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**Datasheet for the decision
of 13 October 2016**

Case Number: T 0704/14 - 3.3.03

Application Number: 08725145.0

Publication Number: 2118193

IPC: C08K5/00, C08J5/18, C08K5/098

Language of the proceedings: EN

Title of invention:

BARRIER PROPERTIES OF SUBSTANTIALLY LINEAR HDPE FILM WITH
NUCLEATING AGENTS

Patent Proprietor:

Equistar Chemicals, LP

Opponent:

THE DOW CHEMICAL COMPANY

Relevant legal provisions:

EPC Art. 56
RPBA Art. 13(1), 13(3)

Keyword:

Inventive step - obvious combination of known features
Late-filed auxiliary requests - change of subject-matter



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Case Number: T 0704/14 - 3.3.03

D E C I S I O N
of Technical Board of Appeal 3.3.03
of 13 October 2016

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Decision under appeal: **Decision of the Opposition Division of the European Patent Office posted on 16 January 2014 rejecting the opposition filed against European patent No. 2118193 pursuant to Article 101(2) EPC.**

Composition of the Board:

Chairman D. Semino
Members: F. Rousseau
C. Brandt

Summary of Facts and Submissions

I. The appeal lies from the decision of the opposition division rejecting the opposition filed against European patent EP 2 118 193.

II. Claim 1 as granted reads as follows:

"A method for improving the barrier properties of a polyethylene film, said method comprising converting into a film a mixture comprising a substantially linear, high density polyethylene (HDPE) having a long chain branching index (LCBI) ; determined according to the method of the description less than or equal to 0.5 and a melt flow ratio (MFR ; ASTM D 1238) less than or equal to 65, and a nucleating agent selected from the group consisting of glycerol alkoxide salts, hexahydrophthalic acid salts, and mixtures thereof, wherein the film has at least a 15% improvement, compared with a control film which is made from the same substantially linear HDPE but does not contain the nucleating agent, in the water vapor barrier property (WVTR ASTM F1249 @ 100% humidity) or in the oxygen barrier property (OTR ASTM D 3985 @ dry conditions)."

III. The following documents had been cited during the first instance proceedings:

D1: "A novel nucleating agent for polyethylene", article of M. Horrocks and C. Kerscher presented at the International Conference on Polyolefin 2007, 25 to 28 February 2007, Houston TX.

D2: W. Todd, "Variables that affect/control high-density polyethylene film oxygen-moisture barrier",

Journal of Plastic Film & Sheeting, Volume 19, July 2003, 209-220

D11: Experimental report submitted by the opponent with letter of 21 October 2013

D12: R. Shroff et al., Macromolecules 1999, 32, 8454 - 8464

D13: Material Data Sheet M6020

D14: W. Todd et al., "Maximize Barrier Performance of reduced-gauge HDPE films", Article presented at the ANTEC conference in 1999

IV. According to the reasons of the decision, the skilled person was given with the patent in suit sufficient information concerning the experimental conditions and the key properties of the suitable HDPEs, meaning that the skilled person would have no problem in selecting suitable HDPEs from commercially available sources in order to carry out the claimed invention. The requirements for sufficiency of disclosure were therefore met. Experimental report D11 had been submitted late and its relevance questioned, as it did not contain sufficient information on the process details and materials used. Accordingly, it was not admitted into the proceedings. As to inventive step, D1 was the closest prior art, from which the method of the patent in suit was distinguished by a selection of ranges for both LCBI and MFR. In view of the experimental evidence contained in the patent in suit, the objective problem solved over D1 was the provision of an improved method for improving the barrier properties of HDPE films. D2 indicated the effect of MFR and LCBI on the barrier properties, but did not disclose the selection of concrete values or a range, let alone the use of a nucleating agent. D12 disclosed commercially available HDPE-MVTR films having LCBI values defined in claim 1 of the patent in suit, but

however did not disclose their MFR values. D13 provided information about HDPE resin M6020 disclosed in D12, but did not disclose its MFR value or its use as a material in barrier films. D14 taught that HDPE M6020 was known for its use as barrier film in food-packaging applications due to its superior WVTR properties, said document also disclosing a MFR value of 44 for that material. Although HDPE resins having the required LCBI and MFR values were commercially available before the priority date of the opposed patent, the cited literature did not suggest to use a) a nucleated HDPE, b) only certain nucleating agents while at the same time c) HDPEs having certain LCBI and MFR values in order to provide an improvement of at least 15 % of the barrier properties of the films. An inventive step was therefore acknowledged.

