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PATENT SPECIFICATION

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COMPLETE SPECIFICATION.

Process for the Production of Substantially Pure Methanol.

I, Etienne Audibert, of 35, rue Saint-Dominique, Paris (Seine Department), France, a French citizen, 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:—

This invention relates to an improved process for producing substantially pure methan involving the known equation:—

$$CO + 2H_2 = CH_3OH$$

It has long been recognised that copper constitutes a catalyst for many hydrogenising processes and that its activity varies considerably according to its manner of preparation. The metal of a light-red colour, and in compact state, which is obtained by reduction with incandescence, has catalytic properties much inferior to those of the violet-coloured metal prepared according to Sabatier's formula by reducing the black tetracupric hydrate at about 200° C.

Observations have shown that the violet-coloured copper will not enable the production of methanol to take place even at high pressures.

Processes for the production of methanol or other oxygenated products employing as a catalyst a reduced mixture of copper and at least one other metal or compound thereof and suitable pressure and temperature conditions, have already been suggested, and it has also been suggested to use copper in place of platinum as contact substance in the preparation of methyl alcohol by passing a current of hydrogen and carbon monoxide, heated to a temperature not exceeding 400 C., through a reaction vessel containing the catalyst, but no pressure conditions were specified.

According to this invention the process for manufacturing substantially pure methanol consists in causing a hydrogen-carbon monoxide mixture under high pressure and at raised temperature, to pass into contact with a catalyst consisting of copper prepared as follows:—

At boiling temperature an aqueous solution of a copper salt is precipitated by an alkali. The copper salt must be pure and in particular should not contain elements of the iron group. In addition it should be suitably chosen. Generally good results are obtained with the nitrate or a salt of an organic acid to the exclusion of the chloride or sulphate. The precipitate is washed with distilled water until the wash water does not indicate the least trace of acid or alkali and then dried in a vacuum at 50° C. The dried product is intimately mixed with a small quantity, for instance 10 to 20%, of violet-coloured copper and the mixture is then reduced under ordinary pressure with hydrogen or carbon monoxide. Care should be taken to circulate the reducing gas at a sufficiently slow rate or to mix it with a sufficiently large amount of an inert gas such as nitrogen or methane so that in spite of the disengagement of heat to which the reaction gives rise it may take place in an isothermal manner and without the temperature rising above 200° C. When the reduction is completed the catalyst is ready for use but care should be taken to avoid its contact with air or an oxidising gas.

Another manner of operating consists in calcining, preferably at as low a temperature as possible, nitrate or an organic salt of copper in pure state, washing very carefully the oxide so obtained, mixing it intimately with a small quantity of violet-coloured copper and reducing the mixture under the same conditions as referred to above.

If the precautions mentioned above are not observed the reduction of oxide or hydrate of copper furnishes a metal which either exhibits no catalytic activity for the reaction now in question, or if it possesses catalytic activity is very much less active than the product prepared in the manner indicated above.

The product causes a mixture of carbon monoxide and hydrogen under raised pressure of the order of about 100 atmospheres or more to react as follows:

$$CO+2H_2=CH_3OH$$

This reaction takes place exclusively under the only condition that the temperature of the mixture on entering the catalytic space does not exceed 160 to 180° C. The speed at which the reaction takes place can readily attain valves of industrial interest. Thus for example it permits of the realisation under pressures of 150 atmospheres or more of productions of several hundreds of grammes of methanol per litre of catalytic space and per hour.

It is necessary to operate in such a manner that in spite of the disengagement of heat to which the combination of carbon monoxide and hydrogen gives rise according to the equation

$$CO+2H_2=CH_3OH$$

the temperature in any point of the catalytic space does not exceed 400 to 450° C. Failure to observe this precaution results in the catalyser rapidly losing its properties.

Having now particularly described and ascertained the nature of my said invention and in what manner the same is to be performed, I declare that what I claim is:—

1. A process for the production of substantially pure methanol, which consists in causing a hydrogen-carbon monoxide mixture under high pressure and at raised temperature to pass into contact with a catalyst consisting of copper and prepared in the manner substantially as hereinbefore described.

2. The production of almost pure methanol substantially as described.

Dated this 30th day of January, 1926.

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