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Datasheet for the decision of 25 October 2018

Case Number: T 0023/16 - 3.3.03

Application Number: 03767757.2

Publication Number: 1572756

IPC: C08F4/651, C08F4/655, C08F210/16

Language of the proceedings: EN

Title of invention:
CATALYST COMPONENTS FOR THE POLYMERIZATION OF OLEFINS

Patent Proprietor:
Basell Poliolefine Italia S.r.l.

Opponent:
INEOS Europe AG

Relevant legal provisions:
RPBA Art. 12(4)
EPC Art. 56

Keyword:
Late-filed facts - submitted with the statement of grounds of appeal
Inventive step - (no)

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Case Number: T 0023/16 - 3.3.03

D E C I S I O N
of Technical Board of Appeal 3.3.03
of 25 October 2018

Appellant: (Patent Proprietor)	Basell Poliolefine Italia S.r.l. Via Pontaccio 10 20121 Milano (IT)
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Representative:	King, Alex Mathisen & Macara LLP Communications House South Street Staines-upon-Thames, Middx TW18 4PR (GB)
Decision under appeal:	**Decision of the Opposition Division of the European Patent Office posted on 12 November 2015 revoking European patent No. 1572756 pursuant to Article 101(3)(b) EPC.**

Composition of the Board:

Chairman	D. Semino
Members:	D. Marquis
	C. Brandt

Summary of Facts and Submissions

I. The appeal lies with the decision of the opposition division posted on 12 November 2015 revoking European patent No. 1 572 756.

II. European patent EP 1 572 756 was opposed on the grounds that its subject matter lacked novelty and inventive step and found no basis in the application as originally filed.

The decision of the opposition division to revoke the patent was announced at the oral proceedings on 15 October 2015. The decision was based on a main request and on a first auxiliary request, filed together with letter of 13 August 2015, and on a second auxiliary request filed during the oral proceedings before the opposition division.

Claim 1 of the main request read:

"1. A solid catalyst component for the polymerization of olefins comprising Mg, Ti, a halogen and an electron donor compound (ED) belonging to ethers, amines, ketones, nitriles or alkyl esters of C1-C20 aliphatic carboxylic acids, characterized in that the molar ratio Mg/Ti ranges from 7 to 120, the molar ratio ED/Ti ranges from 3.7 to 40 and the titanium atoms derive from titanium tetrahalides or the compounds of formula $TiX_n(OR^1)_{4-n}$, where $0 \leq n \leq 3$, X is halogen and R^1 is C_1-C_{10} hydrocarbon group."

Claim 1 of the first auxiliary request corresponded to claim 1 of the main request from which amines and nitriles were deleted from the definition of the

electron donor compound (ED).

Claim 1 of the second auxiliary request was directed to a process for preparing a solid catalyst component.

III. The following documents were cited inter alia during opposition proceedings:

D1: US 5 290 745
D2: US 3 989 881
D3: Simpson and Vaughan, 2001, Ethylene Polymers, LLDPE, Encyclopedia of Polymer Science and Technology, pages 441-482
D4: WO 00/52068
D5: US 5 055 535

In its contested decision, the opposition division arrived at the conclusion that the main request satisfied the requirements of Article 123(2) EPC and that its claims were novel in view of D1 and D2. Document D5 represented the closest prior art. Claim 1 of the main request differed from D5 in that the ratio ED/Ti was in the range 3.7 to 40. Since it had not been established that that difference resulted in an effect, the problem solved in view of D5 was the provision of an alternative catalyst component suitable for olefin polymerisation. D1 and D2 disclosed similar catalysts components and disclosed ranges for the ratio ED/Ti of 2 to 85 (D1) and 6 to 11 (D2). The claimed subject matter resulted from an arbitrary selection in ranges of ED/Ti known from the prior art. Claim 1 lacked therefore an inventive step. The same conclusion applied to claim 1 of the first auxiliary request. The second auxiliary request was not admitted into the proceedings.