- V. An appeal against that decision was lodged by the opponent. The statement of grounds of appeal included a more detailed version of D11 (hereafter D11a).
- VI. The rejoinder of the respondent (patent proprietor) included a data sheet of product Alathon L5906 (D15).
- VII. With letter of 8 April 2015 the appellant submitted new arguments, as well as D14a indicated to be the same article as D14, but published earlier in ANTEC 1999.
- VIII. After a communication of the Board in preparation of oral proceedings, the respondent submitted with letter of 12 September 2016 an auxiliary request. Claim 1 of said auxiliary request read as follows (additions as compared to granted claim 1 in underline, deletions in strikethrough):

"1. A method for improving the barrier properties of a

polyethylene film, said method comprising converting into a film a mixture ~~comprising~~ of a substantially linear, high density polyethylene (HDPE) having a long chain branching index (LCBI); determined according to the method of the description less than or equal to 0.5 and a melt flow ratio (MFR; ASTM D 1238) less than or equal to 65, and a nucleating agent selected from the group consisting of glycerol alkoxide salts, hexahydrophthalic acid salts, and mixtures thereof, said nucleating agent being present in an amount from 0.01 wt% to 1 wt% of the weight of said substantially linear HDPE and said mixture optionally containing antioxidants, UV absorbents, flow agents or other additives in an amount of less than 10 wt% of the total mixture, wherein the film has at least a 15% improvement, compared with a control film which is made from the same substantially linear HDPE but does not contain the nucleating agent, in the water vapor barrier property (WVTR ASTM F1249 @ 100% humidity) or in the oxygen barrier property (OTR ASTM D 3985 @ dry conditions)."

- IX. The oral proceedings took place on 13 October 2016 at the beginning of which the appellant submitted a second auxiliary request. Claim 1 of the second auxiliary request corresponded to claim 1 of the auxiliary request filed with letter of 12 September 2016 with the deletion of "hexahydrophthalic acid salts, and mixtures thereof" from the list of nucleating agents.

- X. The submissions of the appellant, as far as they are relevant for the decision, can be summarized as follows:

Main request

- (a) The starting point for assessing inventive step was the method described on page 13 of D1 which disclosed the addition of Hyperform® HPN-20E, i.e. the same nucleating agent used in the patent in suit, in order to increase the barrier performances of a HDPE film barrier grade. It was admitted, however, that D1 did not disclose the LCBI and MFR of that HDPE.
- (b) Experimental report D11a showed that the values of LCBI and MFR defined in operative claim 1 were not sufficient to obtain barrier properties superior to those achieved in D1. In addition the experimental data of the patent in suit did not compare compositions according to claim 1 as granted with compositions of D1 already having a WVTR improvement of about 20% relative to non-nucleated HDPE. Thus, in view of the teaching of D1, it seemed that the level of improvement in WVTR and/or OTR reductions, which also was part of the solution as a required feature in claim 1, could not be part of the problem definition. Accordingly, the technical problem solved by the subject-matter of claim 1 of the patent in suit was to find another HDPE that would result in an improvement of water vapour and oxygen barrier properties.
- (c) Based on the theoretical guidance provided in D2 to improve barrier properties of a HDPE film by reducing MFR, increasing the melt index (MI) and

minimizing LCBI the skilled person looking at commercially available HDPE barrier resins would have found a suitable HDPE to solve that problem. In particular, he would have considered resin M6020 of Equistar which was commercially available and had been used for example 1 of the contested patent as acknowledged by the patent proprietor. Having regard to documents D12 to D14a which showed that M6020 of Equistar was known to have good barrier properties and to meet the criteria set out in D2 for maximizing barrier properties, the skilled person would have found obvious to use that resin in order to solve the problem posed. In fact, it was logical for the skilled person to use the HDPE resins reported in D2 to have better barrier properties, as the skilled person was concerned with the absolute values for the barrier properties. Moreover, contrary to what had been argued by the respondent, the teachings of D2 and D1 were not in contradiction, because D1 taught that addition of the nucleating agent led to shorter crystallization time and smaller crystal size, whereas D2 taught that the relaxation times of the resin per se could be enhanced so as to provide more randomly orientated crystalline regions. Hence, nothing in D2 prevented adding a nucleating agent.

