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**Datasheet for the decision
of 30 April 2015**

Case Number: T 0023/12 - 3.3.05

Application Number: 01204576.1

Publication Number: 1190770

IPC: B01J29/90, B01J29/89

Language of the proceedings: EN

Title of invention:

Process for regenerating epoxidation catalysts

Patent Proprietor:

Lyondell Chemical Technology, L.P.

Opponent:

Evonik Degussa GmbH

Headword:

Process for regenerating/Lyondell

Relevant legal provisions:

EPC Art. 56

Keyword:

Inventive step - (yes)
Inventive step - non-obvious further method

Decisions cited:

T 0037/82, T 1831/07, T 0162/98

Catchword:



**Beschwerdekammern
Boards of Appeal
Chambres de recours**

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Case Number: T 0023/12 - 3.3.05

D E C I S I O N
of Technical Board of Appeal 3.3.05
of 30 April 2015

Appellant: Evonik Degussa GmbH
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Decision under appeal: **Decision of the Opposition Division of the
European Patent Office posted on 11 November
2011 rejecting the opposition filed against
European patent No. 1190770 pursuant to Article
101(2) EPC.**

Composition of the Board:

Chairman G. Rath
Members: A. Haderlein
P. Guntz

Summary of Facts and Submissions

- I. The appellant (opponent) lodged an appeal against the decision of the opposition division rejecting the opposition against patent No. EP 1 190 770.
- II. The opposition division held that the ground of opposition mentioned in Article 100(a) in conjunction with Article 56 EPC did not prejudice the maintenance of the patent unamended, having regard to the following documents:
- D1: JP 3-114536 and its German translation
D2: EP 230949 A2
- III. In its reply to the statement setting out the grounds of appeal, the respondent (patent proprietor) filed the following document:
- A13: Epoxidation Run Plot
- IV. The appellant filed the following document:
- D3: EP 1 085 017 A1.
- V. The sole independent claim of the patent as granted reads as follows:
- "1. A method of regenerating a titanium-containing zeolite which has been used as an epoxidation catalyst comprising washing the titanium-containing zeolite at a temperature of at least 150°C with a solvent containing a source of a cation selected from the group consisting of ammonium, alkali metals, and mixtures thereof."

VI. Oral proceedings took place on 30 April 2015.

VII. The appellant's arguments may be summarised as follows:

The closest prior art was represented by one of the examples 4 to 7 of D1 since the effect mentioned in paragraph 0026 of the patent in suit only occurred in a continuously operated epoxidation process.

The process according to claim 1 of the patent in suit differed from that of example 5 of D1 in that washing occurred at a temperature of at least 150°C and in that the solvent contained a source of a cation selected from the group consisting of ammonium, alkali metals, and mixtures thereof.

The problem stated in paragraph 0026 of the patent in suit, i.e. the provision of catalysts which more quickly reach high epoxide yields upon recommencement of epoxidation, was already solved in D1. In example 5 of D1, directly after starting the reaction again, the same epoxide yield was achieved as was obtained with a fresh catalyst. Under the reaction conditions of D1, the highest selectivity values were obtained after the start-up of the epoxidation plant when resuming epoxidation after regeneration, followed by a steady decrease in selectivity. There was no indication in D1 that a long time elapsed after resumption of the epoxidation reaction. The skilled person would derive from D1 that the values indicated in its Table 6 reflected the yields obtained directly after resumption of the epoxidation reaction. In comparison, it was due to the specific reaction conditions used in the comparative examples of the patent in suit, i.e. example 3, regeneration without ammonia, and of A13, i.e. regeneration without ammonia at around 500 hours,

that the maximum selectivity was reached only after some 100 hours. In contrast, the conditions in D1 were such that the maximum selectivity was observed right after the start-up of the epoxidation plant after regeneration.

D3 showed in example 22 that by using ammonium in the feed stream a selectivity of as high as 94.8% was obtained already after 8 hours. Thus, the alleged improvement with respect to the lapse of time in order to achieve maximum selectivity did not occur over the whole scope claimed, but occurred at best only under the specific reaction conditions chosen by the respondent. It was not credible that the effect, if any, occurred over the whole scope claimed since claim 1 also covered sources of cations which were acidic, as stated on page 5, lines 54 to 56, of the patent in suit ("The source of ammonium or alkali metal cations may be an acidic, neutral or basic salt such as..."). It was not plausible that using an acidic cation source in the solvent of D1 would shorten the time lapse needed to arrive at the maximum selectivity.

