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D E C I S I O N
of 30 August 2004

Case Number: T 1070/01 - 3.3.3

Application Number: 93121042.1

Publication Number: 0604993

IPC: C08F 10/00

Language of the proceedings: EN

Title of invention:

Method for operating reactor for polymerizing olefins

Patentee:

NIPPON PETROCHEMICALS CO., LTD.

Opponent:

BP Chemicals Ltd

Headword:

-

Relevant legal provisions:

EPC Art. 56

Keyword:

"Inventive step - yes (main request)"

Decisions cited:

-

Catchword:

-



Case Number: T 1070/01 - 3.3.3

D E C I S I O N
of the Technical Board of Appeal 3.3.3
of 30 August 2004

Appellant: NIPPON PETROCHEMICALS CO., LTD.
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Respondent: BP Chemicals Ltd
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Decision under appeal: Decision of the Opposition Division of the
European Patent Office posted 17 July 2001
revoking European patent No. 0604993 pursuant
to Article 102(1) EPC.

Composition of the Board:

Chairman: R. Young
Members: W. Sieber
H. Preglau

Summary of Facts and Submissions

I. The mention of the grant of European patent No. 0 604 993, with 5 claims, in respect of European patent application no. 93 121 042.1, filed on 28 December 1993 and claiming JP priorities of 29 December 1992 (JP 360307/92) and 30 December 1992 (JP 360001/92 and JP 360004/92), was published on 5 August 1998 (Bulletin 1998/32). Granted Claim 1 read as follows:

"A method for operating a fluidized bed reaction system for polymerizing olefins which comprises the steps of

feeding a catalyst comprising titanium and magnesium; or vanadium and magnesium; or titanium, vanadium, and magnesium, an organoaluminum compound, and olefins having 2 to 8 carbon atoms into said reaction system;
polymerizing or copolymerizing said olefins regularly under vapor phase conditions at a temperature in the range of 10 to 200°C and under a pressure in the range of atmospheric pressure to 7 MPa (70 kg/cm²·G);
stopping the reaction of said polymerization or copolymerization by discontinuing the addition of catalyst and the feed of olefins with or without feeding a deactivator;
purging the reaction system with an inert gas;
restarting the polymerization or copolymerization of olefins by:

(i) feeding an organoaluminum compound into the reaction system without discharging the previously

formed polymer particles from the reaction system, wherein when no deactivator is used, the quantity of the organoaluminum compound to be fed is such an amount corresponding to 0.2 to 10 aluminum atoms in said organoaluminum compound relative to 1 aluminum atom in the organoaluminum compound remaining in the reaction system before stopping the polymerization or copolymerization, and when a gaseous deactivator is used, the quantity of the organoaluminum compound to be fed is such an amount corresponding to 1 or more aluminum atoms remaining in the reaction system at the stopping of the polymerization or copolymerization, (ii) then feeding olefins and hydrogen as a molecular weight modifier with the circulation of nitrogen, thereby gradually raising the pressure of the reaction system, and (iii) then supplying the catalyst into the reaction system.

Claims 2 to 5 were dependent claims directed to elaborations of the method according to Claim 1.

II. A notice of opposition was filed on 5 May 1999 by BP Chemicals Limited, requesting revocation of the patent in its entirety on the grounds of Article 100(a) EPC, ie lack of novelty and lack of inventive step, and on the grounds of Article 100(b) EPC, ie insufficiency of disclosure, the latter being withdrawn during the opposition proceedings. The opposition was - *inter alia* - supported by the following documents:

D1: US-A-4 326 048; and

D3: EP-A-0 180 420.

III. By a decision which was announced orally on 27 June 2001 and issued in writing on 17 July 2001, the opposition division revoked the European patent for the following reasons:

(a) The proprietor's main request (rejection of the opposition and maintenance of the patent as granted) was refused because the subject-matter of Claim 1, although novel, was not inventive over a combination of documents D1 and D3.

(b) The claims of the proprietor's auxiliary request corresponded to those of the main request, except that the introductory part of step (i) of Claim 1 had been amended to "(i) feeding an organoaluminum compound into the reaction system without discharging from the reaction system the previously formed polymer particles, which are held intact in the reactor under airtight conditions".

