BESCHWERDEKAMMERN DES EUROPÄISCHEN PATENTAMTS

BOARDS OF APPEAL OF THE EUROPEAN PATENT OFFICE

CHAMBRES DE RECOURS DE L'OFFICE EUROPEEN DES BREVETS

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File Number: T 595/91 - 3.3.3

Application No.: 83 303 453.1

Publication No.: 0 099 646

Title of invention: Preparation of ethylene terpolymers

Classification: C08F 210/02

D E C I S I O N
of 2 December 1992

Applicant: Exxon Research and Engineering Company

Opponent: 01 BASF Aktiengesellschaft, Ludwigshafen
02 Hoechst Aktiengesellschaft, Frankfurt(Main)

Headword:

EPC Articles 54, 56

Keyword: "Novelty (confirmed)"
"Inventive step (main request: no) - obvious modification of known products"
"inventive step (auxiliary request: no) - process generally known in the art"

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Case Number : T 595/91 - 3.3.3

D E C I S I O N
of the Technical Board of Appeal 3.3.3
of 2 December 1992

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Decision under appeal :
Decision of the Opposition Division of the European Patent Office dated 23 January 1990, issued in writing on 12 June 1991 rejecting the oppositions filed against European patent No. 0 099 646 pursuant to Article 102(2) EPC.

Composition of the Board :

Chairman : F. Antony
Members : C. Gérardin
R. Schulte

Summary of Facts and Submissions

I. The mention of the grant of the patent No. 99 646 in respect of European patent application No. 83 303 453.1 filed on 15 June 1983 and claiming the priority of 18 June 1982 from an earlier application in the United Kingdom, was published on 17 September 1986 on the basis of 12 claims, Claim 1 reading as follows:

"An ethylene/vinyl acetate/iso olefin terpolymer characterised by having a $\bar{M}_n$ of from 1500 to 5500, having from 6 to 15 methyl groups per 100 methylene groups other than the methyl groups on the vinyl acetate and comprising from 10 to 20% by weight of vinyl acetate and from 3 to 15% by weight of an iso olefin of the general formula:

$$R\text{-}CH_2\text{-}\overset{\displaystyle CH_3}{\overset{|}{C}}\text{=}CHR' \qquad (I)$$

wherein R and R' which may be the same or different each represent hydrogen or an alkyl group having from 1 to 4 carbon atoms."

Claims 2 to 4 are dependent claims directed to preferred terpolymers according to the main claim.

Further, Claim 5 reads as follows.

"A process for the preparation of an ethylene terpolymer comprising from 10 to 20% vinyl acetate and from 3 to 15 wt% of an iso olefin of the general formula I as defined in claim 1 and having a $\bar{M}_n$ of from 1500 to 5500 and 6 to 15 methyl groups per 100 methylene groups other than the methyl groups in the vinyl acetate, in which ethylene is polymerised with vinyl acetate at a pressure

of at least 500 bar and at a temperature of at least 150°C in the absence of any significant amount of inert solvent and in the presence of from 7 to 25% by weight, based on the total reaction mixture, of the iso olefin."

Claims 6 to 9 concern preferred embodiments of that process. As to Claims 10 to 12, they refer to a distillate fuel oil composition containing such terpolymer as well as to specific uses of such terpolymer.

II. On 26 February 1987 and 16 June 1987 Opponents 1 and 2 respectively filed Notices of Opposition against the grant of the patent and requested revocation thereof in its entirety for lack of novelty and inventive step under Article 100(a) EPC. These objections, which were emphasised and elaborated in several later submissions as well as during oral proceedings, were based essentially on the following documents:

(1) GB-A-1 368 159,
(2) Journal of Polymer Science, Part A-1, 1966, (4), 881 to 900,
(3) US-A-3 961 916,
(1-bis) GB-A-1 263 151.

III. By a decision of 23 January 1990, with written reasons posted on 12 June 1991, the Opposition Division rejected the oppositions. It was first stated in that decision that the terpolymer as defined in the main claim was novel; more specifically, the description made of the terpolymers in Claim 13 of document (1-bis) was too general to anticipate the specific terpolymer according to the patent in suit. Regarding the issue of inventive step, it was then stated that none of the documents relied upon by the Opponents gave a hint towards these specific terpolymers. First, document (3) was limited to

dipolymers; secondly, the terpolymers disclosed in document (1-bis) were wax growth arresters, not wax growth stimulators; thirdly, as far as the terpolymers described in document (1) are concerned, besides the fact that the amount of comonomer was too high, they were not wax growth stimulators either.

