

III. PREPARATION OF HYDROGENATION CATALYSTS.

I.G. FARBENINDUSTRIE A.G. - LUDWIGSHAFEN,
May 29th, 1947

Dr. Wimmer

In various reports on the German industry mention has been made of the application of a copper-chrome-silicagel catalyst for hydrogenation purposes. This type of catalyst has proved to be especially suitable for the hydrogenation of aldehydes to alcohols (aldol \rightarrow butol, Oxo-aldehydes \rightarrow Oxo-alcohols) and of esters to alcohols (fore-run fatty acids \rightarrow primary straight chain alcohols). There is, however, some confusion in the description of its preparation, which in some cases was given to proceed via impregnation of the nitrates ¹⁾ and in another instance was said to proceed via impregnation of an ammoniacal Cu-solution."

In order to clarify this point, the relevant type of catalyst was discussed with Dr. Wimmer and it therefrom became clear that I.G. had developed 2 catalysts, both consisting of 20% Cu - 0.7% Cr on silicagel, but which were obtained by different ways of preparation.

Catalyst B.B.E.

The silicagel used for the preparation was silicagel B of Oppau prepared by precipitation from sodium silicate solution through adding this solution to dilute sulphuric acid. Silicagel was used in granules of 2-6 mm size. It had relatively wide pores and contained only 0.01 - 0.02% Fe. The impregnating solution was obtained by dissolving 800 kg wet CuCO_3 (containing 50% water and 400 kg CuCO_3 corresponding to 235 kg Cu) in 1000 kg 25-% NH_4OH solution, whereupon 140 kg $(\text{NH}_4) \text{HCO}_3$ was added in order to improve the solubility. The solution, 25° Bé, contained 10.5 - 12% Cu. Per 100 kg. Cu 0.6 kg chromic acid

¹⁾ C.I.O.S. XXII-21: "Synthetic Rubber Plant
Chemische Werke - Hüls"

") B.I.O.S. FR 748 : "Manufacture of fatty acids by
oxidation of paraffins, hydrogenation of the fatty acids at
I.G. Ludwigshafen-Oppau"

was added. It is not possible to add more chromic acid in view of the risk of soluble CuCr double salts being precipitated.

The silicagel was placed on metal screens and was soaked with this solution at room temperature, whereupon the sieves were placed in a direr and kept at 100°C for 24 hours. This operation was thereafter repeated twice in order to yield finally a catalyst of the required composition.

The final catalyst was calcined at 250°C and sieved to 1.5 - 3 mm size.

The catalyst is usually reduced prior to use in the hydrogenation plant at temperatures of 150-200°C with hydrogen.

Catalyst No. 4711.

The same silicagel is being used for this catalyst. The impregnating solution in this case, however, is obtained by dissolving copper nitrate and chromic acid in water to a solution containing abt. 20% wt. Cu and 0.2% wt. Cr. The silicagel is impregnated only once at a temperature of 50-60°C, subsequently dried at 100-120°C and calcined at 450°C until evolution of NO₂ ceases. After sizing to 1.5 - 3 mm the catalyst is reduced with hydrogen at 400°C.

According to Dr. Wimmer general experience with I.G. had been that the B.B.E. - catalyst was slightly more active than 4711, but that the differences, if any, were very small.

On this occasion the preparation of another catalyst on silicagel base was also discussed:

Methanization catalyst A-1

This catalyst is prepared similar to 4711 by impregnating Oppau silicagel B with a solution containing nickel nitrate, copper nitrate and chromic acid at about 60°C to a final catalyst consisting of

15% Ni,
5% Cu,
1.5% Cr.

III-2

This catalyst is used for the purification of hydrogen under pressure by converting traces CO into CH₄.

Amsterdam, 19th December 1947,

III-3