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**Datasheet for the decision
of 20 November 2018**

Case Number: T 0024/16 - 3.3.03

Application Number: 08803367.5

Publication Number: 2185606

IPC: C08F10/02, C08F2/12, C08F2/14,
C08F6/00, C08F6/20

Language of the proceedings: EN

Title of invention:
SLURRY PHASE POLYMERISATION PROCESS

Patent Proprietor:
INEOS Manufacturing Belgium NV

Opponent:
Total Research & Technology Feluy

Relevant legal provisions:
EPC Art. 83, 56

Keyword:
Sufficiency of disclosure - (yes)
Inventive step - (yes)



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Case Number: T 0024/16 - 3.3.03

D E C I S I O N
of Technical Board of Appeal 3.3.03
of 20 November 2018

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Decision under appeal: **Interlocutory decision of the Opposition**
Division of the European Patent Office posted on
9 November 2015 concerning maintenance of the
European Patent No. 2185606 in amended form.

Composition of the Board:

Chairman D. Semino
Members: D. Marquis
R. Cramer

Summary of Facts and Submissions

I. The appeal lies with the decision of the Opposition Division posted on 9 November 2015 concerning maintenance of European patent N° 2 185 606 in amended form. The decision was based on a main request filed with letter of 25 August 2015.

II. The main request contained twelve claims, claim 1 reading as follows:

"1. Polymerisation process in which polyethylene is produced in slurry in a polymerisation reactor in the presence of a Ziegler Natta catalyst and an activator, and a slurry containing the polymer is withdrawn from the reactor and transferred to a flash tank operating at a pressure and temperature such that at least 50mol% of the liquid component of the slurry is withdrawn from the flash tank as a vapour and at least 98mol%, more preferably at least 98.5mol%, and most preferably at least 99.5mol%, of the vapour withdrawn from the flash tank is capable of being condensed at a temperature of between 15 and 40°C, without compression, wherein a chloromethane of the formula $\text{CH}_x\text{Cl}_{4-x}$ where x is an integer from 1 to 3, is present in the slurry, and the molar ratio of the chloromethane added to the reactor to titanium added to the reactor is between 0.2 and 1."

III. The following documents form, *inter alia*, part of the decision of the opposition division:

F2: Experimental report of the proprietor dated 27 May 2010

F4: WO 9947251

F5: US 5863995

F6: US 2002/0007024

F7: US 5118769

IV. The contested decision, as far as it is relevant to the present decision, can be summarized as follows:

- (a) The main request fulfilled the requirements of Article 123(2) EPC.
- (b) The subject matter of the main request was sufficiently disclosed. In particular, the patent in suit provided sufficient guidance for the definition of the specific temperature and pressure conditions in the flash tank and for the condensation. The temperature was defined in claim 1 and the pressure was discussed in the description. The measurement of the average activity in the reactor(s) belonged to the common general knowledge of the skilled person as indicated in the patent in suit. While there was no example in the patent in suit, the opponent did not provide evidence of an insufficient disclosure with respect to the operation of the claimed process.
- (c) The main request satisfied the requirements of Articles 54 and 56 EPC. With respect to inventive step, F4 was the document representing the closest prior art. The process of F4 differed from the process of the patent in suit in that no by-product suppressor was used. As a consequence, the molar ratio of chloromethane as by-product suppressor to titanium was not indicated in F4 either. The technical problem was to provide a more efficient "medium pressure flash" process. The solution to that problem was the addition of a chloromethane as by-product suppressor in the slurry polymerization

reactor. The addition of chloromethane to the reactor led to a simplified post treatment step in a flash tank, which did not need compression, but allowed recondensation just by cooling.

- (d) F4 taught the skilled person that condensation without compression could be achieved by a heat exchange with a fluid having a temperature between circa 18°C and 57°C. There was no incentive in F4 to add chloromethane as by-product suppressor.
- (e) F5-F7 were directed to the improvement of the polymerization reaction by using a halogenated hydrocarbon to reduce the amount of light components generated during polymerization. The processes of F5 and F6 were not relevant to the polymerization in slurry according to the patent in suit since the process disclosed in these documents was a gas phase polymerization, for which there was no need for a condensation without compression. As to F7, it was silent about the post treatment of the reaction mixture such as recycling, condensation or compression.
- (f) There was thus no hint in the prior art towards the solution disclosed in claim 1 of the main request.

