PATENT SPECIFICATION

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PROVISIONAL SPECIFICATION

Improvements in or relating to the Preparation of Catalysts

We, SYNTHETIC OILS LIMITED, a British Company, of 31, East Street, Epsom, in the County of Surrey, and WILLIAM WHALLEY MYDDLETON, a British Subject, of 3, Woodlands Avenue, New Malden, in the County of Surrey, do hereby declare the nature of this invention to be as follows:—

This invention relates to the preparation of catalysts and has particular reference to the preparation of metallic catalysts intended for use in the synthesis of hydrocarbon oils from gaseous mixtures of hydrogen and carbon monoxide.

It has already been proposed to protect such catalysts from atmospheric oxidation during the interval between their preparation by reduction and the starting of the synthesis by dowsing them in hydrocarbon oils or waxes, the products of a previous synthesis being preferred in order to ensure the absence of catalyst poisons. An additional advantage of this treatment is that during the initial stages of the synthesis, when the activity of the catalyst would otherwise be at a maximum, the activity of the catalyst is reduced by the protective covering of oils or waxes, which is only gradually removed by the reaction gases; the reaction thus proceeds in a more uniform manner.

According to the present invention the above advantages are secured to an enhanced extent by replacing the hydrocarbon oils or waxes used for dowsing the catalyst by a liquid which is strongly adsorbed by the catalyst surface, thus giving better protection against oxidation, and having a sharp boiling point, thus ensuring its smooth and rapid removal by the reaction gases during the initial stages of the synthesis. To ensure the degree of adsorption desired, the substance employed should contain hydroxyl and/or carbonyl groups. We have obtained particularly satisfactory results by dowsing the catalyst in cyclohexanol, methyl cyclohexanol, cyclo hexanone, and methyl cyclohexanone produced by catalytic synthesis, but the invention is not limited to the use of these particular substances.

Dated this 6th day of August, 1938.

A. A. THORNTON,
Chartered Patent Agents,
7, Essex Street, Strand, London, W.C.2,
For the Applicants.

COMPLETE SPECIFICATION

Improvements in or relating to the Preparation of Catalysts

We, SYNTHETIC OILS LIMITED, a British Company, of 31, East Street, Epsom, in the County of Surrey, and WILLIAM WHALLEY MYDDLETON, a British Subject, of 3, Woodlands Avenue, New Malden, in the County of Surrey, do hereby declare the nature of this invention and in what manner the same is to be performed, to be particularly described and ascertained in and by the following statement:—

This invention relates to the preparation of catalysts and has particular reference to the preparation of metallic catalysts intended for use in the synthesis of hydrocarbon oils from gaseous mixtures of hydrogen and carbon monoxide.

It has already been proposed to protect such catalysts from atmospheric oxidation during the interval between their preparation by reduction and the starting of the synthesis by dowsing them in hydrocarbon oils or waxes, the products of a previous synthesis being preferred in order to ensure the absence of catalyst poisons. An additional advantage of this treatment is that during the initial stages of the synthesis, when the activity of the catalyst would otherwise be at a maximum, the activity of the catalyst is reduced by the protective covering of oils or waxes, which is only gradually removed by the reaction gases; the reaction thus proceeds in a more uniform manner.

According to the present invention the above advantages are secured to an enhanced extent by replacing the hydrocarbon oils or waxes used for dowsing the catalyst by a liquid which is strongly adsorbed by the catalyst surface, thus giving better protection against oxidation, and

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preferably having a boiling point within specified limits, thus ensuring its smooth and rapid removal by the reaction gases during the initial stages of the synthesis. To ensure the degree of adsorption desired, the substance employed should contain hydroxyl and/or carbonyl groups, and in this connection a synthetic hydrocarbon oil to which a proportion in the region of 10% of the organic hydroxyl and/or carbonyl liquid has been added will be found to be effective.

The invention consists broadly of a method of preparing a catalyst for use in the synthesis of hydrocarbon oils according to which the catalyst, after reduction according to known practice, is dowsed in a liquid containing or composed of organic bodies in which hydroxyl and/or carbonyl groups are present.

An organic liquid should be chosen which not only contains hydroxyl and/or carbonyl groups but which in addition boils at a temperature preferably within 100° C. below, and a few degrees above, the temperature at which the catalyst is to be used. We have obtained particularly satisfactory results by dowsing the catalyst in cyclohexonal, methyl cyclohexonal, cyclohexanone, and methyl cyclohexanone produced by catalytic synthesis, but the invention is not limited to the use of these particular substances.

In preparing catalysts according to the invention it has been found convenient to cool the catalyst upon reduction to a temperature below 100° C. and then to flood the reduction chamber with the liquid.

After a few minutes contact the liquid is drained off and the process of removing surplus liquid is effected by circulating a gas such as nitrogen or hydrogen downwards through the mass of catalyst.

Alternatively the reduced catalyst can be dropped into cold liquid and thereafter drained.

The protected catalyst can be loaded into the reaction chamber and the liquid removed by heating to the boiling point of the liquid either in an atmosphere of hydrogen or an inert gas or in the gas which is to take part in the subsequent catalytic gas reaction.

Having now particularly described and ascertained the nature of our said invention and in what manner the same is to be performed, we declare that what we claim is:—

1. A method of preparing a catalyst for use in the synthesis of hydrocarbon oils according to which the catalyst, after reduction according to known practice, is dowsed in a liquid containing or composed of organic bodies in which hydroxyl and/or carbonyl groups are present.

2. A method of preparing a catalyst for use in the synthesis of hydrocarbon oils according to claim 1, according to which the selected organic liquid boils at a temperature within 100° C. below and a few degrees C. above the temperature at which the catalyst is to be used.

3. A method of preparing a catalyst for use in the synthesis of hydrocarbon oils according to claim 1 or 2, according to which the catalyst, after reduction, is cooled to a temperature below 100° C. before the dowsing operation.

4. A method of preparing a catalyst for use in the synthesis of hydrocarbon oils according to claim 1, 2, or 3, according to which after the dowsing operation surplus liquid is removed by subjecting the dowsed catalyst to the action of a downward flow of hydrogen or an inert gas.

5. A method according to claim 1, 2 or 3, according to which the dowsed catalyst is loaded into the reaction chamber and surplus liquid removed by heating to a temperature approaching the boiling point of the liquid used for dowsing or by heating continuously to the temperature at which the catalyst is to be used in an atmosphere of hydrogen or inert gas or gas which is to be used in the subsequent catalytic reaction.

6. The improved method of preparing catalysts for use in the synthesis of hydrocarbon oils substantially as described.

Dated this 4th day of August, 1939.

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