- (d) Therefore, the subject-matter of claim 1 lacked an inventive step.

First auxiliary request

- (e) The first auxiliary request should have been submitted earlier. Moreover, it was objectionable under Articles 84 and 123(3) EPC, as well as under

Rule 80 EPC, and the amendments introduced did not overcome the objection for lack of inventive step over D1. Accordingly, it should not be admitted into the proceedings.

Second auxiliary request

(f) The entire discussion in the opposition and appeal proceedings had put the focus on a nucleating agent different from those now remaining in claim 1. The only remaining exemplified embodiment was example 7 for which no comparison was provided with the closest prior art. As that subject-matter would require an entire new assessment of inventive step, the second auxiliary request submitted in course of the oral proceedings should not be admitted into the proceedings.

XI. The submissions of the respondent, as far as they are relevant for the decision, can be summarized as follows:

Main request

(a) In relation to sufficiency of disclosure, it was confirmed that the starting material in example 1 of the patent in suit was the product M6020 commercially available from the patent proprietor Equistar, as shown in D13. Furthermore, the claimed LCBI and MFR were not sufficient to produce the claimed barrier effect, the other required feature being the presence of the nucleating agent as claimed.

- (b) The closest state of the art was the method of improving the barrier properties of a HDPE film described on page 13 of D1.
- (c) The technical problem solved by the subject-matter of claim 1 of the patent as granted in suit was an improvement of the barriers properties of HDPE polymers already known for their barrier properties, which as shown by comparative example 1 of the patent in suit were not sufficient.
- (d) Contrary to the teaching of D1, it was not sufficient to add the nucleating agent Hyperform® HPN-20E in order to solve that problem, since it was also necessary as shown by the examples and comparative examples of the patent in suit to select HDPE resin having LCBI and MFR values within the ranges defined in claim 1. None of the documents cited by the opponent pointed to that combination of features, including the improvement of 15% of the barrier properties. Furthermore, D1 taught a fast crystallization using the nucleating agent, whereas D2 taught fast relaxation in order to allow more time to crystallize and form more randomly orientated crystalline regions. Therefore, the teachings of D1 and D2 were contradictory.
- (e) Hence an inventive step should be acknowledged for the subject-matter of claim 1.

First auxiliary request

- (f) The first auxiliary request was based on the main request, but contained amendments meant to restrict the subject-matter of the claims in in view of the comments in points 6.3.3, 6.5.2 (b) and (c) of the

Board's communication concerning the possibility of using additional polyethylene resins. Therefore, it constituted a timely and appropriate answer to the points raised by the Board. Consequently, it should be allowed into the proceedings.

Second auxiliary request

(g) The second auxiliary request was late filed but nevertheless it should be admitted to the proceedings because the Board's communication contained a hint that a subject-matter restricted to embodiments using a glycerol alkoxide salt as nucleating agent would not be obvious in view of the prior art cited. The class of nucleating agents now defined in claim 1 was already defined in granted claim 1 and the restriction to that class of nucleating agents in the second auxiliary request could not be considered to disadvantage the opponent. Furthermore, as shown by example 7 and (now comparative) example 1 of the patent in suit, that class of nucleating agents provided even better results than the nucleating agent used in D1, justifying that the second auxiliary request should be admitted into the proceedings.

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

XIII. The respondent requested that the appeal be dismissed, or, alternatively, that the decision under appeal be set aside and the patent be maintained on the basis of the set of claims according to the first auxiliary request as filed with letter dated 12 September 2016, or on the basis of second auxiliary request as filed during the oral proceedings on 13 October 2016.

