PATENT SPECIFICATION

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COMPLETE SPECIFICATION

Process for the Treatment of Spent Cobalt Catalysts

We, RUHRCHEMIE AKTIENGESELLSCHAFT, of Oberhausen-Holten, Germany, a Company organised and existing according to the Laws of the German State, 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:—

Although the catalysts that are produced by the precipitation of the catalytically active metals in the presence of Kieselguhr and that are employed for benzine synthesis from mixtures of carbon monoxide and hydrogen last for a very long time, they finally absorb during the synthesis large quantities of organic substances that do not volatilise readily, whereby their activity is gradually lowered, so that from time to time it becomes necessary to regenerate them by re-dissolving in acids and precipitating the catalytically active constituents.

It is common chemical practice to use concentrated nitric acid for dissolving cobalt catalysts in order to obtain cobalt solutions that are very highly concentrated.

A disadvantage of this method however is that the concentrated cobalt solution contains Kieselguhr in such a fine state of suspension that it is not possible to separate the Kieselguhr from the solution except by the use of very special and expensive means.

It has been found according to the invention that when a more dilute nitric acid is used the Kieselguhr that was contained in the catalyst settles after a short time and can then readily be removed from the cobalt solution. It has been found to be an advantage to employ an acid containing from 3—5% of free nitric acid.

It is inadvisable to use nitric acid of a strength greater than 20%. It is particularly advantageous to employ for the solution an acid that already contains in solution considerable quantities of cobalt nitrate. Such a dissolving liquor can be obtained for example by adding the necessary quantity of nitric acid to a concentrated solution of cobalt nitrate. At the same time the nitric acid required for dissolving all the cobalt should be added to the dissolving liquor during the dissolving operation, either continuously or intermittently in the form of concentrated nitric acid and in such quantities that the content of free nitric acid in the solution remains for example within the limits of from 3—5%. The catalysts are dissolved at a temperature that lies above the melting point of the paraffins contained in the catalyst and advantageously at temperatures between 70 and 90° C. Dissolving is assisted by a gentle stirring, but too vigorous stirring should be avoided. The solution can be easily separated from the Kieselguhr by decantation or by simple filtration. The paraffin separates out from the solution after cooling in the form of a solid cake.

The following is an example of the process according to the invention:—

6.9 kg. of spent cobalt catalyst (corresponding to 1.0 kg. of cobalt) containing 55% paraffin, 15% cobalt, 2% thoria, 27% Kieselguhr and 1% moisture are added in a dissolving vessel to 100 litres of a cobalt nitrate solution that contains 68 gm. of cobalt and 50 gm. of free nitric acid per litre. During a period of 20 minutes while the mass is being gently stirred 4 lires of a 50% nitric acid solution are added in such small quantities that the free nitric acid contained in the solution does not exceed 50 gm. per litre. The temperature is maintained at 88° C. After two hours the dissolving of the cobalt is finished and the reaction mixture is allowed to stand for half an hour. During this time the Kieselguhr separates out in the form of sand and is withdrawn from the vessel together with part of the adherent cobalt solution, from which it is filtered after cooling and washed with distilled water. The washed Kieselguhr contains only slight traces of cobalt and thorium. After the cooling of the cobalt solution that remains in the dissolving vessel the paraffin that floats as an oily film on the cobalt solution separates off as a solid layer. This paraffin is removed from time to time and

[*Price* 1/-]

purified by being melted and washed. The washing waters are poured into the dissolving vessel. Part of the cobalt solution withdrawn from the dissolving vessel is employed for preparing the catalyst, the remainder being used repeatedly for dissolving fresh charges of 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. A process for the treatment of cobalt catalysts that are produced by the precipitation of the catalytically active metals in the presence of Kieselguhr and that are employed for benzine synthesis from carbon monoxide and hydrogen, by dissolving in nitric acid and re-precipitating the catalytically active constituents, in which the spent catalysts are treated with nitric acid that contains no more than 20% and preferably no more than 3 to 5% of free nitric acid.

2. A process according to claim 1, in which a dissolving liquor is used that contains in addition to nitric acid the catalytically active constituents.

3. A process according to claims 1 and 2, in which the spent cobalt catalysts are dissolved at temperatures that are not far above the melting point of the paraffin contained in the catalyst.

4. A process for the treatment of cobalt catalysts substantially as hereinbefore described.

Dated this 8th day of September, 1938.

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