EFFECT OF PREPARATION METHOD OF NICKEL-ZEOLITE CATALYSTS ON THEIR CATALYTIC PROPERTIES

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The effect of the carrier-gas (H\textsubscript{2}, N\textsubscript{2}, CO\textsubscript{2} and air) and the influence of additives (H\textsubscript{2}S and water vapors) in the activation of Ni- and Cr-containing zeolites on their catalytic activity have been studied in toluene disproportionation.

It is known that the activity of synthetic zeolite catalysts can be enhanced by the addition of metals via ion exchange and impregnation or by the addition of proper amounts of catalyst-promoting compounds into the reaction mixture /1/. The purpose of the present study was to elucidate the factors responsible for changes in the catalytic activity of nickel–zeolite catalysts in the disproportionation of toluene, by investigating the effect of the carrier gas (H\textsubscript{2}, N\textsubscript{2}, CO\textsubscript{2} and air) and the influence of additives (H\textsubscript{2}S and water vapors) introduced to the carrier gas at some moment of treatment.

EXPERIMENTAL

Catalyst samples were prepared on the basis of a calcium Y-type zeolite (degree of Ca\textsuperscript{2+} exchange 85%) with a molar ratio of SiO\textsubscript{2}/Al\textsubscript{2}O\textsubscript{3} = 5, modified by Ni\textsuperscript{2+} or by the mixture of Ni\textsuperscript{2+} and Cr\textsuperscript{3+} via successive ion exchange from 0.1M solutions of their nitrates. NiO/CaY was prepared via impregnation (precipitation of Ni(OH)\textsubscript{2}) of the Ca-form of the zeolite and its calcination at 110 °C. The composition of the samples studied is given in Table 1.
Table 1
Composition of catalysts

<table>
<thead>
<tr>
<th>Catalyst</th>
<th>Composition (wt. %)</th>
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<tbody>
<tr>
<td></td>
<td>Ni²⁺</td>
</tr>
<tr>
<td>NiCaY</td>
<td>2.9</td>
</tr>
<tr>
<td>CrNiCaY</td>
<td>2.8</td>
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<tr>
<td>NiO/CaY</td>
<td>2.9</td>
</tr>
</tbody>
</table>

Catalytic studies were carried out in a flow reactor at atmospheric pressure, 430 °C, volume gas flow rate of 1.0 h⁻¹ and the carrier gas to toluene ratio of 10. Pretreatment was performed in a stream of hydrogen, nitrogen, carbon monoxide and air at 450 °C for 2 h after gradual temperature elevation. H₂S was added to H₂ for 30 min at room temperature, then the reduction was carried out. Water vapors (24.4 g/m³) were added with H₂ during the gradual temperature elevation.

X-ray diffraction analysis of the samples in air-dried and reduced state (with or without additives of H₂S and water vapors) was made on a “Philips” spectrometer at U = 36 kV and with CuKα irradiation.

RESULTS AND DISCUSSION

The catalytic activity of NiCaY and CrNiCaY after their pretreatment in the presence of nitrogen, carbon monoxide or air is high but decreases rapidly (Fig. 1, Curves 2 and 4). In these cases the predominant process is cracking accompanied