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12/20/2001 - 16:06:47

(12) **Patent:**

(11) **CA 530986**

(54) PROCESS FOR THE CATALYTIC HYDROGENATION OF CARBON MONOXIDE

(54) PROCEDE POUR L'HYDROGENATION CATALYTIQUE D'OXYDE DE CARBONE

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(74)	
(45)	**Sep. 25, 1956**
(22)	
(43)	
(52)	**260/753.6**
(51)	**N/A**
	No
(30)	**None**
	N/A
	Unknown

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

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In the catalytic hydrogenation of carbon monoxide with certain catalysts of the iron group (cobalt, nickel), it is known to use synthesis gases, which contain acetylene or other hydrocarbons with a triple bond.

According to the process of German Patent No. 764,165, increased olefin yields are, for example, in this way obtained with cobalt catalysts. To a certain extent the acetylene hydrocarbons added to the synthesis gas may be replaced by ethylene, and other hydrocarbons with a double bond. Hitherto iron catalysts have not been used in the treatment of carboh monoxide-hydrogen mixtures containing acetylene.

The surprising observation has been made that even at reduced temperatures a sufficiently high conversion is obtained with iron catalysts, particularly when impregnated with alkali salts of a non-volatile acid, such as potassium phosphate and/or potassium silicate, if synthesis gases are used which contain acetylene. The required acetylene content amounts to 0.5 - 10% by volume, preferably 0.5% - 1% by volume. The reduction possible in the synthesis temperature amounts to approximately 20°C.

It is known that with iron catalysts the synthesis temperature has a direct relation to the catalyst load and to the formation of high molecular hydrocarbons. A greater proportion of high molecular hydrocarbons are obtained only if a low synthesis temperature is used. In these circumstances, however, the possible load is very small and the ($CO + H_2$) conversion hardly exceeds 40% - 50%. Even though increased synthesis temperatures increase the conversion, they cause an increased methane formation, and a reduction in the paraffin formation.

In the process according to the invention, that is to say, if 0.5% - 10% acetylene are added, conversions of 65% - 70% may be obtained at synthesis temperatures of 195°C. - 200°C., the methane formation remaining below 7%. If acetylene is not added, total conversion of only 40% - 50% is obtained at these temperatures. It is appreciated that at an increased synthesis temperature of 220°C. - 225°C. a ($CO + H_2$) conversion of 65% - 70% is obtained, but on the other hand, there is a methane formation of 12% - 16%.

A further advantage of the process according to the invention lies in the fact that, due to the reduced synthesis temperature, a high yield in hydrocarbons boiling above 320°C. is obtained. Approximately 30% of hydrocarbons boiling above 320°C. are obtained including the paraffin which may be extracted from the catalyst.

If acetylene is added according to the process of the invention, not only the temperatures required for starting the iron catalysts but also the gradual increase in the synthesis temperature may be reduced by about 20°C. as compared with the temperatures required if the gases used are free from acetylene. In this way, there is a considerable lengthening in the life of the catalyst, because a temperature interval increased by approximately 20°C. is available for gradually increasing the synthesis temperature.

A high impregnation of the iron catalyst with alkali salts of a non-volatile acid is particularly advantageous. The alkali content (calculated as K_2O) may amount to 1 - 10% K_2O, preferably 3% K_2O, of the iron content.

The acetylene added to the synthesis gases may be replaced by other gaseous hydrocarbons with a triple bond, for example, vinylacetylene, methylacetylene and similar compounds. In place of acetylene an addition of hydrocarbons with a double carbon bond, for example, an addition of ethylene, may in part be used.

EXAMPLE.

From a solution of the corresponding nitrates, a catalyst was precipitated which contained 100 parts iron and 5 parts copper. After thorough washing the precipitated catalyst slurry was impregnated with normal potassium orthophosphate (KH_2PO_4) in such manner that the finished catalyst contained 3 parts K_2O to 100 parts iron (Fe). The finished catalyst had a reduction value equivalent to 40% free iron.

When this catalyst was loaded with 100 normal litres of water gas per litre, and per hour, there resulted a ($CO + H_2$) conversion of 50% - 55% at a synthesis temperature of 200°C. The methane formation amounted to approximately 11% of the liquid synthetic products. The synthetic products, including the paraffin obtained in the catalyst extraction, consisted of approximately 20% hydrocarbons boiling above 320°C.

When water gas containing 1.5% by volume of acetylene was converted with the same catalyst, and the same load at 200°C., the ($CO + H_2$) conversion was increased to 70% - 72%. The methane formation was reduced to approximately 7%. Approximately 30% of the synthetic products, including the paraffin obtainable by means of catalyst extraction, consisted of hydrocarbons boiling above 320°C.

The precipitated and dried catalyst was reduced at 250°C. with a hydrogen-nitrogen mixture which was passed at a flow velocity of 1.4 m. per second for 60 minutes over the catalyst. The gas flow velocity must be between 1 and 2 m. per second.

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

1. A process for the catalytic hydrogenation of carbon monoxide which comprises reacting a carbon monoxide hydrogen containing synthesis gas with a content of 0.5 - 10% by volume of a gaseous hydrocarbon with a triple bond while in contact with iron synthesis.catalysts impregnated with an alkali salt of a substantially non-volatile inorganic acid.

2. A process as defined in claim 1, wherein said gaseous hydrocarbon is acetylene.

3. A process as defined in claim 1, wherein said alkali salt is at least one of potassium phosphate and potassium silicate.

4. A process as defined in claim 2, wherein said alkali salt is at least one of potassium phosphate and potassium silicate.

5. A process as defined in claim 1, 2 or 3, wherein the iron catalysts used are reduced at a temperature of approximately 250°C. with a nitrogen-hydrogen mixture which is passed for approximately 60 minutes over the catalyst at a flow velocity of approximately 1-2 m. per second.

6. A process as defined in claim 1, 2 or 3, wherein synthesis temperatures are used which are approximately 20°C. below the corresponding temperature required when using iron catalysts with synthesis gases free from said gaseous hydrocarbons.

7. A process as defined in claim 1, 2 or 3, wherein the alkali content of said catalyst, calculated on potassium, comprises approximately 3% K_2O in relation to the iron content of the catalyst.

8. A process as defined in claim 2 or 4, wherein the acetylene added to the synthesis gases is, in part, replaced by a gaseous hydrocarbon with a double carbon bond.

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9. A process as defined in claim 2 or 4, wherein the acetylene added to the synthesis gases is in part replaced by ethylene.

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