IV. On 7 August 1991 the Appellant (Opponent 2) filed a Notice of Appeal against this decision and paid the prescribed fee at the same time.

(i) In the Statement of Grounds of Appeal filed on 14 October 1991 it was first pointed out that Claim 1 of the patent in suit was directed to a product characterised by a certain combination of features; it followed that the applications envisaged for that product were irrelevant for the issue of novelty. Novelty could not be acknowledged since all the features of the claimed terpolymer as well as of the process for preparing it were disclosed in document (1-bis). Further, it was underlined that document (3) not only mentioned the possibility to include an alpha-olefin in the copolymer, but even specified the criteria to be met by wax growth stimulators and wax growth arresters; in particular, in view of their molecular weight the suitability of the claimed terpolymers as wax growth stimulators could not be regarded as surprising.

(ii) In a later statement filed on 20 November 1992 the Appellant relied additionally on two documents which had played a minor role in the opposition procedure, GB-A-1 263 152 (document (2-bis)) and DE-A-2 345 807 (document (3-bis)), and further sought to introduce a new document, the "British Standard Method for Determination of cold filter plugging point", in

support of the objection of lack of inventive step. Simultaneously the Appellant informed the Board that it would not attend the oral proceedings scheduled on 2 December 1992.

V. Although the Respondent (Patentee) requested two extensions of time to submit a written statement, it did not file an Appeal Counterstatement, but merely made a conditional request for oral proceedings. In its oral submission the Respondent underlined the differences between the copolymer defined in Claim 13 of document (1-bis) on the one hand, and the claimed terpolymer on the other hand, and argued along the line of a selection invention. In the first place, a branched chain olefin did not necessarily mean an α-olefin; in the second place, taking into account that document (1-bis) was published in 1969, nuclear magnetic resonance could only be interpreted as proton nuclear magnetic resonance, which meant that the degree of branching did not have the same technical significance as in the patent in suit, wherein that parameter was determined from the ^{13}C nuclear magnetic resonance spectrum of the terpolymer. As far as the issue of inventive step was concerned, document (3) should be regarded as the closest state of the art; on that basis, the technical problem underlying the patent in suit was to provide a further nucleator molecule, which could be produced by a solvent-free process. Although document (1) mentioned the possibility of working without solvent, the main teaching of that citation was the choice of a specific transfer agent; nor could document (1-bis) give the skilled man any hint, since it dealt basically with dipolymers prepared by a solution process.

In accordance with these arguments the Respondent asked the Board to also consider the maintenance of the patent in suit on the sole basis of the process Claims 5 to 9 as

granted. The Board, however, expressed serious doubts regarding the inventiveness of such process in view of the teaching of DE-A-1 924 823 (document (5) in the opposition procedure) which showed that ethylene, vinyl acetate and isobutylene could be copolymerised without solvent.

VI. Opponent 1, as a party to the proceedings as of right, had been duly summoned to the oral proceedings, but did not attend them.

VII. The Appellant requested that the decision under appeal be set aside and that the patent be revoked.

The Respondent requested that the appeal be dismissed and that the patent be maintained as granted, or, as an auxiliary request, that the patent be maintained on the basis of Claims 5 to 9 as granted and a description to be adapted.

Reasons for the Decision

1. The appeal complies with Articles 106 to 108 and Rule 64 EPC and is admissible.

2. The Board has examined the document referred to by the Appellant for the first time in the statement filed on 20 November 1992 in order to determine its relevance, namely its evidential weight compared with that of the documents filed in time, and has found that it was not sufficiently relevant to be taken into consideration. This document, therefore, will be disregarded hereinbelow pursuant to Article 114(2) EPC.

Main Request

3. The patent in suit concerns an ethylene/vinyl acetate/isoolefin terpolymer, a process for the preparation thereof and specific applications thereof. Since the Opposition Division in its decision and the Respondent during oral proceedings regarded document (3) as the closest state of the art, the Board has decided to follow that approach too. This citation describes fuel compositions based on a middle distillate petroleum fraction whose fluidity and filterability at low temperature are improved by the addition of a combination of wax growth stimulator and wax growth arrester (column 1, lines 43 to 63). Both additives are typically copolymers of ethylene and vinyl acetate which differ by the amount of vinyl acetate and the molecular weight; whereas a relatively high molecular weight copolymer with a relatively low content of vinyl acetate acts as a wax growth stimulator (also referred to as "nucleator" hereinbelow, or as "nucleating agent"; cf. (3) column 1, lines 49 to 50), a relatively low molecular weight copolymer with a relatively high content of vinyl acetate acts as a wax growth arrester (column 5, lines 15 to 21; column 6, lines 4 to 28). The wax growth stimulator with a molecular weight of 500 to 10 000 and a vinyl acetate content of 1 to 30% is said to be particularly effective (column 5, lines 26 to 30). Although such wax growth stimulators in combination with the above-defined wax growth arresters give satisfactory results as additives for distillate fuel oil compositions, their preparation occurs by solution polymerisation, i.e. a process which extends over several hours and involves the use of large amounts of solvents (column 7, lines 8 to 44; Example 1, column 8, lines 12 to 24).