V. The opponent (appellant) lodged an appeal against that decision.

VI. The following documents were additionally filed with the statement setting out the grounds of appeal:

F10: declaration of Aurelien Vantomme dated 18 March 2016

F11: US 5192729

- VII. In a communication sent in preparation of oral proceedings, the Board summarised the points to be dealt with and provided a preliminary view on the disputed issues.
- VIII. Oral proceedings were held on 20 November 2018.
- IX. The arguments provided by the appellant, as far as relevant to the present decision, can be summarised as follows:

Main request

Sufficiency of disclosure

- (a) The patent in suit taught the use of a secondary flash vessel to remove residual volatiles from the polymerization medium, but it was silent about the operating conditions of this second flash tank. There was no guidance in the patent in suit whether the secondary flash vessel was operated at similar operating conditions as the first flash tank, or if it required other conditions to remove residual volatiles. Therefore, the claimed subject matter was insufficiently disclosed.

Inventive step

- (b) F4 was the document representing the closest prior art. The difference between F4 and the opposed patent was the presence of a by-product suppressor and consequently the molar ratio of this suppressor to titanium. No evidence of an effect over the process of F4 was provided. Indeed, the patent in suit did not contain any examples of the claimed

process. As to the experimental report F2, it did not disclose the use of a flash tank, nor did it provide operative conditions corresponding to the withdrawal of the liquid component of the slurry from the flash tank as claimed. F2 only concerned the use of chloroform to limit the production of ethane during the polymerization process. The problem solved was the provision of a polymerization process for which the production of ethane was reduced.

- (c) The solution to that problem, the presence of chloromethane during polymerization in slurry was obvious in view of documents F5, F6, F7 and F11. F11 first established that ethane formation in the course of ethylene polymerization in slurry was known to be detrimental to the catalyst stability and activity. F11 thus provided a motivation to limit ethane formation in polymerizations in slurry. In that respect, F5 taught that the formation of ethane could be reduced by using halogenated hydrocarbons in gas phase polymerization processes. While F5 primarily dealt with gas phase polymerization processes, it also indicated that the ethylene polymerization could be performed in slurry. The skilled person would have thus applied the teaching of F5 concerning gas phase polymerization to a slurry polymerization according to F4. Alternatively, F6 and F7 also taught the use of a halogenated hydrocarbon in polyethylene polymerization processes in order to reduce the formation of ethane. The claimed subject matter thus lacked an inventive step.

X. The arguments of the respondent, as far as relevant to the present decision, can be summarised as follows:

Main request

Sufficiency of disclosure

(a) The secondary flash vessel was an optional addition for removal of residual volatile material of the solid polymer produced in the reaction. It was not referred to in claim 1, and was not part of the invention. Therefore it was of no relevance to the sufficiency of the invention itself. In any case, the operation of such a secondary flash vessel was known in the art, and the skilled person would have had no difficulty determining suitable temperature and pressure values to remove any residual volatile material from the solid polymer. The claimed subject matter was therefore sufficiently disclosed.

Inventive step

(b) F4 was the document representing the closest prior art. The claimed subject matter differed from F4 in the addition of a chloromethane to the reaction and in the specific ratio of the chloromethane to titanium. The experimental report F2 established that the addition of chloromethane led to a reduction of the formation of ethane, a light component that is produced during slurry polymerization. It was straightforward that a reduction of the formation of light components such as ethane meant that the liquid component resulting from the polymerization could then be more

efficiently condensed, in particular without compression. The problem solved was thus to provide an improved process by reducing the need for compression.

- (c) The teaching of F4 would not have led the skilled person to the claimed subject matter as that document was silent about reducing the formation of light components or by-products during polymerisation. F11 did not provide that teaching either as it only pertained to the use of highly active catalysts for the polymerization of ethylene in slurry. The use of halogenated hydrocarbons to reduce the formation of ethane was disclosed in document F5, however specifically in the context of gas phase polymerizations. While F5 also mentioned polymerization in slurry, its teaching only pertained to gas phase polymerization. The skilled person would thus not have applied the teaching of F5 to a polymerization in slurry according to F4. Claim 1 of the main request involved therefore an inventive step.

XI. The appellant requested that the decision under appeal be set aside and that the patent be revoked.

XII. The respondent requested that the appeal be dismissed.

Reasons for the Decision

Main request

1. Sufficiency of disclosure

1.1 The appellant made an objection of lack of sufficiency of disclosure based on the lack of description in the

patent of the operating conditions of the second flash vessel that is disclosed in paragraph 56 of the patent in suit as an option to remove residual volatiles in the process. It was in particular submitted that the person skilled in the art did not know whether this secondary flash vessel functioned at similar operating conditions as those used in the first flash tank mentioned in claim 1 of the main request.