IV. The proprietor (appellant) lodged an appeal against that decision. The appellant submitted with the statement setting out the grounds of appeal, a main request and four auxiliary requests. The main request and the first auxiliary request corresponded to the main request and the first auxiliary request filed with letter of 13 August 2015 that formed part of the decision of the opposition division.

Claim 1 of the second auxiliary request corresponded to claim 1 of the main request with the limitation in the definition of the electron donor (ED) to alkyl esters of C1-C20 aliphatic carboxylic acids.

Claim 1 of the third auxiliary request corresponded to claim 1 of the first auxiliary request with the limitation of the range defining the molar ratio ED/Ti to 4.5 to 30.

Claim 1 of the fourth auxiliary request corresponded to claim 1 of the third auxiliary request from which the definition of the electron donor (ED) was limited to alkyl esters of C_1-C_4 alkyl esters of aliphatic mono carboxylic acids.

With the statement setting out the grounds of appeal the appellant submitted also a declaration of Mr. Morini as document D9.

V. In its reply to the statement of grounds of appeal, the opponent (respondent) requested that the appeal be dismissed.

VI. 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.

VII. Oral proceedings were held on 25 October 2018.

VIII. The arguments provided by the appellant, as far as relevant to the present decision, can be summarised as follows:

Main request

Admittance of document D9

(a) Document D9 was provided in reply to the non admittance by the opposition division of the experimental report filed during the opposition procedure. D9 addressed the reasoning of the contested decision relating to the rejected experimental report and also provided arguments concerning the objection of lack of inventive step in view of D5 as developed in the contested decision. D9 should therefore be admitted into the proceedings.

Inventive step

(b) The claimed subject matter differed from example 3 of D5 in the choice of the molar ratio ED/Ti within the range of 3.7 to 40.

(c) The data contained in D9 showed that the use of a solid catalyst component (INV-A) having an ED/Ti ratio according to claim 1 of the main request (4.2), led to an ethylene/1-butene polyethylene having less xylene solubles (XS) at the same density as a result of a narrower distribution of

1-butene as a function of the molecular weight than a comparative solid catalyst component (COMP-C) with an ED/Ti ratio of 3.6. A comparison of the samples INV-B and COMP-D, which displayed comparable ED/Ti ratios showed that differences in Mg/Ti ratios did not significantly impact the XS of the resulting copolymer. The polymerisation processes performed in D9 involved the same amount of external electron donor so that its presence did not change the conclusion that could be drawn from the comparison of two samples within D9.

(d) D9 thus established that the use of a catalyst component with an ED/Ti ratio within the claimed range led to an ethylene copolymer having a more homogeneous intermolecular comonomer distribution indicative of an improved quality as shown by a smaller quantity of xylene solubles.

(e) Starting from D5 as the closest prior art, the problem was to provide a catalyst leading to a ethylene copolymers with a lower amount of hydrocarbon solubles.

(f) The teaching of D3 relating to the relation between XS and the molecular weight distribution of ethylene copolymers only concerned polymers produced from single site catalysts and not from Ziegler-Natta catalysts as in the patent in suit. The teaching of D3 was thus not relevant.

(g) None of the documents D1 or D2 provided a teaching that led the skilled person to the claimed ED/Ti ratio. The teaching of D1 pertained to the reduction of titanium IV in titanium III in order to prepare ethylene copolymers having reduced

hexane extractable contents. The problem solved in D1 was unrelated to the ED/Ti ratio in the solid catalyst component for which very broad ranges were disclosed in D1. As to D2, it disclosed the presence of an electron donor was used to complex the magnesium compound and the transition metal compound to be reacted each other. Nowhere in D2 it was indicated that the final amount of donor with respect to Ti had an impact on comonomer distribution or on extractables. Actually, the problem connected to high amounts of hydrocarbon soluble matter in LLDPE was totally ignored in D1 which exclusively described the preparation of ethylene homopolymers. Claim 1 of the main request involved therefore an inventive step.

Auxiliary requests

(h) There were no further arguments for the claims of the first to fourth auxiliary requests with respect to the arguments provided for the main request.

