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PATENT SPECIFICATION

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PROVISIONAL SPECIFICATION

Improvements in or relating to the Reactivation of Catalysts

We, SYNTHETIC OILS LIMITED, a British Company, of 31, East Street, Epsom, in the county of Surrey, WILLIAM WHALLEY MYDDLETON, a British subject, of 3, Woodlands Avenue, New Malden, in the county of Surrey, and ALFRED AUGUST AICHER, a British subject, of 22, Holland Avenue, Wimbledon, in the county of Surrey, do hereby declare the nature of this invention to be as follows:—

This invention relates to the reactivation of the catalyst masses used in the synthetic production of hydrocarbons, and particularly in connection with the synthetic production of hydrocarbon oils from gaseous mixtures containing hydrogen and carbon monoxide.

The catalyst masses employed in synthetic reactions of this kind are subject to a progressive loss of efficiency which is due in a large measure to the accumulation on the surface of the particles of the catalyst, of paraffin waxes varying in their melting point range from about 50° C. to considerably over 100° C.

It has been proposed to maintain or restore the activity of the catalyst by periodically flushing the catalyst chamber with hydrogen, gases containing hydrogen or gases capable of yielding hydrogen in the conditions of their employment. The effect achieved by this treatment appears to be due, as far as the dewaxing action is concerned, to the mechanical scouring action exerted by the gas stream, aided by a tendency for the wax to become detached from the surface of the catalyst particles due to the affinity of the catalyst for hydrogen and its tendency to absorb hydrogen at the surface.

It has also been proposed to remove wax deposited upon the catalyst by a periodical interruption of the working of the plant and extraction of the wax with the aid of a solvent such as a hydrocarbon oil.

The former of these two proposed methods has the disadvantage that the reactivation and dewaxing achieved at each successive treatment becomes progressively less effective. This is shown by the fact that the drop in the working reaction temperature, in the given conditions of working, achieved by the reactivation treatment becomes less at each successive treatment, with the result that the reactivation has to be effected at progressively shorter intervals in order to keep within the required limits of the reaction temperature range. A limit is thus set to the useful life of a batch of catalyst mass handled in this way.

The second of the above-mentioned proposed methods of treatment has the disadvantage that the residual solvent oil left clinging to the catalyst particles after the treatment has to be washed off by flushing with lighter oils and/or inert gas before the synthesizing run can be resumed while the life of the catalyst mass is again limited owing to the fact that the flushing treatment with hydrocarbon oil serves merely to remove the wax without effecting any appreciable reactivation of the catalyst itself.

We have found that provided certain conditions are observed in the conduct of the synthesizing reaction, it is possible to reactivate the catalyst and to maintain its economic working efficiency substantially unimpaired over a protracted period, by flushing the catalyst chamber with hydrogen at intervals of from 7 to 10 days.

However, the periodical reactivation of the catalyst becomes progressively less effective at each treatment with hydrogen, owing to the fact that waxes of very high melting point do not become dislodged and eventually accumulate to such an extent that hydrogen is no longer capable of restoring the activity in a period of time which could be considered satisfactory.

In this case again, therefore, a limit appears to be set to the useful life of a batch of catalyst, and it is the purpose of the present invention to regenerate a catalyst mass when it has ceased to be capable of further reactivation economically by flushing with hydrogen or gas containing or yielding hydrogen.

According to the invention, this result is achieved by charging the hydrogen used for flushing out the wax and reactivating the catalyst, when this stage has been reached, with a vapour of a solvent capable of dissolving or mixing with

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waxes of higher melting point.

We have found that a suitable solvent is a fraction of the synthetic hydrocarbon product of reactions of the type with which the invention is concerned.

In a preferred mode of carrying out the invention hydrogen destined for use in reactivating the catalyst by flushing the catalyst chamber is bubbled through a tank containing the liquid hydrocarbon product and the tank is maintained at such a temperature that the desired fraction is vapourised and entrained by the hydrogen in a concentration found by trial to be the optimum for the purpose in view.

