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12/19/2001 - 16:23:58

(11) CA 379299

(12) Patent:

(54) BENZINE CATALYST REGENERATION

(54) REGENERATION DE CATALYSEURS DE BENZINE

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(74)		
(45)		Jan. 31, 1939
(22)		
(43)		
(52)		252/I.E
(51)		N/A

(70) (Country): No

(30) (Country): None

(71) (Country): N/A
(71) (Country): Unknown

*** Note: Data on abstracts and claims is shown in the official language in which it was submitted.

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S P E C I F I C A T I O N

TO WHOM IT MAY CONCERN:

Be it known that we, Walter Feisst, of 99 Bahnstrasse, Oberhausen-Holten, Germany, Otto Roelen, of 30 Bruchsteg, Oberhausen-Holten, Germany and Walter Schuff, of 21 Erich Ficklerstrasse, Oberhausen-Holten, Germany, Chemists, having invented certain new and useful "Process for regenerating catalysts for benzine synthesis", do hereby declare that the following is a full, clear, and exact description of the same:-

It is known that the catalysts necessary for benzine synthesis from carbon monoxide and hydrogen lose in efficiency after a period of use. This reduction in activity is mainly due to the fact that high-molecular organic substances settle on the catalysts. The catalytic efficiency of such catalysts is successfully restored by methods of treatment by which the high-molecular organic deposits are removed from the catalysts. Thus, for example, the weakened catalysts may be treated with hydrogen or a suitable liquid at synthesis temperature. Such measures for the regeneration of catalysts can be frequently repeated, but in the end the high-molecular organic substances adhere so tenaciously to the catalysts that further use of the material is possible only by dissolving the contact substance and restoring it by again precipitating the catalytic metals. It has been found that if the spent contact substances be thus treated the deposits remaining in the contact substance increase the difficulty

of restoring the catalysts. Thus for example the filtration of the metal salt solution produced by decomposition with acid is rendered more difficult by the high-molecular paraffins contained in the spent catalysts. Further the re-precipitation of the catalytic metals is affected by the fatty acids and other oxygen-containing compounds which are produced from the paraffins on the dissolving of the catalysts in nitric acid, the compounds referred to preventing precipitation by the formation of complexes.

It has been found that all these difficulties are avoided if hydrogen or inert gases and vapours, such as nitrogen, and carbon dioxide, be passed over the spent catalyst at temperatures above 300°C . - preferably at temperatures of from 350 to 400°C . - before the catalyst has been dissolved in acids. This preliminary treatment removes the organic substances from the catalysts completely.

If it is desired first to recover by itself the high-melting hard paraffin that has been separated from the spent catalysts, it is advisable before carrying out the preliminary treatment to extract the catalysts with a suitable solvent or scouring agent. It is preferable to employ during the extraction temperatures that lie above the melting points of the hard paraffins. The preliminary extraction of the catalysts whose efficiency has been reduced has the further advantage that when, immediately after, the catalysts are treated with hydrogen there is a substantial saving of this gas.

The time needed for removing the organic deposits from the catalysts is dependent upon the temperature employed and the velocity of the gases or vapours passed over, so that by an increase of velocity and increase of the temperature of treatment the time needed for the preliminary treatment of the catalysts is shortened. It is possible completely to remove the deposited organic substances from the contact substance within half an hour by passing the gases over at temperatures of from 350 to 400°C. at a velocity of from 50 to 60 L per hour per sq. cm. This time can be reduced by further increasing the velocity and by suitably raising the temperature of the treatment still further.

The organic compounds are removed from the contact substance more rapidly with hydrogen than with nitrogen or carbon dioxide under otherwise similar conditions. In any case the working conditions may be such that it is preferable to use the inert gases or vapour before mentioned in place of hydrogen. Further experiments have shown that instead of using inert gases or vapour alone mixtures thereof with hydrogen may be employed. The use of water vapour or the addition of water vapour to the gases employed, has the added advantage that the pyrophoric properties of the contact substance are removed from the latter by the oxidising action of the water vapour, so that the contact substance can be brought into contact with the atmosphere without the danger of spontaneous combustion.

The present process may be employed in many different ways, the working conditions being always adjusted to prevailing practical requirements. For instance the contact substance may first be treated with hydrogen and thereupon with inert gases; finally the treatment with hydrogen or inert gases may be followed by treatment with water vapour in order to remove the pyrophoric properties from the catalyst from which the organic substances have previously been removed.

The following is an example of the carrying out of the invention:

A catalyst that has been used for benzine synthesis by reduction of carbon monoxide with hydrogen and which has been regenerated in the synthesis furnace very many times by treatment with hydrogen at synthesis temperature until its efficiency has finally departed, is treated with a rapidly flowing stream of hydrogen at 350°C. After two hours have elapsed the mass of catalyst has lost about 40% of its weight, paraffin and other organic substances having been withdrawn from the catalyst with the hydrogen, 80 to 85% of the organic substances removed from the catalyst in the form of a paraffin with a melting point of from 60 to 65°C. being obtained. The catalyst mass which under the hydrogen treatment has lost practically all organic substances is moistened with water after cooling, and dissolved in nitric acid. The catalytically active metals are precipitated by means of a soda solution from the metal salt solution thus obtained, after

purification by filtration. The washed and dried catalyst is then reduced with hydrogen and is again suitable for effecting synthesis.

We claim:

1. In a process for regenerating catalysts that have been used for benzine synthesis from mixtures of carbon monoxide and hydrogen by dissolving the catalytically active metals in acids and then again precipitating, treating the catalysts that have lost in efficiency at temperatures of 300°C. or over with gases or vapours which do not chemically react with the impurities in the catalysts prior to the dissolution of the catalysts in acid.
2. A process for regenerating catalysts according to claim 1, wherein hydrogen and/or inert gases is or are employed.
3. A process for regenerating catalysts according to claim 1, wherein water vapour is added to the gases employed.
4. A process according to claim 1, wherein the treatment with gases or vapours is carried out in stages in such manner that in the different stages different gases are used, the catalyst being treated in the final stage advantageously with water vapour or gases charged with water vapour.
5. A process according to claim 1, wherein the catalysts before being subjected to the gases or vapours are extracted with a suitable solvent for the partial removal of organic substances.