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

Main request

Admittance of document D9

(a) The additional examples provided in document D9 suffered from the same defects as the earlier experimental data which the patent proprietor tried to submit at first instance and which were rejected by the opposition division as being late-filed and not relevant. D9 was also not relevant since the preparation method used did not correspond to that

of the closest prior art D5 and the examples differed in more than one parameter. D9 should thus not be admitted into the proceedings.

Inventive step

(b) Document D5 represented the closest prior art. The claimed subject matter differed from example 3 of D5 in that the molar ratio ED/Ti was in the range of 3.7 to 40.

(c) The examples of the patent in suit did not establish the presence of an effect resulting from the choice of the molar ratio ED/Ti in the claimed range, nor did D9. In particular, the experimental evidence provided in D9 was not relevant since the catalyst components disclosed therein also differed from one another by their Mg/Ti ratio and their preparation was not according to D5.

(d) It was common general knowledge that both internal and external electron donors involved in polymerisation processes using Ziegler-Natta catalysts impacted the catalyst activity and the molecular weight distribution of the ethylene copolymers produced. That teaching was shown in D3 and D4 and was corroborated by the examples of the patent in suit (Table 2). The ED/Ti ratio reported in D9 only accounted for the amount of internal electron donor relative to the amount of titanium atoms in the catalyst component. That ratio did not account for any effect resulting from the addition of the same amount of external electron donor to catalyst components with different amounts of titanium during polymerisation in all the examples of D9. The data provided in D9 did therefore not

establish that any effect observed on the ethylene copolymers produced was causally linked to the ED/Ti ratio reported.

(e) The problem solved in view of D5 was thus the provision of an alternative solid catalyst component.

(f) The use of molar ratios of ED/Ti as claimed was already known from the relevant art as shown in D1 and D2. D1 showed that an ED/Ti ratio of 2 to 85 was commonly known for Ziegler-Natta catalysts and its examples disclosed values of 7.8 (example 1) and 10.9 (comparative example A) falling within the claimed range. D2 also disclosed a broad range of ED/Ti ratio and disclosed values of 6.6 (example 1), 7.7 (example 2) and 11.4 (example 8). The prior art thus showed that values within the claimed range were commonly known in the art. Claim 1 of the main request thus lacked an inventive step.

Auxiliary requests

(g) As the appellant had not provided additional arguments for the auxiliary requests, they lacked inventive step for the same reasons as the main request.

X. The appellant requested that the decision under appeal be set aside and that the patent be maintained in amended form on the basis of the main request or, alternatively, on the basis of any of the first to fourth auxiliary requests, all requests filed with the statement of grounds of appeal.

XI. The respondent requested that the appeal be dismissed and that document D9 filed with the statement of grounds of appeal not be admitted into the proceedings.

Reasons for the Decision

Main request

1. Admittance of document D9

1.1 D9 was filed by the appellant with the statement setting out the grounds of appeal, i.e. pursuant to the requirements of Article 12(1) and (2) RPBA. Its admittance into the proceedings, which was contested by the respondent, undergoes the stipulations of Article 12(4) RPBA.

1.2 The appellant submitted that D9 was filed in response to the decision of the opposition division not to admit an experimental report filed during the opposition procedure. Indeed, the relevant part of the contested decision indicates that the opposition division did not admit the experimental report into the proceedings because it was found to be of minor relevance to the question of inventive step as it did not disclose data related to the Mg/Ti ratio and contained only one example of a catalyst component for which the ED/Ti ratio was within the claimed range (point 4 of the contested decision). Moreover, it was filed only shortly before the oral proceedings, so that the opposing party did not have enough time to perform further experimental tests in reply.