It was questionable whether the problem to be solved was to provide an alternative. Rather, the problem to be solved was to provide a further regeneration method.

According to T 37/82, features which did not contribute to the solution of a technical problem should be disregarded when assessing inventive step. D1 suggested carrying out regeneration at a temperature of 150°C or more. Since the presence of cations in the solvent did not contribute to the solution of the problem, the subject-matter of claim 1 lacked an inventive step in view of D1 alone.

Moreover, D2 was concerned with the improvement of epoxide selectivity and disclosed on page 3, lines 24 to 33, the use of a basic substance before and during the reaction. It was true that D2 taught on page 4, first paragraph, that by including a neutralisation agent in the feed the catalyst would be prevented from deteriorating over time. But D1, which was published later than D2, proved that it was recognised in the art after D2 that regeneration was indeed necessary. Since D2 taught that the catalyst could also be neutralised before the reaction, the skilled person would have included the neutralisation in the solvent wash during regeneration, i.e. before resumption of the epoxidation reaction. Thus, the skilled person would have arrived at the subject-matter of claim 1 in an obvious way.

VIII. The respondent's arguments may be summarised as follows:

D1 was the closest prior art. In D1, regeneration was done by solvent wash only, i.e. in the absence of a source of cations. D1 did not disclose the time at which the maximum yield was achieved. By using a cation source in the solvent wash, as shown in example 3 of the patent in suit, it was possible to increase substantially the selectivity at the start of the reaction and to achieve the maximum selectivity in a substantially shorter time period. This effect was also shown in the tests submitted as document A13.

In contrast, the appellant did not provide any tests that would cast doubt on the occurrence of this effect. As to obviousness, D2 did not deal with the problem of regenerating a titanium-containing zeolite, but dealt with the problem of reducing the amount of undesirable by-products of the epoxidation reaction as evidenced on

page 2, lines 25 to 30. D2 taught on page 4, lines 10 to 23, that in a continuous process the addition of a basic compound during the epoxidation process was sufficient in order to avoid deterioration of the catalyst over time.

There was therefore no motivation for the skilled person to add a basic compound to the methanol wash of D1.

IX. Requests

The appellant requested that the impugned decision be set aside and that the patent be revoked.

The respondent requested that the appeal be dismissed.

Reasons for the Decision

1. Inventive step

1.1 Invention

The invention concerns a method for regenerating epoxidation catalysts.

1.2 Closest prior art

Such a process is known from D1, which the parties took as the starting point for assessing inventive step.

D1, and in particular example 5, discloses a method of regenerating a titanium-containing zeolite which has been used as an epoxidation catalyst comprising washing the titanium-containing zeolite at 85°C with methanol. As conceded by the appellant, example 5 of D1 discloses

neither that regeneration is carried out at a temperature of at least 150°C nor that the solvent contains a source of a cation selected from the group consisting of ammonium, alkali metals, and mixtures thereof.

1.3 Problem

According to the patent in suit (see in particular page 6, lines 5 to 7) and according to the respondent's pleading during the written and oral proceedings, the problem was to provide a method of regenerating a titanium-containing zeolite which has been used as an epoxidation catalyst, resulting in catalysts which more quickly reach high epoxide yields upon recommencement of epoxidation.

1.4 Solution

As to the solution of this problem, the patent in suit proposes a method of regenerating a titanium-containing zeolite which has been used as an epoxidation catalyst characterised in that the washing of the titanium-containing zeolite is carried out at a temperature of at least 150°C and in that the solvent contains a source of a cation selected from the group consisting of ammonium, alkali metals, and mixtures thereof.

1.5 Success of the solution

Independently of the conclusion on the success of the proposed solution to the above-mentioned problem, the technical problem underlying the patent in suit at least can be seen as the provision of a further method of regenerating a titanium-containing zeolite which has been used as an epoxidation catalyst, that problem

being in line with the submissions of the appellant.

The board is satisfied that this problem is solved.

