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(54) PROCESS FOR INCREASING THE PARAFFIN YIELD IN THE CATALYTIC HYDROGENATION OF CARBON MONOXIDE

(54) PROCÉDE POUR ACCROITRE LE RENDEMENT DE PARAFFINE DANS L'HYDROGENATION CATALYTIQUE D'OXYDE DE CARBONE

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The invention relates to a process for the catalytic hydrogenation of carbon monoxide in the presence of iron catalysts and particularly to a process whereby the yield of paraffin wax obtained at the beginning of the process is maintained or even increased during continuation of the process.

The expression "paraffin wax" signifies high molecular paraffin hydrocarbons which are solid at normal temperature.

It is known that the formation of paraffin wax in the catalytic hydrogenation of carbon monoxide depends on the alkali-metal content of the iron catalyst, i.e., the paraffin yield will increase up to a certain degree with the amount of alkali-metal. Unfortunately, however, the formation of paraffin wax diminishes as the synthesis process proceeds. This is particularly evident where the process is operated under normal pressure.

In medium pressure operation, attempts have been made to cope with this inconvenience by treating the catalyst with oxygen as soon as decrease in the formation of paraffin wax occurs to an appreciable degree. Although this procedure is reported to have brought about some success it suffers from the disadvantage that the catalyst must every time be freshly reduced or formed, i.e. reactivated, which necessary measure involves loss of time and of gas. This method is not applicable to normal pressure operation as the catalyst, having been treated with oxygen, cannot be revived by a formation action.

It has now been found that the iron catalyst, both in normal and medium pressure operation, will lose none of its paraffin wax-forming quality if alkali-metal compounds are periodically added to the catalyst during operation of the process. This step is preferably combined with the step of extracting the paraffin wax, an alkali-metal compound being added to the extraction agent after the bulk of the paraffin wax has previously been extracted. In doing so, it is of particular importance that the alkali-metal compound, unless it is soluble in the extraction agent, should be ground as finely as possible, and that the suspension of extraction agent and alkali-metal compound should have a good degree of stability - e.g., by using ultrasonics in preparing the suspension - as the efficiency of the process according to the invention depends greatly upon these factors. The finer the division of the alkali-metal compound and the more stable the suspension, the more intensive is the supplementary alkalization or re-alkalization. If the particles of the alkali-metal compound are too coarse and sedimentation of the alkali-metal compound occurs too rapidly, the greater part of it will be deposited upon the catalyst and but little of the alkali-metal compound will penetrate into the upper layers of the catalyst and none at all into the bulk of the catalyst mass.

The alkali-metal compound used for the realkalization is preferably used in such a fine state of division that not more than 80%, preferably less than

50%, of the alkali-metal compound suspended in the extraction agent will precipitate within one hour.

Suitable alkali-metal compounds for use in the process of the invention, are the oxides, hydroxides, carbonates, hydrocarbonates, phosphates, silicates and borates of sodium and potassium, also organic alkali-metal compounds, such as alkali-metal alcoholates, formates, acetates or the alkali-metal salts of higher organic acids, such as soaps.

The re-alkalization is not only a means for preventing any decrease in the formation of paraffin wax during the course of a period of operation, but the quantity of alkali-metal compound subsequently added will also induce an increase in the yield of paraffin wax from such iron catalysts which at first produce only small amounts of paraffin wax.

The process in accordance with the invention is further illustrated by the following examples:-

Example 1

An iron catalyst precipitated with soda and containing, apart from alkali-metal and copper, magnesium as activator and kieselguhr as carrier or support, is employed with water gas under normal pressure at 230°C., the space velocity being 70 normal cubic metres of water gas per hour per cubic metre of reactor space. At the end of 345 hours, the catalyst is extracted for the first time; operation is then immediately resumed and continued under the same conditions. Up to the 345th hour of operation, the yield of paraffin wax amounts to 35.7% of the

total products. Extraction is repeated at the end of the 605th hour of operation. Between the 346th and 605th and between the 606th and 896th hours of operation the yields of paraffin wax amount to 27.5% and 24.2% respectively. On the other hand, the production of paraffin wax is almost constant when the catalyst is treated according to the invention, which fact will be seen from Table 1 and from the following description.