Reasons for the Decision

Main request - Inventive step

1. Having regard to the assessment of sufficiency of disclosure, it was questioned during the appeal proceedings whether or not the wording of operative claim 1 allowed one or more additional polymeric film forming components to be employed in addition to the HDPE resin mandatorily used. However, it is sufficient for the purpose of the present decision as shown below to give reasons for the decision only with respect to methods in which the sole polymeric film forming component is the HDPE defined in claim 1. As these methods are surely covered by the claim and they are found not to be inventive, it is not necessary to establish whether further methods are also covered.

Closest prior art

- 1.1 Both in the decision under appeal and in the arguments of the parties document D1 is considered as the closest prior art. The Board sees no reason to take a different approach.
- 1.2 D1 concerns as the patent in suit HDPE films having barrier performances used in the area of cereal liners and dry food packaging. It discloses on page 13 a method of enhancing the barrier properties by using a nucleating agent for the HDPE which is a hexahydrophthalic acid salt as defined in operative claim 1, namely Hyperform® HPN-20E, the same compound as used in examples 1 to 6 of the patent in suit. Figure 16 on page 13 shows in particular an improvement

in MTRV above 15% through the use of Hyperform® HPN-20E with barrier grade HDPE. While the method of measurement of MTRV is not indicated in D1, the Board is persuaded that this is not relevant with regard to the relative improvement, because claim 1 as granted does not define an absolute value, but only an improvement as a percentage variation. Novelty of the method defined in operative claim 1 over that prior art is, however, not disputed as D1 does not disclose the LCBI and the MFR of the HDPE resin disclosed on its page 13.

Problem and solution

1.3 Having regard to the disclosure of D1, the appellant submitted that the technical problem solved by the subject-matter of claim 1 of the patent in suit was the provision of another HDPE that would result in an improvement of water vapour and oxygen barrier properties, whereas the respondent formulated the technical problem solved over D1 as an improvement of the barriers properties of HDPE polymers known to have barrier properties.

1.4 Thus, there is consensus that the incorporation of the nucleating agent according to claim 1 into the HDPE resin defined therein brings about an increase of the barrier properties of films made out of said resin. The Board also notes that an increase of the water vapour or oxygen barrier properties of the film resulting from that method is part of the definition of the subject-matter of claim 1, so that that technical effect must be considered to be achieved when assessing inventive step of the subject-matter of claim 1. However, the desired improvement is already obtained in D1, which already discloses also the use of a nucleating agent

according to claim 1. It is moreover noted that it was not put forward that the films obtained with the method of operative claim 1 would exhibit water vapour or oxygen barrier properties whose absolute values are superior to those of the film obtained in D1. Also taking into account that the claimed subject-matter is not directed to a product, i.e. films, but to the activity of making them (method) the Board concludes that the technical problem which can be considered to be successfully solved over the disclosure of D1 is the provision of further methods of increasing the water vapour or oxygen barrier properties of HDPE films.

- 1.5 The solution to this problem as defined by the method of operative claim 1 is characterized by the use of an HDPE selected from those having a LCBI ≤ 0.5 and a MFR ≤ 65 in combination with a nucleating agent selected from the groups consisting of glycerol alkoxide salts or hexahydrophthalic acid salts added to said resin before making a film, the method resulting in at least a 15% improvement in the water vapour or oxygen barrier property, compared with a control film which is made from the same substantially linear HDPE but does not contain the nucleating agent.

Obviousness

- 1.6 It remains to decide whether or not the proposed solution to the objective problem underlying the patent in suit is obvious in view of the state of the art.
- 1.7 D1, in which the specific method considered to represent the closest prior art is disclosed, is an article concerning the use of Hyperform® HPN-20E as nucleating agent for polyethylene (abstract). Hyperform® HPN-20E is explained to have an impact on