1.3 The newly filed document D9 is a declaration of Mr. Morini containing a series of four solid catalyst

components employed in the copolymerisation of ethylene/1-butene according to the same general procedure as that described in the patent in suit. The values of the ratios Mg/Ti and ED/Ti are disclosed for all catalyst components and the document further discusses the presence of an effect resulting from the ED/Ti ratio. It can be concluded that D9 provides examples containing the data that was found to be missing from the experimental report and which appears to be the reason why the opposition division did not admit the experimental report into the proceedings. Moreover, it concerns a central issue regarding the inventive step analysis in the decision, namely the presence of an effect related to a specific range of the ED/Ti molar ratio.

1.4 It can be deduced therefrom that D9 aims at providing a remedy to the deficiencies of the earlier report as raised in the contested decision, that D9 was filed at the earliest possible stage of the appeal proceedings so as to give to the opposing party full opportunity to react and that it addresses a central issue that led to the revocation of the patent.

In view of this, the Board concludes that there is no reason to hold D9 inadmissible pursuant to Article 12(4) RPBA.

2. Inventive step

2.1 The object of the patent in suit was to provide a catalyst component displaying ability to give a homogeneous comonomer distribution, a high polymerization activity and was suitable for gas-phase polymerization (paragraph 14).

2.2 D5 was seen as the closest prior art in the decision of the opposition division. That was not disputed at the oral proceedings in appeal and the Board does not see a reason to deviate from that choice.

2.3 D5 describes the use of monoethers, known as external electron donors in the art, added during the gas-phase polymerization of ethylene to produce linear ethylene homo- or copolymer with narrow molecular-weight distribution, while noticeably maintaining the productivity of the catalyst (column 2, lines 48-59). The process of D5 is based on a Ziegler-Natta catalyst component being the result of the combination of at least one transition metal compound, such as a titanium compound, a magnesium compound, a halogen and possibly an electron donor (column 3, lines 20-45). The electron donor present in that catalyst component, also known as internal electron donor in the prior art, may be chosen in a list comprising aliphatic or aromatic carboxylic acids and their alkyl esters, aliphatic or cyclic ethers such as tetrahydrofuran (THF) and vinyl esters (column 3, lines 45-60).

2.4 In particular, example 3 of D5 discloses the preparation of a solid catalyst component. That preparation comprises in a first step the reaction of a magnesium compound (butyloctylmagnesium), an aluminium compound (tetraisobutylaluminoxane) and tertiobutyl chloride in the presence of an ether (diisoamylic ether), followed by reaction of the precipitate obtained with a titanium compound ($TiCl_4$). The solid catalyst component obtained comprised, according to the table on column 7, lines 11-15, Mg, Ti and Cl (a halogen) and the molar ratio Mg/Ti derivable from that table (13.62) is according to claim 1 of the main request (7 to 120). The presence of diisoamylic ether

as an electron donor on the catalyst component was not in dispute by the parties. Its amount in the solid catalyst component is however not disclosed in example 3, nor is it derivable from the whole of D5.

2.5 It was not in dispute that the solid catalyst component according to claim 1 of the main request only differed from that disclosed in example 3 of D5 in the value of the molar ED/Ti ratio. While it ranges from 3.7 to 40 in claim 1, it is not disclosed at all in D5.

2.6 The problem solved over the closest prior art had to be determined on the basis of the information made available. In that respect, it was not disputed that the data contained in the patent in suit did not show the presence of an effect over the closest prior art D5, the appellant relying on the data contained in document D9 for that purpose.

2.6.1 Document D9 describes the preparation of four solid catalyst components from magnesium dichloride, ethyl acetate as internal donor and titanium tetrachloride. The amounts of the reactants used in the preparation is disclosed in the table of page 3 of D9. The table also reports the molar ratios ED/Ti (AcOEt/Ti) and Mg/Ti obtained for each of the catalyst component, showing that the samples INV-A (ED/Ti=4.2 and Mg/Ti=9.9) and INV-B (ED/Ti=5.0 and Mg/Ti=8.1) were according to claim 1 of the main request whereas COMP-C (ED/Ti=3.6 and Mg/Ti=10.8) and COMP-D (ED/Ti=4.8 and Mg/Ti=6.7) were not. In particular, the solid catalyst components INV-A an COMP-C were seen by the parties to be particularly relevant since their differences in ED/Ti ratio was seen to represent the difference between claim 1 of the main request and the closest prior art

D5.