In the light of this shortcoming the technical problem underlying the patent in suit can thus be seen in the provision of further nucleators which can be obtained in shorter reaction times by a process not involving the use of solvents, without impairing the effectiveness of the additive combination.

According to Claim 1 of the main request this problem is to be solved by an ethylene/vinyl acetate/isoolefin terpolymer (i) having a number average molecular weight of from 1500 to 5500, (ii) having from 6 to 15 methyl groups per 100 methylene groups other than the methyl groups on the vinyl acetate, (iii) comprising 10 to 20% by weight of vinyl acetate, and (iv) comprising 3 to 15% by weight of an isoolefin (v) as defined by general formula (I).

The experimental data in the patent in suit demonstrate that terpolymers characterised by such a combination of features (i) to (v) provide an effective solution to the above-defined technical problem.

4. According to the Appellant ethylene terpolymers meeting the compositional conditions (i) to (v) are disclosed in document (1-bis).

This document describes copolymers of ethylene and vinyl alcohol esters, particularly vinyl acetate, having a reduced level of branching. More specifically, this citation concerns copolymers comprising 3 to 40 molar proportions of ethylene per molar proportion of a mixture of 30 to 99 mole % of said ester with 70 to 1 mole % of a branched or straight chain α-monoolefin having 3 to 16 carbon atoms; these terpolymers have a number average molecular weight of 1000 to 2900 and 6 or less methyl terminating side branches per 100 methylene groups, as measured by nuclear magnetic resonance, other than methyl

groups on said ester (page 1, lines 40 to 72; page 2, lines 16 to 24; Claim 13). As pointed out by the Appellant in the Statement of Grounds of Appeal (page 7, Table), this means that a terpolymer containing 3 to 40 moles of ethylene, 0.3 moles of vinyl acetate and 0.7 moles of monoolefin is known, in other words that depending on the isoolefin - isobutene or isooctene - terpolymers having the following weight percent compositions are explicitly described: 59.47 to 94.5/2.17 to 18.24/3.3 to 27.75 respectively 44.64 to 91.5/2.1 to 13.7/6.4 to 41.6. To a large extent there is thus correspondence between these ranges and the above features (i), (iii) and (iv).

By contrast, as underlined by the Respondent during oral proceedings, even if one leaves the straight chain α-monoolefins out of account, the reference to a branched α-monoolefin in document (1-bis) cannot be equated with the isoolefin as defined in the patent in suit, for a branched α-monoolefin does not necessarily have such a structure. It follows that the isoolefins of formula (I) cannot be regarded as deriving implicitly from the compounds envisaged in the prior art teaching, so that novelty of the claimed subject-matter can be acknowledged on the basis of feature (v).

Under these circumstances it is not necessary to consider to what extent the method of determination of the degree of branching - proton nuclear magnetic resonance or ^{13}C nuclear magnetic resonance - has an influence on the actual value of that parameter (feature (ii)); nor is it necessary to decide whether ^{13}C nuclear magnetic resonance is actually used to analyse terpolymers prepared from an isoolefin other than isobutylene (compare patent in suit, page 3, lines 8 to 24 and Claim 2).

5. It still remains to be decided whether the above terpolymer characterised by the combination of features (i) to (v) involves an inventive step with regard to the teaching of the documents relied upon by the Appellant.

5.1 A first information available to the skilled man looking for alternative copolymers suitable as wax growth stimulators can be found in document (3) itself.

