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THE LIGNINS OF RIPE RICE-PLANT STEMS AND OF RICE HUSKS

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UDC 547.992:002.61

Studying the structure of lignin, we have performed the nitrobenzene oxidation and cleavage with sodium and liquid ammonia of the natural and the dioxane lignins of rice husks and ripe stems of rice plants. The isolation, characterization, and oxidation of the DLA (dioxane lignin) of rice husks has been described previously [1]. When the DLA of ripe rice-plant stems was oxidized, 44% of products were obtained, of which the following compounds were identified (in % on the total): p-hydroxybenzaldehyde, 17.08; p-hydroxyacetophenone, 2.1; guaiacol, 2.71; vanillin, 33.9; acetoguaiacone, 1.45; syringaldehyde 14.08. The ratio of p-coumaryl, guaiacyl, and syringyl structures was 0.51:1:0.38.

To determine the nature of the propane chains of the phenylpropane structural units of the lignin, we performed the reductive degradation with metallic sodium and liquid ammonia of the natural and DLA lignins of rice husks and of ripe rice-plant stems as described previously [2]. The total yield of products amounted to 26% for the DLA of the ripe stems, 24.8% for the DLA of the rice husks, 12.5% for the ripe stems, and 11.5% of the rice husks of the DLA and the Komarov lignins, respectively. Of the total materials at pH 8 the following products of cleavage by sodium and liquid ammonia were identified by the GLC method on a Chrom-4 instrument (4% on the total).

Substance	Rice husks	Rice husk DLA	Ripe rice stems	DLA of ripe rice stems
p-Hydroxyphenylethane	1.66	0.41	11.07	5.0
p-Hydroxyphenylpropane	13.64	23.28	1.29	10.47
1-(p-Hydroxyphenylpropan)-1-ol	—	1.3	0.93	—
Guaiacol	—	6.33	0.37	—
Vanillin	7.40	6.27	2.00	13.12
Guaiacylethane	—	2.17	0.74	—
Guaiacylpropane	2.85	14.0	4.64	5.72
1-Guaicylpropan-1-ol	5.54	—	—	2.90
3-Guaicylpropan-3-ol	—	5.32	2.80	—
Syringylpropane	9.74	27.5	17.33	24.26

Thus, by the nitrobenzene oxidation and reductive degradation with sodium and liquid ammonia of natural and dioxane lignins from rice husks and the ripe stems of rice plants we have shown the presence of three structural units of lignin — p-coumaryl, guaiacyl, and syringyl — in ratios of 0.73:1:0.79 for the rice husks DLA and 0.71:1:1.12 for the ripe rice stem DLA. The presence of a number of phenols in the products of cleavage with sodium and liquid ammonia shows that the initial lignins contain phenylpropane structural units

Institute of the Chemistry of Plant Substances, Academy of Sciences of the Uzbek SSR, Tashkent. Translated from Khimiya Prirodnykh Soedinenii, No. 6, pp. 794-795, November-December, 1982. Original article submitted June 22, 1982.

0009-3130/82/1806- 0765$07.50

with free OH groups in the α and γ positions of the side chain.

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COMPONENTS OF *Gomphocarpus fruticosus*

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UDC 547.918

The preparation "Gomfotin," which is close in its pharmacological properties to strophanthin, is obtained from the epigeal mass of *Gomphocarpus fructicosus* (L) R. Br. The main active agents of this preparation are the cardiac glycosides that have been obtained previously from the seeds [1] and leaves [2] of *G. fruticosus*. One of the glycosides has been synthesized independently [3]. The other components of the plants have been studied.

We have investigated the leaves, stems, roots, and follicles of *G. fruticosus* grown on an experimental plot of the botanical garden of the Academy of Sciences of the Moldavian SSR, the material for analysis being taken in the fruit-bearing phase. The amounts of alkaloids in the various organs of the plant were determined by Van'kovskii's method [4]. It was established that the roots contained about 0.5%, the valves of the seeds 0.5%, and the leaves 0.15% of alkaloids.

Preliminary analysis with the aid of spot reactions, and also by paper and thin-layer chromatography on silica gel in various solvent systems showed that the epigeal part contained flavonoids (Shinoda's test) and coumarins (visualization on paper or thin-layer chromatograms in UV light before and after spraying with a 5% solution of alkali), while the roots contained tanning substances (test with 1% ferric chloride solution) and flavonoids. Cardiac glycosides were detected only in the epigeal organs of the plant (Legal and Baljet tests). Quantitative analysis of the combined glycosides calculated as Gomfotin performed by the method of Tsarenko and Shraiber [5] showed that the largest amount of them was present in the leaves and follicles (about 1%) while the stems contained 0.06%, i.e., the epigeal mass of the plant is of interest for obtaining the combined glycosides.

The sum of the flavonoids was obtained by the following scheme: the comminuted air-dry material (epigeal part and roots separately) was extracted with chloroform and then with hot methanol. The methanolic extract was extracted to small volume, diluted with water, and treated with ethyl acetate. The organic layer was diluted with three volumes of chloroform. The yellow amorphous precipitate that deposited was separated off; it consisted of a mixture of flavonoid compounds — 9% in the epigeal part and 4% in the roots. On the basis of Bryant's test [6], aglycones and glycosides were detected in the mixture. Crystals with mp 189—190°C deposited from ethanol. Hydrolysis gave an aglycone with mp 302—305°C, identical according to PC with quercetin. Glucose and rhamnose were detected in the mother solution of the hydrolysate by paper chromatography.

Thus, the crystals with mp 189-190°C were rutin, as was confirmed by a direct comparison of its chromatographic mobility and UV spectrum with those of an authentic sample [7].

On studying the chloroform extract of the epigeal parts of the plant, in addition to carotenoids and chlorophyll, a substance was detected which was colored pink by the Sannié reagent. Column chromatography on silica gel in the hexane—ether (7:3) system yielded a compound with mp 135—136°C (ethanol), coinciding in chromatographic mobility with

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Translated from Khimiya Prirodnykh Soedinenii, No. 6, pp. 795-796, November-December, 1982. Original article submitted May 24, 1982.

0009-3130/82/1806-0766$07.50