2.6.2 The above mentioned solid catalyst components were employed in the copolymerisation of ethylene/1-butene in the presence of triethylaluminium/diethylaluminum chloride (TEA/DEAC) or trimethylaluminum (TMA) as a cocatalyst according to the general procedure described in example 22 of the patent in suit. That polymerisation process also involved the use of THF as external donor at a total Al/THF molar ratio of 5. The specific conditions and results are reported in the table of page 4 of D9. In figures 1 and 2 the graph of the GPC/FTIR characterization carried out on the copolymers produced with the four solid catalyst components in combination with TEA/DEAC as cocatalysts are reported.

2.6.3 With regard to these results, the appellant submitted that the use of the solid catalyst component INV-A, according to claim 1 of the main request, resulted in an ethylene/1-butene copolymer with less xylene solubles at the same density as a result of a narrower distribution of 1-butene as compared to the solid catalyst component COMP-C (conclusions, page 5 of D9) and that these improvements originated in the ED/Ti ratio defined by the amount of electron donor relative to the amount of titanium in the solid catalyst component. With respect to the electron donor mentioned by the appellant in that context, it needs to be underlined that both claim 1 of the main request and the description of the patent in suit, by reference to the electron donor used in the determination of the ratio ED/Ti in paragraph 16 and a further reference to an external electron donor in paragraph 35, are congruent in that the electron donor mentioned by the appellant in the ED/Ti ratio refers to the electron

donor used in the preparation of the solid catalyst component only, known as an internal electron donor in the art, i.e. ethyl acetate in the case of the samples of D9. By contrast, the external electron donor added to the catalyst system during polymerisation is not included in the ED/Ti ratio since it is not part of the definition of the solid catalyst component.

2.6.4 Beside the issue concerning the meaningfulness of a comparison of the catalyst components INV-A and COMP-C, especially in view of the their differing Mg/Ti ratios, the prominent question with respect to D9 is whether the effect derived by the appellant from the properties of the copolymers can effectively be attributed to the specific range of ED/Ti as defined in claim 1 of the main request or not.

2.6.5 In that regard, the density, the XS and the distribution of 1-butene in the polymer as a function of the molecular weight are properties that are determined in D9 from the polyethylene produced in the presence of the solid catalyst component with a defined ED/Ti ratio, the cocatalyst and the external electron donor. These properties of the polyethylene are not only influenced by the amount of internal electron donor relative to titanium of the catalyst component but also by any amount of external electron donor added during the polymerisation process. Indeed, the use of external electron donors in the polymerisation process of olefins with Ziegler-Natta catalysts is long known in the art. The effects of using an external electron donor on the polymerisation parameters, in particular on the molecular weight distribution and the activity of the catalyst, was not only known from D5 (column 2, lines 21-30) but is also generally described in D4 (passage starting on page 2, line 16 until page 3,

line 11). That passage of D4 further attests that internal and external electron donors were both known to have an effect on the activity of the catalyst and the molecular weight distribution of the polyethylene produced. That is confirmed by the fact that an external electron donor is also used to modify the polymer in the patent in suit "In many cases, particularly when an external donor is used, the comonomer is also well distributed in and among the chain as shown by the substantial lowering of the density even in respect of relatively minor amount of comonomer introduced" (paragraph 39).

2.6.6 The impact the external electron donor may have on the properties of the polyethylenes produced in D9 is particularly relevant since the appellant ascribes the technical effect to the relative amount of internal electron donor to titanium present in the catalyst component. As noted by the appellant, the absolute amount of external electron donor (THF) added in the course of the polymerisation process according to D9 was the same for all solid catalyst compounds (total Al/THF molar ratio of 5; last paragraph on page 3 of D9). However, the amount of external electron donor relative to the titanium content in the solid catalyst component, which was the definition chosen by the appellant to establish the technical effect, will ultimately differ for each solid catalyst component tested in D9 since the amount of titanium present in each of INV-A, INV-B, COMP-C and COMP-D is different, as shown in table on page 3 of D9.

