based system, respectively. Electron impact (EI), Peak matching and Fast Atom Bombardment (FAB) experiments were performed on a MAT-312A or a Joel-JMS HX-110 mass spectrometers. The mass spectra showed maximum at 794 and minimum at 65. The other fragmentation patterns were 600, 325, 200, 125, 200 and 110.

RESULTS AND DISCUSSION

From the study of IR, Proton NMR, Mass Spectra, different functional group tests and nitrogen contains estimation the structure of the uro-lignin may be as follows:

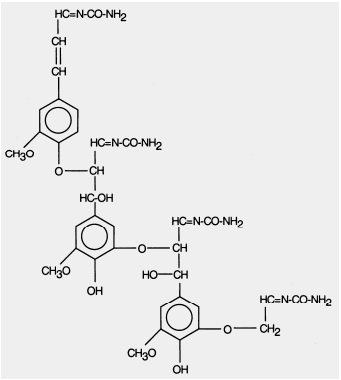

Fig.1. Probable structure of Uro-lignin of banana pseudostem

CONCLUSION

Low molecular weight uro-lignin has been found due to the alkali degradation of lignin from banana pseudostem. During the isolation of lignin with alkali solution, the carbonyl group developed may be attracted by urea to form the structure shown in figure-I.

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