The vapour-laden hydrogen is preferably passed through the catalyst chamber from above downwards, and it is found that the accumulated wax, after absorbing a proportion of the vapourised solvent, becomes fluid and trickles through the catalyst mass, while the hydrogen dislodges any remainder of wax and effects reactivation of the catalyst, by adsorption at the surface thereof, or in other ways.

It will be clear that the temperature maintained during the process of charging the hydrogen with solvent vapour, and the consequent concentration of such vapour in the treating gas, will depend in any one instance on the working conditions of the reaction concerned and the nature of the wax contamination of the catalyst.

For the efficacy of the described method of reactivation it is desirable that the temperature of the catalyst chamber be maintained at a point above that at which the hydrocarbon vapours entrained by the hydrogen will be condensed in liquid form upon the catalyst.

It will be understood that the solvent vapour laden flushing gas may be prepared in other ways than that referred to above.

Dated this 11th day of October, 1943.
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Chartered Patent Agents,
7 Essex Street, Strand, London, W.C.2,
For the Applicants.

COMPLETE SPECIFICATION

Improvements in or relating to the Reactivation of Catalysts

We, SYNTHETIC OILS LIMITED, a British Company, of 31, East Street, Epsom, in the county of Surrey, WILLIAM WHALLEY MYDDLETON, a British subject, of 3, Woodlands Avenue, New Malden, in the county of Surrey, and ALFRED AUGUST AICHER, a British subject, of 22, Holland Avenue, Wimbledon, 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 ad ascertained in and by the following statement:—

This invention relates to the reactivation of the catalyst masses used in the synthetic production of hydrocarbons, and particularly in connection with the synthetic production of hydrocarbon oils from gaseous mixtures containing hydrogen and carbon monoxide.

The catalyst masses employed in synthetic reactions of this kind are subject to a progressive loss of efficiency which is due in a large measure to the accumulation on the surface of the particles of the catalyst, of paraffin waxes varying in their melting point range from about 50° C. to considerably over 100° C.

It has been proposed to maintain or restore the activity of the catalyst by periodically flushing the catalyst chamber with hydrogen, gases containing hydrogen or gases capable of yielding hydrogen in the conditions of their employment. The effect achieved by this treatment appears to be due, as far as the dewaxing action is concerned, to the mechanical scouring action exerted by the gas stream, aided by a tendency for the wax to become detached from the surface of the catalyst particles due to the affinity of the catalyst for hydrogen and its tendency to absorb hydrogen at the surface.

It has also been proposed to remove wax deposited upon the catalyst by a periodical interruption of the working of the plant and extraction of the wax with the aid of a solvent such as a hydrocarbon oil.

The former of these two proposed methods has the disadvantage that the reactivation and dewaxing achieved at each successive treatment becomes progressively less effective. This is shown by the fact that the drop in the working reaction temperature, in the given conditions of working, achieved by the reactivation treatment becomes less at each successive treatment, with the result that the reactivation has to be effected at progressively shorter intervals in order to keep within the required limits of the reaction temperature range. A limit is thus set to the useful life of a batch of catalyst mass handled in this way.

The second of the above-mentioned proposed methods of treatment has the disadvantage that the residual solvent oil left clinging to the catalyst particles after the treatment has to bewashed off by flushing with lighter oils and/or inert gas before the synthesizing run can be resumed, while the life of the catalyst mass is again limited owing to the fact that the flushing treatment with hydrocarbon oil serves merely to remove the wax without effecting any appreciable reactivation of the catalyst itself.

We have found that provided certain conditions are observed in the conduct of the synthesizing reaction, it is possible to reactivate the catalyst and to maintain its economic working efficiency substantially unimpaired over a protracted period, by flushing the catalyst chamber with hydrogen at intervals of from 7 to 10 days.