the crystallization behaviour of HDPE, in particular on the growth behaviour and size of the spherulites as indicated in the last paragraph of page 4, Figure 5 on page 5 and page 6. D1 describes in particular in relation with the method described on page 13 and taken as starting point for assessing inventive step the effects of nucleation with Hyperform® HPN-20E when preparing blown HDPE and LLDPE films, in particular in respect of their barrier properties (page 7, first paragraph and page 13). Hence, it can be inferred not only from D1, but also on the basis of the skilled person general knowledge according to which water and oxygen molecules diffuse easily through amorphous regions of the HDPE films, whereas crystal regions of the HDPE films are impervious to water and oxygen transmission (see D2, page 210) that the improvement of barrier properties of HDPE and LLDPE films reported in D1 is the consequence of the nucleation and modified crystallization process. Accordingly, D1 discloses that the improvement of barrier properties of the specific HDPE resin tested in D1 is the result of the modification of crystallization behaviour brought about by Hyperform® HPN-20E.

1.8 Moreover, the abstract and the conclusion of D1 point out that Hyperform® HPN-20E is a new chemistry that shows breakthrough performance in polyethylene nucleation, meaning that the teaching of D1 with respect to the method of improving properties such as water vapour or oxygen barrier properties is not limited to the sole specific HDPE and LLDPE tested in D1, but is suggested in that document to extend to other at least similar resins.

1.9 Consequently, the idea of adding Hyperform® HPN-20E to other known HDPE resins in order to improve the water

vapour or oxygen barrier properties of films made thereof, which means in fact providing further methods increasing the water vapour or oxygen barrier properties of HDPE films, is already implicit from the disclosure of D1.

- 1.10 In order to put into practice that invitation of D1 to test nucleation of other HDPE resins known to provide barrier properties with Hyperform® HPN-20E the skilled person would naturally turn to commercial HDPE resins known for that purpose or select HDPE resins on the basis of criteria known to be favourable to barrier properties. Striving for HDPE films having sufficient barrier properties for food packaging, which is the purpose underlying the present invention, he would in particular consider HDPE resins having low MFR values and low LCB Indices, as well as high MI, i.e. three factors recommended in D2 for maximizing barrier properties in blown HDPE films (see page 219, conclusions). In particular, he would try the HDPE resin M6020 described in D12, D13 and D14 which, as shown in those documents and acknowledged by the appellant, was commercially available from Equistar at the date of priority of the application and is recommended for blown film processes and applications including dry food packaging in view of its water vapour transmission rate (see D13 and D14). Furthermore, it is also not disputed that resin M6020 of Equistar was known, as confirmed in D12, D13 and D14, to have low LCBI, low MFR and high MI, i.e. some of the factors recommended in D2 to maximize WVTR barrier in blown HDPE films, its LCBI and MFR falling within the ranges of values defined in operative claim 1.

1.11 Moreover, the teachings provided by D2 and D1 concern two separate phenomena, the first concerning the ability of the melt (i.e. the molecules of the resins) exiting the machine to relax faster which allows to maximize the random orientation of the crystalline lamella formed (see D2, page 211, middle paragraph), whereas the second concerns the number of nucleation centres increased by adding the nucleating agent leading ultimately to smaller crystals (see D1, pages 1 to 5). Accordingly, the argument of the respondent that the teachings of D1 and D2 are contradictory so that the skilled person would not combine them fails to convince. On the contrary, the skilled person would be guided to take advantages of these two aspects which have been indicated in prior art documents D1 and D2 to contribute to an improvement of the barrier properties of HDPE.

1.12 Consequently, starting from the disclosure of D1 and wishing to put into practice the invitation of D1 to test nucleation of further HDPE resins known to provide barrier properties with Hyperform® HPN-20E, the skilled person would have found obvious to try resin M6020 commercially available from Equistar, a resin which falls within the definition of operative claim 1 and which, as acknowledged by the patent proprietor, is in fact the resin used in example 1 of the patent in suit.

1.13 Finally, it is not disputed, as confirmed by the arguments brought forward in relation to sufficiency of disclosure and the teaching of the patent in suit that an improvement of at least 15% of the barrier properties would be the result of using a resin falling within the definition of claim 1, in particular resin M6020 used in example 1 of the patent in suit.

1.14 Accordingly, starting from the method described in D1 and wishing to solve the problem defined in above point 1.4, the skilled person would arrive in an obvious manner to a method falling within the ambit of operative claim 1, namely the method wherein the HDPE resin is M6020, commercially available from Equistar. Accordingly, claim 1 which encompasses an obvious embodiment does not meet the requirement of Article 56 EPC.