According to the decision, the amendment that the reactor be held under airtight conditions did not add an inventive teaching to the claimed subject-matter.

IV. On 24 September 2001, the proprietor (hereinafter referred to as the appellant) filed a notice of appeal against the above decision, the prescribed fee being paid on the same day.

In the statement of grounds of appeal, filed on 22 November 2001, the appellant argued in essence as follows:

The patent in suit related to a new technical concept of solving the problems occurring during the restart of a gas phase polymerization in a fluidized bed reactor after the polymerization had been interrupted, whereby the previously formed polymer was retained in the reactor. In fact, the unstable polymerization which occurred in a restart operation could be avoided by first adjusting the Al/Ti ratio in the catalytic system, and then introducing the olefin(s) to restart the polymerization. The skilled person in the relevant field could not derive this technical concept from either of D1 or D3 or from a combination of both documents. Furthermore, D3 related to the (new) start of an olefin polymerization, whereas the patent in suit and D1 were directed to the restart after an interruption of the polymerization. Thus, the opposition division had combined two pieces of prior art which related to different polymerization systems and which, therefore, could not properly be combined.

- V. With registered letter of 30 November 2001, a copy of the statement setting out the grounds of appeal was sent to the opponent (hereinafter referred to as the respondent) and a time limit of four months was set to file any submissions in answer to the appellant's statement. However, no submissions have been filed by the respondent, a fact confirmed by the respondent in a telephone conversation on 3 August 2004.

VI. The appellant requested that the decision under appeal be set aside and that

- the patent be maintained as granted (main request), or, in the alternative,
- the patent be maintained on the basis of the auxiliary request filed on 27 December 1999 during the opposition procedure, and
- oral proceedings be held in case none of the previous requests could be granted.

No request has been filed by the respondent.

Reasons for the Decision

1. The appeal complies with Articles 106 to 108 EPC and Rule 64 EPC and is therefore admissible.
2. A decision is possible at this stage since both parties, and in particular the respondent (see point V, above), have had an opportunity to comment on the grounds and evidence on which this decision is based (Article 113(1) EPC).
3. *Main request*
 - 3.1 The main request relates to Claims 1 to 5 as granted which have never been objected to under Article 100(c) EPC.

Despite the fact that the opponent withdrew the objection based on Article 100(b) EPC (section II, above), the decision under appeal held that the patent as granted met the requirements of Article 83 EPC. Furthermore, the decision under appeal acknowledged novelty of the subject-matter of the granted claims. The board sees no reason to depart from these findings.

Hence, it remains to be decided whether the subject-matter of Claims 1 to 5 involves an inventive step in the sense of Article 56 EPC.

3.2 The closest state of the art; the technical problem

3.2.1 The patent in suit is in the field of gas phase polymerization of olefins in the presence of a Ziegler-Natta-catalyst in a fluidized bed reactor. In particular, it is concerned with a method of restarting the gas phase polymerization after the polymerization has been interrupted whereby the previously formed polymer particles are retained in the fluidized bed reactor. By this method which comprises three steps (i) to (iii) as set out in Claim 1 (section I, above), the restarting operation after the temporary stopping of the reactor is improved, in particular with respect to the formation of sheet-like polymer and unstable reaction conditions in the initial period of the operation (column 3, lines 12 to 21 and lines 33 to 35 of the patent specification).

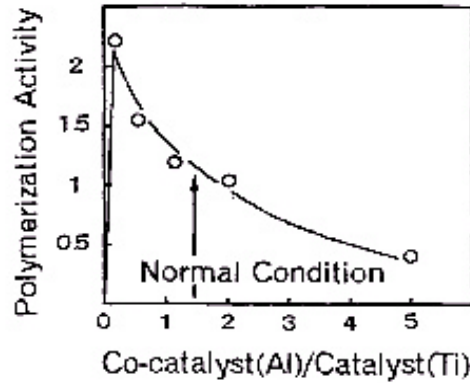
3.2.2 A method of rapidly terminating and efficiently restarting a gas phase olefin polymerization reaction using a Ziegler-Natta catalyst system is known from document D1 which is considered by the board, in line

with the decision under appeal, to represent the closest state of the art.

