

## STUDY OF THE COMPOSITION OF THE TOTAL PRODUCTS OF THE OXIDATION OF LIGNIN

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In industry, vanillin is obtained by the oxidation of liginosulfonate by atmospheric oxygen in the presence of the catalyst anthraquinone (AQ). In the oxidation process, in addition to vanillin, monomeric and dimeric lignin products with various structures are formed.

There is no information in the literature on the composition of the products of the oxidation of lignin in the presence of AQ. In view of this, we have studied the composition of the total oxidized material in the production of vanillin and have isolated the oxidation products.

The total products of the oxidation of liginosulfonates were obtained under the industrial conditions of the Syas' TsBK [Pulp and Paper Combine]. The pH of an alkaline solution of the total products was brought to 2, and they were separated by extraction with various solvents (hexane, ethyl acetate, chloroform). Their compositions were studied with the aid of TLC and GLC (Table 1, % in the mixture).

The results show that with an increase in the polarity of the solvents the sum of the extracted oxidation products increases and so does the number of substances present in it.

The total material remaining after the vanillin had been distilled off was separated by column chromatography. The sorbent used was silica gel passed through a sieve with 0.125-0.250 mm apertures. Chloroform was used for elution. The structures of the substances isolated were established with the aid of IR, NMR, and mass spectrometry.

Substance (1) —  $R_f$  0.23 (Silufol, chloroform), mp 206-208°C, yield 2.2%,  $M^+$  272, was identified as 4,4'-dihydroxy-3,3'-dimethoxystilbene [1].

Substance (2) —  $R_f$  0.39, mp 156-158°C, yield 0.001%,  $M^+$  302, was identified as formyl-4,4'-dihydroxy-3,3'-dimethoxystilbene.

Substance (3) —  $R_f$  0.18, mp 232-234°C,  $[\alpha]_D^{22}$  -63.83°, yield 1.5%,  $M^+$  356 was conidendrin [2].

TABLE 1

| Substance                     | Hexane total<br>(0.12%) | Ethyl acetate<br>total (0.64%) | Chloroform total<br>(0.92%) |
|-------------------------------|-------------------------|--------------------------------|-----------------------------|
| Guaiacol                      | 0.9                     | 2.79                           | 9.70                        |
| Vanillin                      | 72.6                    | 68.76                          | 49.39                       |
| Acetoguaiacone                | 26.5                    | 11.46                          | 9.34                        |
|                               | -                       | 3.35                           | 8.83                        |
| <i>p</i> -Hydroxybenzaldehyde |                         |                                |                             |
| Syringaldehyde                | -                       | -                              | 9.09                        |
| X <sub>1</sub>                | -                       | 6.04                           | 13.35                       |
| X <sub>2</sub>                | -                       | 7.65                           | 0.30                        |

### REFERENCES

1. J. Gierer and O. Lindeberg, Acta Chem. Scand., **B 34**, 161 (1980).
2. Eight Peak Index of Mass Spectra, Vol. 1, Part 1 (1983).

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