2.6.7 It follows from the above that the variations of the density, the XS and the distribution of 1-butene in the polymer as a function of the molecular weight cannot be unambiguously attributed to variations of the ED/Ti

ratio since a further unknown and changing amount of external electron donor relative to titanium was present in course of each polymerisation reported in D9. Under these circumstances, it cannot be concluded on the basis of D9 that any effect reported therein is causally linked to the ED/Ti ratio which is the feature differentiating the claimed solid catalyst component from that of example 3 of D5. These effects can therefore not be taken into account for the formulation of the technical problem solved over the closest prior art D5.

2.7 Under these circumstances, the problem that can be formulated for the main request in view of the information made available can only be the provision of a further solid catalyst component for the polymerisation of olefins.

2.8 The question that has then to be answered is whether the solution provided to that problem in the main request, namely the selection of an ED/Ti ratio of 3.7 to 40, is inventive in view of the prior art, in particular in view of D1 or D2.

2.8.1 D1 is a document relating to ethylene polymerisation using a Ziegler-Natta catalyst (column 1, lines 5-10). The catalyst system in that document comprises magnesium and titanium trichloride derived from the reduction of titanium tetrachloride (claim 1). The Mg/Ti ratio of these catalysts is in the range of 1 to 56, which overlaps that of 7 to 120 according to claim 1 of the main request. The catalyst system also contains an electron donor ED which can be an ether (THF in claim 3). A general formula describing these catalysts is disclosed in column 4, lines 45-55 of D1:

$$Mg_mTiCl_p[ED]_q$$

wherein ED is an electron donor compound, m is 1 to 56, preferably 1.5 to 5, p is 5 to 115, preferably 6 to 13, and q is 2 to 85, preferably 3 to 12. It is immediately apparent that the ED/Ti ratio as defined in claim 1 of the main request corresponds to q in the formula according to D1. D1 therefore teaches that solid catalyst components having an ED/Ti ratio of 2 to 85 were known in the art for the same type of polymerisation process as that described in the patent in suit. In particular, the examples of D1 disclose catalyst components wherein the ED/Ti values are 7.8 (example 1) and 10.9 (comparative example A) falling within the claimed range. Under these circumstances, the use of a solid catalyst system characterized by an ED/Ti ratio of 3.7 to 40 as claimed in the main request, completely included in a range already known in the art and for which no effect was established, cannot be seen as inventive.

2.9 The Board concludes that claim 1 of the main request lacks an inventive step.

First to fourth auxiliary requests

3. Inventive step

3.1 The statement setting out the grounds of appeal contains a section on pages 2 to 6 relating to inventive step of claim 1 of the main request. The statement setting out the grounds of appeal concludes by stating that in view of these arguments the claims of the main request and those of the various auxiliary requests involved an inventive step. No arguments specific to the auxiliary requests in view of inventive step were thus provided by the appellant in writing. The appellant also stated during the oral proceedings

before the Board that there were no further arguments relating to inventive step for the solid catalyst component according to claim 1 of the first to fourth auxiliary requests other than the arguments provided for claim 1 of the main request. In view of this, no further arguments were provided by the respondent for the auxiliary requests.

3.2 The Board arrived at the conclusion that claim 1 of the main request lacked inventive step taking into account the arguments provided by the parties (see point 2 above). As there were no further arguments to consider for the assessment of inventive step of claim 1 of the first to fourth auxiliary requests, there is no compelling reason for the Board to conclude otherwise than for the main request on inventive step.

3.3 Under these circumstances, the Board concludes that claim 1 of the first to fourth auxiliary requests lacks an inventive step for the same reasons as those given for the main request (Article 56 EPC).

Order

For these reasons it is decided that:

The appeal is dismissed.

The Registrar: The Chairman:

L. Malécot-Grob D. Semino

Decision electronically authenticated