However, the periodical reactivation of the catalyst becomes progressively less effective at each treatment with hydrogen, owing to the fact that waxes of very high melting point do not become dislodged and eventually accumulate to such an extent that hydrogen is no longer capable of restoring the activity in a period of time which could be considered satisfactory.

In this case again, therefore, a limit appears to be set to the useful life of a batch of catalyst, and it is the purpose of the present invention to regenerate a catalyst mass when it has ceased to be capable of further reactivation economically by flushing with hydrogen or gas containing or yielding hydrogen.

According to the invention, this result is achieved by charging the hydrogen used for flushing out the wax and reactivating the catalyst, when this stage has been reached, with a vapour of a solvent capable of dissolving or mixing with the waxes

We have found that a suitable solvent is a fraction of the synthetic hydrocarbon product of reactions of the type with which the invention is concerned.

In a preferred mode of carrying out the invention hydrogen destined for use in reactivating the catalyst by flushing the catalyst chamber is bubbled through a tank containing the liquid hydrocarbon product and the tank is maintained at such a temperature that the desired fraction is vapourised and entrained by the hydrogen in a concentration found by trial to be the optimum for the purpose in view.

The vapour-laden hydrogen is preferably passed through the catalyst chamber from above downwards, and it is found that the accumulated wax, after absorbing a proportion of the vapourised solvent, becomes fluid and trickles through the catalyst mass, while the hydrogen dislodges any remainder of wax and effects reactivation of the catalyst, by adsorption at the surface thereof, or in other ways.

It will be clear that the temperature maintained during the process of charging the hydrogen with solvent vapour, and the consequent concentration of such vapour in the treating gas, will depend in any one instance on the working conditions of the reaction concerned and the nature of the wax contamination of the catalyst.

For the efficacy of the described method of reactivation it is desirable that the temperature of the catalyst chamber be maintained at a point above that at which the hydrocarbon vapours entrained by the hydrogen will be condensed in liquid form upon the catalyst.

It will be understood that the solvent vapour laden flushing gas may be prepared in other ways than that referred to above.

It should, perhaps, be mentioned that it has been suggested to reactivate catalysts in processes of the kind to which this invention belongs by passing over the catalyst at intervals vapours of organic liquids in which the conversion products are soluble under conditions that condensation of the vapours occur. Such a process, apart from not employing hydrogen in the reactivating process which is essential to the present invention differs radically from the present invention in that it involves cooling of the catalyst chamber to such a point that the vapour used condenses to a liquid which dissolves the wax and mechaically washes it away.

In the present method no cooling of the catalyst chamber is required, the process depending on the vapour being partly extracted by the wax so that the volatile hydrocarbon tends to compete with the wax at the catalyst surface for attachment at the active centres of the catalyst body. The wax is thus loosened and eventually detached and is replaced by hydrogen which reactivates the catalyst.

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. In processes for the synthetic production of hydrocarbon oils from the gaseous mixtures containing hydrogen and carbon monoxide, the method of reactivating the catalyst mass according to which the hydrogen used for flushing out the wax and reactivating the catalyst is charged with the vapour of a solvent capable of dissolving or mixing with waxes.

2. The method of reactivating catalysts according to claim 1, wherein the solvent vapour employed is a fraction of the hydrocarbon product obtained as a result of the synthetic reaction.

3. The method of reactivating catalysts according to claims 1 and 2, wherein the

hydrogen stream is charged with the solvent vapour by bubbling it through liquid hydrocarbon which latter is maintained at such a temperature that the desired fraction is vapourised by and entrained in the hydrogen.

4. The method of reactivating catalysts according to any of the preceding claims, according to which the vapour laden hydrogen is passed downwardly through the catalyst chamber.

5. The improved method of reactivating catalysts used in processes for the synthetic production of hydrocarbons substantially as described.

Dated this 11th day of October, 1944.

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