D1 discloses a method of rapidly terminating and restarting a gas phase olefin polymerization reaction using a titanium halide/aluminum alkyl catalyst system comprising (1) discontinuing catalyst addition, (2) discontinuing reactor quench liquid flow, (3) discontinuing reactor off gas flow, (4) injecting an amount of carbon oxide sufficient to terminate the reaction, (5) discontinuing recycle gas flow, (6) venting and flushing polymerization reactor, (7) resuming quench liquid, off gas and recycle gas flow, (8) injecting an amount of alkylaluminum sufficient to initiate polymerization and (9) resuming titanium halide addition. Thus, according to the teaching of D1, the polymerization is restarted by firstly resuming the recycle gas flow which comprises the olefin to be polymerised. Only later, the alkylaluminum is introduced. In contrast, the method according to the patent in suit feeds the organoaluminum compound to the reactor first (step (i) of Claim 1) followed by feeding the olefin (step (ii) of Claim 1). Hence, the claimed method differs from the method of D1 not only by using a catalyst system that additionally contains magnesium, but also by reversing the order of the steps in the restart of the polymerization.

- 3.2.3 As explained by the appellant in the statement of grounds of appeal, in a reaction system for olefin polymerization using a catalyst system comprising vanadium and/or titanium and magnesium (hereinafter referred to as "Ti") and an organoaluminum compound (hereinafter referred to as "Al") the polymerization

activity depends on the ratio of Al/Ti as shown in the following figure:



At a low Al/Ti ratio the catalytic activity increases abruptly with a slight increase of the Al/Ti ratio, and after the catalytic activity has reached a maximum it decreases gradually with higher Al/Ti ratios. Normally, the polymerisation is carried out employing an Al/Ti ratio after the maximum of catalytic activity in view of the stability of the reaction (in the examples of the patent in suit the polymerization is carried out at a ratio of Al/Ti = 1.1). When the polymerisation is interrupted in such a system, Al is liable to be consumed due to the contact with impurities and deactivator, if used. Thus, when the reaction is stopped without using a deactivator, active Ti still exists while Al becomes insufficient. When, for example, carbon dioxide is introduced as a deactivator, Al is consumed by reaction with carbon dioxide. In each case, the Al/Ti ratio decreases to a value in the region on the left side of the maximum peak in the above figure.

When the polymerization is restarted and (as in the process of D1) the olefin is fed first into the reaction system which still contains active solid Ti, the polymerization starts under the condition that the

amount of alkylaluminum is insufficient. In other words, the Al/Ti ratio is in the region left to the maximum of the graph in the above figure where the catalytic activity largely increases with a small increase in the Al/Ti ratio with the result that the polymerisation starts abruptly and in an unstable way, resulting in the formation of undesired polymers, such as sheet-like polymers. On the contrary, when (as according to the patent in suit) the organoaluminum compound is fed in advance to obtained a desired Al/ratio, the polymerization starts in a stable manner after the introduction of the olefin without fluctuation in polymerization activity so that the formation of sheet-like polymers may be suppressed and a stable operation may be attained.

3.2.4 Thus, the technical problem objectively arising may be seen in the improvement of the restart operation after the temporary stopping of the reactor in a gas phase olefin polymerization. In particular, the formation of sheet-like polymers and unstable reaction conditions should be avoided.

3.2.5 The decision under appeal criticized that Comparative Example 1 in the patent in suit was not suitable to demonstrate a surprising technical effect over D1 since Comparative Example 1 could not be considered as a reworked example of D1. However, the board cannot accept this criticism for the following reasons.

Firstly, as pointed out by the appellant, Comparative Example 1 was carried out in order to show that the order of feeding alkylaluminum and olefin is essential to the claimed subject-matter. Secondly, and even more

important, the appellant provided with the experiments and declaration of Mr Niwa, filed already during the examination procedure on 17 October 1996, a further experimental proof of the effect attained by the claimed method. Although the appellant referred to these experiments during the opposition procedure (letter filed on 27 December 1999, point IV), they were not taken into account in the decision under appeal when formulating the objective technical problem.

