ALKYLATION OF PHENOLS BY METHANOL WITHOUT CATALYST

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The alkylation of phenols by methanol has required the presence of catalysts [1]. We have discovered that methanol in the supercritical state is an agent for the alkylation of phenols without the use of catalysts.

In typical experiments, a 10:1 mixture of methanol and phenol was introduced into a 50-ml steel autoclave and maintained for 60 min at the reaction temperature. The major reaction products were o-cresol and 2,6-xylenol as determined by gas—liquid chromatographic analysis. The phenol conversion is strongly dependent on the temperature and pressure. Thus, the conversion of phenol at 410°C and 20 MPa is 4 mole %, while it rises to 70 mole % at 460°C and 20 MPa. An increase in the pressure from 5 to 38 MPa at 460°C increases the phenol conversion from 40% to 83%.

Increases in the temperature, pressure, and contact time lead to more extensive alkylation of the aromatic ring. Thus, at 410°C and 20 MPa, 2,6-xylenol is found only in trace amounts in the reaction products. On the other hand, the yields of o-cresol and 2,6-xylenol are 40 and 20 mole %, respectively, at 460°C and the same pressure.

In order to eliminate the catalytic effect of the autoclave walls, runs were carried out in a glass ampul under the same conditions. The results for the experiments in the steel autoclave and glass ampule were identical.

LITERATURE CITED

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