It is only if the solution to this least ambitious problem were found to be obvious vis-à-vis the closest prior art that the issue of whether or not an improved technical effect was achieved over that prior art, as alleged by the respondent, representing a more ambitious problem, would arise (cf. T 1831/07, Reasons 6.4; T 162/98, Reasons 4.2, second paragraph).

1.6 Obviousness

It remains to be decided whether the proposed solution was obvious in view of the prior art.

- 1.6.1 In view of the teaching on page 2, right-hand column, fourth paragraph, of the translation of D1, it was certainly obvious to operate the regeneration method of example 5 of D1 at a temperature of at least 150°C.

D1 however does not contain any hint to include ammonium or alkali metals in the solvent during regeneration.

- 1.6.2 The appellant referred to decision T 37/82, in which it was held that features which did not contribute to the solution of a technical problem should be disregarded when assessing inventive step.

According to the appellant, D1 suggested carrying out regeneration at a temperature of 150°C or more, and since the presence of cations in the solvent did not contribute to the solution of the problem, the subject-matter of claim 1 lacked an inventive step in view of

D1 alone.

1.6.3 The board does not agree.

The technical solution proposed according to claim 1 comprises the presence of cations in the solvent and a temperature of at least 150°C. Both features are elements of the technical solution and contribute to the solution of the problem posed, namely the provision of a further method of regenerating a titanium-containing zeolite. Hence, the feature relating to the source of a cation cannot be disregarded when assessing obviousness of the proposed solution, which is also in line with the decision referred to by the appellant (cf. T 37/82, Reasons 3).

1.6.4 D2 teaches using ammonium or alkali metal salts (see page 3, lines 24 et seqq.) in order to reduce the amount of undesirable by-products during epoxidation using a titanium-containing zeolite. Further according to D2, the catalyst needs to be neutralised "before and/or during the reaction" (page 3, line 25). A regeneration step is not explicitly disclosed in D2, but it is said that "[t]he catalyst is stable under the reaction conditions and may be completely recovered and reused" (see page 5, lines 13 and 14).

Further according to D2, when using a continuous epoxidation process it was "sufficient" to add to the feed a corresponding amount of neutralisation agent (page 4, lines 10 et seqq.). This teaching is already implemented in D1, in that ammonium is present in the feed stream (see examples 3 to 5).

For the board, the skilled person faced with the teaching of D2 would already for this reason not be

incited to include ammonium or alkali metal salts in the solvent during the regeneration step in D1.

- 1.6.5 As pointed out by the appellant, D2 teaches that it was possible to prevent the catalyst from deteriorating over time (page 4, lines 14 to 19), whereas later on it was recognised in the art that regeneration of the catalyst was indeed necessary, as evidenced by D1, published after D2.

The board concludes that for this reason too the skilled person would not have consulted D2 to solve the problem posed. In fact, the skilled person would not have consulted D2 when it comes to regeneration of the catalyst since D2 teaches that such regeneration is not necessary.

- 1.6.6 According to a further argument of the appellant, the skilled person would have included a neutralisation agent such as ammonium in the solvent during regeneration in D1, since that regeneration step occurred before a further epoxidation step and D2 explicitly taught the possibility carrying out neutralisation of the catalyst before and during epoxidation.

The board is not convinced by this argument. It is true that D2 also teaches the possibility of neutralising the catalyst before and during the epoxidation reaction. This teaching needs however to be seen in the overall context of D2. Examples 1 and 2 of D2 deal with neutralisation of the catalyst before the epoxidation reaction. In these examples, the fresh catalyst is neutralised, washed several times and calcined for several hours at 550°C. This means that the skilled person would, in the light of the general teaching on

page 3, lines 24 to 26, of D2, at most have prepared the fresh catalyst, i.e. the catalyst which had not yet been used for epoxidation, in D1 by contacting it with ammonium or alkali metal salts followed by washing and calcination. But the skilled person would not have included this step during regeneration since this would have entailed multiple washing steps and calcination of the catalyst, eventually requiring the removal of the catalyst from its bed.

1.6.7 The board therefore concludes that the skilled person would not have arrived in an obvious way at the subject-matter of claim 1.

1.7 The requirements of Article 56 EPC, corresponding to the sole ground of opposition invoked by the appellant, are met.

Order

For these reasons it is decided that:

The appeal is dismissed.

The Registrar:

The Chairman:



C. Vodz

G. Rath

Decision electronically authenticated