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D E C I S I O N
of 29 July 2003

Case Number: T 0265/00 - 3.3.1

Application Number: 93307531.9

Publication Number: 0590882

IPC: C07C 1/04

Language of the proceedings: EN

Title of invention:

Method of rejuvenation of hydrocarbon synthesis catalyst

Patentee:

ExxonMobil Research and Engineering Company

Opponent:

Mobil Oil Corporation

Shell internationale Research Maatschappij B.V.

Headword:

Catalyst rejuvenation/EXXONMOBIL

Relevant legal provisions:

EPC Art. 56

Keyword:

"All requests: inventive step (no) - obvious to try"

Decisions cited:

-

Catchword:

-



Case Number: T 0265/00 - 3.3.1

D E C I S I O N
of the Technical Board of Appeal 3.3.1
of 29 July 2003

Appellant: ExxonMobil Research and Engineering Company
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Decision under appeal: Decision of the Opposition Division of the
European Patent Office posted 4 February 2000
revoking European patent No. 0590882 pursuant
to Article 102(1) EPC.

Composition of the Board:

Chairman: A. J. Nuss
Members: P. P. Bracke
S. C. Perryman

Summary of Facts and Submissions

I. The appeal lies from the Opposition Division's decision to revoke European patent No. 0 590 882 since the claimed Process was not inventive over the cited prior art.

In particular, the Opposition Division found that the claimed process essentially differed from the prior art processes, as described, for example, in document

(4) J. Chem. Soc. Ind., 65, pages 128 to 136 (1946),

by the fact that the catalyst to be rejuvenated by treatment with hydrogen is dispersed in a hydrocarbon liquid in a slurry bubble column mode of operation instead of being treated with a gas on a fixed bed. As cobalt-catalysed Fischer-Tropsch reactions operated in slurry bubble columns were known and it was operationally convenient to operate a rejuvenation process in situ, it was obvious to conduct the rejuvenation process in a slurry bubble column mode of operation rather than being treated with hydrogen gas on a fixed bed.

II. As a response to amended claims filed by the Appellant (Proprietor of the patent) with the statement setting out the grounds of appeal, the Respondent (Opponent) filed with letter of 5 January 2001 *inter alia* documents

(19) US-A-2 440 109 and

(21) P.A. Ramachandran, Three Phase Catalytic Reactors, Gordeon and Breach, New York, 1983, pages 6 and 7.

III. During the oral proceedings before the Board, which took place on 29 July 2003, the Appellant filed, as a main request, a set of seven claims and, as a first, a second and a third auxiliary request, sets of claims containing seven, respectively five and four claims.

Claim 1 according to the main request read:

"1. A method for rejuvenating a deactivated or partially deactivated cobalt-containing slurry phase hydrocarbon synthesis catalyst having an initial catalyst activity, which method comprises: treating the catalyst, while dispersed in hydrocarbon liquids comprised primarily of C₁₀-C₅₀ linear paraffins in a slurry bubble column, with hydrogen or a hydrogen-containing gas, in the absence of carbon monoxide, at elevated temperature and pressure of 10.1 to 101.3 bar (10-100 atmospheres) for a period sufficient to recover at least 80+% of the initial catalyst activity."

Claim 1 according to the first auxiliary request read:

"1. A method for rejuvenating a deactivated or partially deactivated cobalt-containing slurry phase hydrocarbon synthesis catalyst having an initial catalyst activity, which method comprises: treating the catalyst, while dispersed in hydrocarbon liquids comprised primarily of C₁₀-C₅₀ linear paraffins sufficient to fully immerse the catalyst in a slurry bubble column, with hydrogen or a hydrogen-containing gas, in the absence of carbon monoxide, at a

temperature ranging from hydrocarbon synthesis temperature to substantially 40°C below hydrocarbon synthesis temperature and a pressure of 10.1 to 101.3 bar (10-100 atmospheres) for a period sufficient to recover at least 80+% of the initial catalyst activity."