First auxiliary request - admittance and inventive step

2. The first auxiliary request was submitted with letter of 12 September 2016, i.e. one month before the oral proceedings before the Board and after the communication of the Board setting out its preliminary view of the case had been received. Therefore it represents an amendment to a party's case and its admittance to the proceedings undergoes the stipulations of Articles 13(1) and 13(3) RPBA. The first auxiliary request is based on granted claims 1-16, the amendments aiming at excluding the use of polyethylene resins other than the specific HDPE resin defined in claim 1 of the main request. That amendment proposed is therefore a direct and appropriate reaction to the Board's question raised in points 5.3.2. and 6.3.3 of the board's communication in relation to the issues of sufficiency of disclosure and inventive step when considering that claim 1 of the main request did not contain any limitation defining the amount of HDPE of claim 1 and that it allowed the use of further polyethylene resins. Moreover, it focuses the claim to the core embodiment, which has been largely discussed by the parties and cannot represent a surprise for the appellant. Hence, the board exercises its discretion

and admits the request into the appeal proceedings (Article 13 RPBA).

- 2.1 However, the subject-matter of claim 1 still encompasses the embodiment which according to above point 1.14 is considered to be obvious. In this respect the specification of a broad range for the amount of the nucleating agent represents only an arbitrary selection of a reasonable quantity without any specific surprising effect. Hence, for the same reasons as above, the subject-matter of claim 1 lacks an inventive step and the first auxiliary request is not allowable.

Admittance of the second auxiliary request

3. The admittance to the proceedings of the second auxiliary request submitted at the beginning of the oral proceedings also undergoes the stipulations of Articles 13(1) and 13(3) RPBA. That second auxiliary request corresponds to the first auxiliary request wherein the claims methods have been modified inter alia by deleting the embodiments relating to the use of hexahydrophthalic acid salts, therefore restricting the claims methods to those using glycerol alkoxide salts as nucleating agent.
- 3.1 The whole discussion in the opposition proceedings, including the contested decision, and in the appeal proceedings up to the oral proceedings before the Board was focused only on the use of hexahydrophthalic acid salts as nucleating agent. Therefore there was no indication, even implicit that the patent proprietor might wish to continue prosecution of the case on the basis of claims relating only to a different type of nucleating agents, namely glycerol alkoxide salts. The fact that the patent in suit contains an exemplified

embodiment relating to glycerol alkoxide salts which corresponds to the best result in terms of barrier properties and that no document was cited against patentability of embodiments concerning glycerol alkoxide salts cannot justify the course of action of the patent proprietor in submitting that new request only at the oral proceedings before the Board. To the contrary, based on the submission of the patent proprietor that the exemplified embodiment relating to glycerol alkoxide salts in light of the specification was obviously the best embodiment of the patent in suit in terms of improvement of the barrier properties, i.e. the effect underlying the claimed invention, one should even more expect from the patent proprietor to timely submit a set of claims limited to that class of nucleating agents, if it was sought to safeguard patent protection for methods relating to them. In the absence of any set of claims limited to their use up to the oral proceedings before the Board, let alone any argument of the patent proprietor suggesting that an auxiliary request might be submitted in respect thereof, the other party could only conclude that the patent proprietor had no interest in pursuing the claimed subject-matter with respect to that sole class of glycerol alkoxide salts nucleating agents.

- 3.2 Consequently, the admission to the proceedings of that second auxiliary request would start a completely new discussion on inventive step and therefore raise issues which the other party or the Board could not reasonably be expected to deal without adjournment of the oral proceedings and therefore run counter to the principle of procedural fairness and to the need for procedural economy. Accordingly the Board finds it appropriate to exercise its discretion under Article 13 RPBA by not

admitting the second auxiliary request into the proceedings.

Order

For these reasons it is decided that:

1. The decision under appeal is set aside.
2. European patent No. 2118193 is revoked.

The Registrar:

The Chairman:



B. ter Heijden

D. Semino

Decision electronically authenticated