- 1.2 The use of a secondary flash vessel in the polymerization process of the patent in suit is not part of any claim of the main request. Also, the use of a secondary flash vessel is only described in the patent in suit as an optional aspect of the polymerization process (paragraph 56). Since a secondary flash vessel is not required in the claims of the main request, its operating conditions can thus by no means be of relevance to the sufficiency of disclosure of any essential aspect of the claimed subject matter. Under these circumstances, the Board concludes that the objection of lack of sufficiency of disclosure of the appellant is not convincing.
2. The question of whether the claims extend beyond the content of the application as originally filed and the question of novelty are not part of the present appeal as the conclusions thereon of the opposition division have not been disputed.
3. Inventive step
 - 3.1 The patent in suit pertains to the separation and recycling of liquid components present after polymerization of ethylene in slurry, whereby the liquid components withdrawn with the polymer from the reactor are separated in a flash tank which is operated

at a temperature and pressure such that the liquid components can be recondensed just by cooling, without recompression (paragraphs 1, 7 and 9). As an example of such a process, the patent in suit mentions the process of document F4 (paragraph 7), which was the document seen as representing the closest prior art in the contested decision. Both parties acknowledged that F4 represented the closest prior art. The Board does not see a reason to depart from that choice.

- 3.2 Both parties acknowledged that the claimed subject matter differed from the process of F4 in that a chloromethane of formula $\text{CH}_x\text{Cl}_{4-x}$ where x was an integer from 1 to 3, was used in the course of the polymerization in slurry, and in that the molar ratio of the chloromethane to titanium added to the reactor was between 0.2 and 1. It is additionally noted that F4 does not indicate which catalyst is used while the process of claim 1 foresees the presence of a Ziegler-Natta catalyst with an activator.
- 3.3 With regard to the the separation and recycling of the liquid components present in the polymerization slurry, the patent in suit aims more specifically at reducing the formation of light components produced in the course of the polymerization of ethylene, such as ethane (paragraphs 8 and 12). The use of a chloromethane in the polymerization slurry is identified as the solution in paragraph 18.
- 3.4 As evidence of the effect of using a chloromethane, the respondent relied on the experimental report F2. F2 contains two examples of continuous polymerization of ethylene in slurry with a titanium catalyst (Ziegler Natta). In both examples of F2, ethane formation was monitored over time during polymerization and the

weight ratio of ethane generated to polyethylene produced is shown in the graphs of pages 1 and 2. It was not disputed that the graphs of F2 showed that ethane formation was reduced significantly by as much as 44% when chloroform was added to the reactor during polymerization. Although the examples of F2 do not describe the operation of a flash tank followed by the separation of part of the liquid component in the slurry and its condensation without compression, it is credible, based on the general principles of vaporisation and condensation, that the reduction of the amount in ethane generated in the course of the polymerization implies that the resulting slurry contained a smaller amount of light components, allowing to flash the slurry at a temperature and pressure which would allow both vaporisation of a substantial portion of those components and also recondensation thereof without compression (paragraph 8 of the patent in suit).

- 3.5 Under these circumstances the problem that can be formulated in view of F4 is that of providing a process for the polymerization of ethylene in slurry for which condensation of the vapour withdrawn from the flash tank is made easier and with no need of recompression. The solution is the presence of a chloromethane in the slurry as defined in claim 1.
- 3.6 It remains to be determined whether the claimed subject matter was obvious to a person skilled in the art starting from the closest prior art F4. The question posed in that respect is whether the skilled person would have used a chloromethane as defined in claim 1 in order to provide a process for the polymerization of ethylene in slurry for which condensation of the vapour withdrawn from the flash tank was made easier and with

no need of recompression.

3.7 Document F11 does not suggest the solution provided in claim 1 of the main request. While F11 pertains to the same type of polymerization process in slurry as that disclosed in the closest prior art (F11, column 1, lines 8-14 and column 2, lines 20-25), that document does not address the problem posed which pertains to the separation and recycling the liquid components from the slurry after polymerization. While it is true that the passage in column 2, lines 20-27 of F11 mentions that the formation of ethane in the course of the polymerization is undesirable, it is only referred to as affecting the activity of the catalyst for which the solution as proposed in F11 involves the activation of the catalyst with an organoaluminum compound. That solution is not related to the use of by-products suppressants such as chloromethane as in the patent in suit. In that respect, the teaching of F11 is not relevant to the process according to claim 1 of the main request.