Table 1

Formation of paraffin wax without and with treatment of the iron catalyst according to the process of the invention in normal pressure operation.

Formation of paraffin wax			
Without supplementary alkalization		With supplementary alkalization	
Hours of operation	Total % yield of paraffin wax	Hours of operation	Total % yield of paraffin wax.
1 - 345	35.7	1 - 374	34.9
346 - 605	27.5	375 - 624	36.2
606 - 896	24.2	625 - 915	34.1

The same catalyst is employed under the conditions already described; at the end of the 374th hour of operation, it is extracted with heavy gasoline. After the bulk of the paraffin wax has thus been removed, the final extraction is carried out with a heavy gasoline in which finely ground potassium carbonate is suspended. After completion of the extraction, employment of the catalyst is continued under the previous operating conditions. The yield of paraffin wax in the first operating period amounts to 34.9% of the

total products. At the end of the 624th hour of operation, the same procedure as during the first extraction according to the invention is repeated. The yields of paraffin wax in the 2nd and the 3rd operating periods amount to 36.2% and 34.1% respectively. It is thus possible to maintain, by means of the supplementary alkalization, the yield of paraffin wax at the value obtained during the first period of operation.

Example 2

A precipitated iron catalyst, containing dolomite as carrier, is employed under an absolute pressure of 11 atmospheres and at a temperature of 214°C. with a synthesis gas in which the H<sub>2</sub>/CO ratio is 1.7, the space velocity being 600 normal cubic metres of synthesis gas per hour per cubic metre of reactor space. At the end of the 1037th and 1875th hours of operation, the catalyst is extracted. In the three operating periods up to the 2558th hour of operation, the yields of paraffin wax are shown in Table 2

Table 2

Formation of paraffin wax without and with treatment of the iron catalyst according to the process of the invention in medium pressure operation.

Formation of paraffin wax			
Without supplementary alkalization		With supplementary alkalization	
Hours of operation	Total % yield of paraffin wax.	Hours of operation	Total % yield of paraffin wax.
1 - 1037	28.7	1 - 275	30.5
1038 - 1875	24.2	276 - 995	38.5
1876 - 2558	21.8	996 - 1668	41.2
		1669 - 2124	39.4
		2125 - 2678	38.9

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It will be seen from this Table that under the same operating conditions, the same catalyst will produce a considerably higher yield of paraffin wax if it is more frequently extracted and at the same time subjected to supplementary alkalization in accordance with the invention.

The embodiments of the invention in which an exclusive property of privilege is claimed are defined as follows:-

1. In a process for the synthesis of paraffin wax by the catalytic hydrogenation of carbon monoxide in the presence of a stationary iron catalyst by re-alkalisation of the catalyst after a period on-stream, the improvement which comprises effecting the re-alkalisation with the alkali in suspension in a solvent employed for extracting paraffin wax from the catalyst, the alkali being used in a finely divided state which is deposited as a sediment when the suspension is allowed to stand for one hour.

2. In a process for the synthesis of paraffin wax by the catalytic hydrogenation of carbon monoxide in the presence of a stationary iron catalyst, the improvement which comprises interrupting the hydrogenation, extracting paraffin wax from the catalyst with a solvent for the paraffin wax, impregnating the catalyst with an alkali metal compound by means of a suspension of the alkali metal compound in the said solvent the degree of subdivision of the alkali metal compound in the suspension being such that not more than 80% of the alkali metal compound will be precipitated when the suspension is allowed to stand for one hour, and thereafter resuming the hydrogenation.



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3. Improvement according to claim 1 or claim 2, in which less than 50% of the alkali metal compound in the suspension will be precipitated upon standing for one hour.

4. Improvement according to claim 2, in which the extraction and impregnation of the catalyst is effected at intervals of from 300 to 700 hours.

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