A NEW PREPARATION TECHNIQUE OF CATALYSTS CHARACTERIZED BY SMALL METAL CRYSTALLITES

J. Barcicki, D. Nazimek, W. Grzegorczyk, T. Borowiecki, R. Frak and M. Pielach

Chemical Technology Department, Institute of Chemistry, Maria Curie-Skłodowska University, Nowotki 12, Lublin, Poland

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The present study reports a new and original technique for obtaining nickel catalysts characterized by small metal crystallites. The catalysts were prepared on γ-Al₂O₃. The total surface of the carrier was 37.5 m²/g. The preservation of the appropriate procedure and conditions of preparation allow a large expansion of active nickel surface (mean crystallite size 2.0–3.0 nm) in spite of the quite hard conditions of reduction (773 K and 3 h).

INTRODUCTION

A number of studies /e.g. 1–5/ reported supported contact preparations which allow the formation of small metal crystallites. Most of these techniques, however, are quite complicated and seem to have few chances to be eventually employed on an industrial scale. It is possible to obtain small nickel crystallites by precipitation but such contacts, in turn, are hardly reducible /3/.

The classical methods of carrier impregnation with solutions of the appropriate salts also yield small nickel crystallites but only at very low nickel contents in the catalysts (below 2 wt.%) /5/.

The present study reports a new and original technique of obtaining nickel catalysts characterized by small metal crystallites.
The total surface of the examined catalysts was determined by means of argon adsorption at the temperature of liquid nitrogen in a BET apparatus. Active surface and mean crystallite size were determined by hydrogen chemisorption at 295 K, at a pressure of 100 Torr. Nickel content in the catalysts was determined chemically with ethylenediaminetetraacetic acid (EDTA).

The catalysts were prepared on γ-Al₂O₃ (Peshiney) and the support contained such impurities as SiO₂ 0.08 wt.% Fe₂O₃ 0.03 wt.%, and sodium 0.6 wt.%. The total surface of the support was 37.5 m²/g. Experiments also employed disodium EDTA and nickel nitrate, analytical purity grade, both from POCh Gliwice. The catalysts were calcined in air in a muffle stove.

The carrier, γ-Al₂O₃, in the form of globules 5 mm in diameter, was dried at 393 K and then impregnated for 10 min with a solution of disodium EDTA at a given concentration at 343–353 K. The carrier was then separated from the solution of disodium EDTA and dried at 393 K for 2 h. The support was then impregnated with a solution of nickel nitrate at a concentration of 15% of Ni for a determined period of time (from 0.5 to 9 min). The impregnating solution was then removed.

The carrier with the deposited active phase was dried at 393 K for 1 h and then calcined at 673 K for 1 h in air.

The catalysts obtained were reduced with hydrogen at 773 K for 3 h.

Results of the experiments are listed in Table 1. The results of the experiments on three series of catalysts, whose preparations differed in respect to the EDTA concentration for the original impregnation and time periods of nickel nitrate impregnations, seem to reveal several characteristic dependences.

*The carrier was obtained by sintering of Peshiney’s support for 10 hrs at 1173 K.