Claim 1 according to the second auxiliary request read:

"1. A method for rejuvenating a deactivated or partially deactivated cobalt-containing slurry phase hydrocarbon synthesis catalyst having an initial catalyst activity, which method comprises: treating the catalyst, while dispersed in hydrocarbon liquids comprised primarily of C₁₀-C₅₀ linear paraffins sufficient to fully immerse the catalyst in a slurry bubble column, with hydrogen or a hydrogen-containing gas, in the absence of carbon monoxide, at a temperature ranging from hydrocarbon synthesis temperature to substantially 40°C below hydrocarbon synthesis temperature and substantially hydrocarbon synthesis pressure of 10.1 to 101.3 bar (10-100 atmospheres) for a period sufficient to recover at least 80+% of the initial catalyst activity."

Claim 1 according to the third auxiliary request read:

"1. A method for rejuvenating a deactivated or partially deactivated cobalt-containing slurry phase hydrocarbon synthesis catalyst having an initial catalyst activity, which method comprises: treating the catalyst, while dispersed in hydrocarbon liquids comprised primarily of C₁₀-C₅₀ linear paraffins sufficient to fully immerse the catalyst in a slurry

bubble column, with hydrogen or a hydrogen-containing gas, in the absence of carbon monoxide, at a temperature ranging from hydrocarbon synthesis temperature of 175°C to 300°C to substantially 40°C below hydrocarbon synthesis temperature and substantially hydrocarbon synthesis pressure of 10.1 to 101.3 bar (10-100 atmospheres) for a period sufficient to recover at least 80+% of the initial catalyst activity."

- IV. The Respondent submitted that the sets of claims according to the main and first to third auxiliary requests did not meet the requirements of Articles 123(2) and (3), 83 and 84 EPC. Moreover, he contested the novelty of Claim 1 according to the main request over document (19) and he argued that the claimed processes according to all requests were obviously derivable from the teaching of document (4) in combination with the teaching of *inter alia* document (19).
- V. The Appellant submitted that documents (19) and (21) were late-filed and, therefore, should not be taken into the procedure. Moreover, he argued that all the sets of claims according to the main and first to third auxiliary requests met the requirements of Articles 123(2) and (3), 83 and 84 EPC. Furthermore, he submitted that the claimed process was novel over the teaching of document (19) and that it was not directly and unambiguously derivable from the cited prior art documents.
- VI. The Appellant requested that the decision under appeal be set aside and that the patent be maintained on the

basis of the main request, or one of the auxiliary requests 1, 2 or 3, all submitted at the oral proceedings on 29 July 2003.

The Respondent requested that the appeal be dismissed.

Reasons for the Decision

1. The appeal is admissible.
2. *Late filed documents (19) and (21)*

Since the main criterion for deciding on the admissibility of late-filed documents is their relevance and since the content of both documents are relevant, as will appear from the discussion of inventive step, according to Article 114(1) EPC both documents have to be admitted in the proceedings.

3. Since the Board came to the conclusion that neither the main request nor any of the first, second or third auxiliary requests meets the requirement of inventive step, it is superfluous to give any reasoning as to whether the requirements of Articles 123(2) and (3), 83 and 84 EPC and the requirement of novelty are met.

4. *Inventive step*

- 4.1 Main request

In accordance with the "problem-solution approach" applied by the Boards of Appeal to assess inventive step on an objective basis, it is in particular

necessary to establish the closest state of the art forming the starting point, to determine in the light thereof the technical problem which the invention addresses and solves, and to examine the obviousness of the claimed solution to this problem in view of the state of the art.

- 4.1.1 The "closest state of the art" is normally a prior art document disclosing subject-matter aiming at the same objective as the claimed invention and having the most relevant technical features in common. Since Claim 1 relates to a method for rejuvenating a deactivated or partially deactivated cobalt-containing hydrocarbon synthesis catalyst, and since document (4) describes such rejuvenation of cobalt-containing hydrocarbon synthesis catalysts, document (4) can serve, as the closest prior art, as a suitable starting point for evaluating the inventive merit of the invention.