In Mr Niwa's experiment, the quantity of alkylaluminum fed to the polymerization system was made the same as that in Example 1 of the patent in suit, while only the order of feeding alkylaluminum and olefin was reversed, ie the same order of steps as disclosed in D1 was used. It was shown that a desirable restart operation could not be carried out when the order of feeding alkylaluminum and olefin was different from that of the patent in suit. In this context, the board notes that the comparative test submitted by the appellant goes even one step further than the closest prior art because in the comparative test the same catalyst as required in the patent in suit has been used, ie including magnesium. Thus, the comparative test submitted by the respondent lies closer to the patent in suit than the closest state of the art and a possible technical effect arising from the use of a different catalyst has not been taken into account. But even this variant of the closest prior art shows convincingly that the restart of the polymerization is improved.

3.2.6 In summary, the board finds it credible that the measures set out in Claim 1, in particular steps (i)

to (iii), provide an effective solution to the objective technical problem (section 3.2.4, above).

3.3 Inventive step

To assess the question of inventive step, it is necessary to consider whether the skilled person, starting from D1 as the closest prior art and wishing to improve the restart operation, in particular a stable restart without the formation of sheet-like polymers, would have expected that this could be achieved by changing the order of addition of organoaluminum and olefin.

3.3.1 There is no suggestion in D1 itself as to how the restart of the polymerisation might be further improved, let alone a hint to changing the stated order of steps as a more promising variant for the restart of the polymerisation. Apart from that, there is no pointer in D1 to use a catalyst system containing magnesium.

3.3.2 The only other document cited in the decision under appeal is D3. D3 discloses a process for the start-up of polymerization or copolymerization in the gas phase of α -olefins in the presence of a catalytic system of the Ziegler-Natta type and of a charge powder. The charge powder may be chosen from a wide variety of inorganic and organic solid particles, such as silica, alumina, talc, magnesia or a polymer or copolymer, preferably a polyolefin powder of the same nature as that of the polyolefin powder to be produced in the process of D3 (page 12, lines 14 to 21). The charge powder is treated with an organoaluminum compound whereby the treatment may be performed in the presence

of the reaction gas mixture to be used during (co)polymerization (page 14, lines 6 to 12). However, this treatment of the polyolefin charge powder in D3 bears no resemblance to the restart operation in the patent in suit, namely because the polyolefin charge powder of D3 does not comprise an active catalyst component whose activity may be restored upon contact with an organoaluminum compound. In the start-up procedure of D3, the charge powder is **newly** introduced into the reactor with the result that any catalyst residue present in the charge powder would be permanently deactivated. In fact, the treatment of the charge powder with an organoaluminum compound serves only the purpose of achieving a dehydration of the powder. In such a process where no active catalytic system is created it may well be that it is, as alleged in the decision under appeal (point 5 of the reasons), "irrelevant whether organoaluminum is fed before or after the beginning of the olefin feed polymerization". However, it is not permissible to transfer this specific teaching to a completely different process, namely a process involving the creation of a catalytic system.

Thus, D3 cannot provide any hint to the solution of the technical problem relating to the restart of a gas phase polymerization (point 3.2.4, above) because, firstly, D3 is in principle not concerned with the restart of a previously interrupted gas phase polymerization whereby the polymer is retained in the fluidized bed reactor, and secondly, the specific step of treating the charge powder with an organoaluminum compound relied upon in the decision under appeal does not relate to the start, let alone the restart, of a

gas phase polymerization. Hence, any combination of D1 with D3 appears to be based on hindsight.

3.3.3 In summary, the solution to the objective technical problem does not arise in an obvious way from the state of the art. Consequently, the subject-matter of Claim 1 as granted, and, by the same token, the subject-matter of Claims 2 to 5 as granted involves an inventive step (Article 56 EPC).

4. Because the appellant succeeded on the main request, there was no need to consider the auxiliary request or to hold oral proceedings.

Order

For these reasons it is decided that:

1. The decision under appeal is set aside.
2. The case is remitted to the first instance with the order to maintain the patent as granted.

The Registrar:

The Chairman:

G. Magouliotis

R. Young