3.8 Document F5 was also cited by the appellant as a document providing the solution to the posed problem. F5 pertains to an ethylene polymerization process using a titanium containing Ziegler-Natta catalyst and hydrogen (column 1, lines 10-20). As acknowledged by both parties in appeal, the teaching provided in F5 largely relates to gas phase polymerization processes. That is also clear from the detailed description of the preferred process of F5 which refers to a gaseous reaction mixture (column 8, lines 1-10) and from the examples which all imply a gas phase polymerization process (in particular column 10, line 6). It follows that, the preferred process according to F5 and the only one which is disclosed therein in some details,

unlike the process of the closest prior art F4, does not rely on a flash tank operating at conditions such that the vapour withdrawn can be recycled by condensation without compression. On the contrary, the recycling of the gaseous components present in the polymer according to the preferred process of F5 actively relies on several compressors (compressors (10) and (18), column 8, lines 6 and 16). The operation of the gas phase polymerization process of F5, and in particular the steps relating to the post treatment of the polymer produced in the reactor, differs therefore substantially from that of the polymerization in slurry according to F4. While it is not contested that the skilled person knew that the formation of ethane was a common problem to ethylene polymerization in the presence of hydrogen in both gas and slurry, as suggested in F10, nothing indicates that the teaching of F5 would have been considered for the subsequent use of a flash tank followed by a separation and recycling of the liquid components. Under these circumstances, even if F5 teaches the use of a halogenated hydrocarbon to reduce the formation of ethane in a gas phase polymerization process, the teaching of F5 would have not been considered to be relevant by the skilled person to the problem posed, which assumes the use of a flash tank followed by a condensation without compression. The brief mention, in the passage on column 7, line 15 and line 16 of F5, that the ethylene polymerization process can be a slurry or a solution process does not alter that conclusion. In particular, F5 does not reveal how such a polymerization process in slurry would be carried out since the only teaching provided in F5 pertains to gas phase polymerization. Also, the separation and condensation of the liquid component present in the polymerization slurry is not hinted at in F5. Under these circumstances, the Board

concludes that the teaching of F5 does not render obvious the use a halogenated hydrocarbon in the polymerization of ethylene in slurry as disclosed in F4.

3.9 The Board does also not find in the documents F6 and F7 cited by the appellant in the written procedure, a hint towards the solution to the problem posed.

3.9.1 F6 relates to a polymerization process for the production of polyethylene with low level of extractables (paragraph 2) by incorporating a halogenated hydrocarbon in the polymerization medium as a cocatalyst (paragraphs 35 and 42). While paragraph 7 of F6 mentions that halogenated hydrocarbons were known in the art to reduce the formation of ethane, there is no indication in F6 that the ethane formation during polymerization could be relevant to the separation and condensation without compression of the components withdrawn from the reactor. In F6, the presence of a halogenated hydrocarbon is only generally linked to the production of the desired polyethylene (paragraph 60) and compression of the condensed gases after polymerization is not avoided (paragraph 50). In that respect, the Board does not see why a skilled person would have considered the teaching of F6 to be relevant to the problem posed in the present case.

3.9.2 F7 pertains to a process for the preparation of a polyolefin having a weight average molecular weight of not less than 10,000 by the polymerization of an α -olefin at a reaction temperature lower than the melting point of the polyolefin by a multistage polymerization process in the presence of a Ziegler type catalyst with a high activity and hydrogen, wherein said catalyst comprises three components (A), (B) and (C) (claim 1).

Among the components forming part of the catalyst, component (C) is defined as being selected from a group of halogen-containing compounds selected from a group of halogenated hydrocarbon compounds, halogens and interhalogenous compounds (claim 1). F7 teaches that one of the many effects provided by the polymerization process carried out with the highly active Ziegler type catalyst as defined in claim 1 is to avoid the accumulation of ethane in the polymerization system which rendered inevitable a discharge of the gaseous components outside the system (column 13, lines 7-11). In that respect, the teaching of F7 does not pertain to a process in which the liquid components withdrawn from the reactor are flashed in a flask tank and condensed without compression in order to be recycled. Under these circumstances, the Board does not see why and how the teaching of F7 relative to the reduction of ethane would have been applied to the process of F4 which relies on an operation of the polymerization process for the separation and the recycling of the liquid components present in the polymerization slurry. F7 is thus not relevant to the problem posed.

- 3.10 The Board therefore comes to the conclusion that claim 1 of the main request involves an inventive step.

Order

For these reasons it is decided that:

The appeal is dismissed.

The Registrar:

The Chairman:



B. ter Heijden

D. Semino

Decision electronically authenticated