Document (4) discloses that cobalt-containing catalysts used in the synthesis of hydrocarbons from carbon monoxide and hydrogen can be maintained in a high state of activity by intermittent treatment with hydrogen under various conditions (see the abstract and the first paragraph of the article). It describes various methods of maintaining and restoring the catalyst activity of such cobalt-containing catalysts and comes to the conclusion that maximum catalyst life is obtained by frequent treatment with hydrogen at temperatures in the synthesis range and by repetition of the original reduction process, i.e. by passing dry electrolytic hydrogen downwards through the bed of catalyst at 390 to 405°C (see page 126, right-hand column, first full paragraph, page 130, second

paragraph and page 136, left-hand column, first full paragraph).

In this respect, the Appellant submitted that document (4) only concerned a method of dewaxing a cobalt-containing catalyst. Since, however, document (4) clearly concerns methods of **restoring** the catalyst activity (see page 126, right-hand column, line 6 to 9), the Board cannot agree that the disclosure of document (4) would be restricted to dewaxing processes.

- 4.1.2 Starting from document (4), the Appellant submitted, that the problem to be solved consisted in providing a method of rejuvenating a cobalt-containing hydrocarbon synthesis catalyst that has undergone short term, reversible deactivation as a result of slurry phase hydrocarbon synthesis operation, as described in the first paragraph of the patent in suit. Such problem was in particular observed in the hydrocarbon synthesis when a cobalt-containing catalyst was used in a slurry bubble column hydrogenation process.

However, Claim 1 is not restricted to the rejuvenation of cobalt-containing catalysts specifically to be used in a slurry bubble column hydrogenation process, but embraces the rejuvenation of any cobalt containing catalyst used in the hydrocarbon synthesis in a slurry phase.

Therefore the objective, starting from document (4), can only be seen in providing a method of rejuvenating a cobalt-containing catalyst to be used in hydrocarbon synthesis in a slurry phase.

4.1.3 The patent in suit claims to solve this problem by the method defined in Claim 1.

4.1.4 The first point to be considered in assessing inventive step is then whether it has been convincingly shown that by the process according to Claim 1 the problem underlying the patent in suit has effectively been solved.

It has never been contested that with the data described in the patent in suit it has been made plausible that the problem as defined above has effectively been solved.

4.1.5 Therefore, it remains to be decided, whether in the light of the teachings of the cited documents a skilled person seeking to solve the above-mentioned problem would have arrived at the process of Claim 1 in an obvious way or not.

4.1.6 When trying to solve the above stated technical problem, the skilled person would have come across document (19) which relates to a method of effecting catalytic conversion of gaseous reactants wherein the solid catalyst, such as cobalt, in finely divided form is suspended in a carrier liquid, such as petroleum hydrocarbons. More particularly, document (19) describes the catalytic hydrogenation of carbon monoxide wherein the gaseous components are dispersed in a slurry of catalyst in a carrier liquid (see column 1, lines 1 to 5 and 31 to 36, column 3, lines 51 to 54, column 3, line 75 to column 4, line 4 and claim 3). Moreover, in column 4, line 31 to column 5,

line 16, several procedures for removing solid or waxy material formed upon the catalyst are disclosed.

The Appellant submitted that this document teaches away from the claimed method since it concerns "destructive hydrogenation", which is just the opposite to the teaching of the patent in suit.

Certainly, in column 3, lines 50 to 56, a destructive hydrogenation is mentioned as a suitable procedure. However, the disclosure of document (19) is not limited to such destructive hydrogenation. In column 5, lines 12 to 16, it is, namely, taught that, as an **alternative procedure** for removing solid and waxy material from the catalyst, the carbon monoxide feed may be periodically discontinued while continuing to pass hydrogen through the system at elevated temperatures, a fact the skilled person would have noticed.

- 4.1.7 The Appellant also submitted that document (19) only mentioned the removal of wax and solids from the catalyst. As removing wax and solids was only part of the reactivation of the catalyst, document (19) did not give any hint how the catalyst may be reactivated.

The Board cannot accept this, since document (19) clearly suggests a method of rejuvenating a cobalt-containing catalyst. As by the term "rejuvenating" no difference is made between the kinds of reactivation, Claim 1 relates to any method of **rejuvenating** a cobalt-containing catalyst.

4.1.8 The Appellant also objected that document (19) was silent about a treatment of the catalyst in a slurry bubble column and that none of the cited documents teaches that cobalt-containing catalysts can be rejuvenated in a slurry bubble column with hydrogen or hydrogen-containing gas because of the risk of hydrogenolysis of the suspension liquid and all the disadvantages involved therewith.

However, from the drawing in document (19) and the corresponding description in column 1, line 37 to column 3, line 40, and column 4, lines 16 to 30, describing the reaction of carbon monoxide with hydrogen in a slurry of catalyst in a carrier liquid, there can be no doubt that the fluid mixture comprising carrier liquid, suspended catalyst and bubbles of reactant gas rises through a tubular reactor by the lifting effect of the gases and overflows from the top of the reactor into a separating vessel. The Board does not see in what the process described in document (19) differs from the hydrogenation in a slurry bubble column as from document (21) it clearly follows that in a bubble column slurry reactor the particles are suspended by means of gas-induced agitation and that in such a way the slurry mixture rises from the bottom of a tubular reactor to the top of it.

Thus, by the teaching in column 5, lines 12 to 16, that the catalyst may be freed from wax and solids by periodically discontinuing carbon monoxide feed while continuing to pass hydrogen through the system, document (19) teaches the rejuvenation of the catalyst by using a column which the skilled person would regard

as a slurry bubble column in the presence of hydrogen and in the absence of carbon monoxide.

- 4.1.9 It is true that document (19) mentions petroleum hydrocarbons without specifically mentioning the use of C₁₀-C₅₀ linear paraffins under a pressure of 10.1 to 101.3 bar (10-100 atmospheres).

According to the case law of the Boards of Appeal, features which do not contribute to the solution of the problem are not to be considered in assessing inventive step of a combination of features (see Case Law of the Boards of Appeal of the EPO, 4th edition 2001, point I.D.6.5).

As the Appellant never provided any proof, that the choice of the pressure range and the carrier liquid would have any influence on the recovery percentage of the catalyst, those features are not to be taken into consideration in assessing inventive step.

- 4.1.10 In accordance with the case law of the boards of appeal, a course of action can be considered obvious not only when the results are clearly predictable but also when a skilled person would have carried it out with a reasonable expectation of success.

- 4.1.11 Since from the disclosure of document (19) a skilled person would have carried out the process of Claim 1 with a reasonable expectation of success to rejuvenate a cobalt-containing slurry phase hydrocarbon synthesis catalyst, the method of Claim 1 is an obvious solution to the problem underlying the patent in suit. Therefore,

Claim 1 and, thus, the main request cannot be considered to meet the requirement of inventive step.

4.2 First auxiliary request

The method of Claim 1 differs from the method of Claim 1 according to the main request in that the hydrocarbon liquids are sufficient to fully immerse the catalyst and that the rejuvenation is conducted at a temperature ranging from hydrocarbon synthesis temperature to substantially 40°C below hydrocarbon synthesis temperature.

Since in a slurry bubble column the particles are suspended in a carrier liquid, the full immersion of the catalyst is a logical consequence thereof, as acknowledged by the Appellant (see Appellant's letter of 14 June 2000, page 2, last paragraph). Moreover, since it is known from column 5, lines 12 to 16, of document (19) that the rejuvenation is conducted under elevated temperature and since for conducting the rejuvenation specifically at a temperature ranging from hydrocarbon synthesis temperature to substantially 40°C below hydrocarbon synthesis temperature, as mentioned in Claim 1, an effect has never been shown, an inventive step cannot be based on these features.

Therefore, Claim 1 and, thus, the first auxiliary request cannot be considered to meet the requirement of inventive step.

4.3 Second and third auxiliary requests

The method of Claim 1 according to the second and third auxiliary request differs from the method of Claim 1 according to the first auxiliary request by the specification that the pressure range of 10.1 to 101.3 bar (10-100 atmospheres) is a substantially hydrocarbon synthesis pressure, respectively, by the specification that the hydrocarbon synthesis temperature is 175°C to 300°C.

Since, however, for such specifications an effect has not been shown, also these features may not form the basis for an inventive step.

Order

For these reasons it is decided that:

The appeal is dismissed

The Registrar:

The Chairman

N. Maslin

A Nuss