Although the typical polymers mentioned in that citation are defined as copolymers of ethylene with an unsaturated ester monomer, it is indicated that an olefin hydrocarbon having 3 to 30 carbon atoms, preferably an α-monoolefin having 3 to 8 carbon atoms, may also be used as comonomer (column 4, lines 27 to 58). It is admittedly arguable how this passage should be interpreted, i.e. whether it means that such α-monoolefin could be used additionally to ethylene and vinyl acetate, as understood by the Appellant, or whether it means that this unsaturated hydrocarbon should be used instead of the vinyl ester, as contended by the Respondent. In the Board's view, however, whatever was actually intended by the authors, the skilled man would read that passage having in mind the general remarks concerning the features which have an influence on the properties of the wax growth stimulators, i.e. specifically the composition, the molecular weight and the degree of ethylene branching of the ethylene/ester copolymers (column 5, lines 58 to 66). The introduction of units derived from such α-monoolefin into the polymer backbone must thus in any case be regarded as compatible with the use of the resulting copolymer as a wax growth stimulator; since this is the only alternative embodiment dealing with possible compositions of the copolymer in the whole document, it would provide to the skilled man looking for alternative wax growth stimulators a strong incentive to operate along the same line.

5.2 This conclusion concurs with the teaching of document (1) which describes copolymers of ethylene and an unsaturated ester, in particular vinyl acetate, having a molecular weight of from 500 to 4500, as well as the preparation thereof. The main process feature ensuring this low molecular weight consists in the addition of 0.1 to 5% by volume, based on the monomer mixture, of a polymerisation regulator having a transfer constant C_S of at least $1x10^{-2}$ (page 1, line 39 to 59; page 2, lines 45 to 62; Example 2), which is said to be discussed in further detail in the article identified as document (2) in the present proceedings. Among the many chain transfer agents suitable for the process disclosed in document (1), one finds butene-1 in document (1) and isoolefins, such as isobutylene, in document (2). The copolymers referred to in document (1) are mainly used as additives for petroleum fractions, which thereby retain their fluidity and filterability at low temperatures (page 3, lines 27 to 49).

Although the amount of vinyl acetate in these copolymers, i.e. 30 to 75% by weight, would rather suggest a suitability as wax growth arresters than as wax growth stimulators, this teaching is relevant for the solution of the above-defined problem for two reasons. The first is that document (3) gives the same qualitative compositional definition of wax growth arresters and wax growth stimulators and that, in particular, the above-mentioned modification with an α-monoolefin applies to both groups of copolymers; for the skilled man this would clearly mean that low molecular weight wax growth stimulators can equally be prepared by the use of appropriate chain transfer agents, such as those known from documents (1) and (2). The second reason results from the knowledge, as will appear hereinafter when dealing with the auxiliary

request, that olefin monomers serve a dual purpose in that they may act both as chain transfer agents as specified in documents (1) and (2) and as building units of the polymer backbone as envisaged in document (3). It follows that the skilled man is aware that, unlike the other polymerisation regulators mentioned in documents (1) and (2), only olefins enable products different from copolymers of ethylene and vinyl acetate to be prepared and, thereby, offer a solution to the above-defined problem.

5.3 In the Board's view, there was thus a strong incentive for the skilled man to incorporate such an olefin into the copolymers of ethylene and vinyl acetate described in document (3) by using it in the preparation of the copolymer. The determination of the most suitable type of olefin and the amount thereof would not require more than routine experimentation based on trial and error, so that the choice of isoolefins defined by the general formula (I) cannot be regarded as inventive. Since the resulting terpolymers, when used as additives for distillate fuel oil compositions, do not show any advantageous property with regard to the copolymers known from document (3), as conceded by the Respondent, the claimed subject-matter does not involve an inventive step.

6. Claim 1 not being allowable, the main request must be rejected, since a request can only be considered as a whole.

Auxiliary Request

7. As mentioned above (point V, last paragraph), the submission during oral proceedings of an auxiliary request directed to process claims only led the Board to introduce into the procedure document (5), as numbered by the Opposition Division.

8. This document is concerned with an improved process for the copolymerisation of ethylene and vinyl acetate, the improvement consisting in the use of 0.2 to 10% by weight of copolymer of a compound selected among propylene, isobutylene and butene-1 as well as mixtures thereof (page 3, paragraphs 1 and 4). Although this olefin is primarily intended to act as a chain transfer agent regulating the molecular weight, IR analysis of the resulting copolymer shows that the latter is a true terpolymer, i.e. that this olefin behaves as well as an additional comonomer (page 4, paragraph 4; page 9, paragraph 2); in particular, the product obtained by copolymerising ethylene and vinyl acetate in the presence of isobutylene shows sub-units derived from these three compounds (page 5, third formula). Although various methods would be suitable to carry out that reaction, the clearly preferred one is a high pressure polymerisation process (page 11, paragraph 1; Example 1).

This means that more than 12 years before the date of priority of the patent in suit (date of publication of document (5): 4 December 1969), it was known that the copolymerisation of ethylene and vinyl acetate in the presence of up to 10% by weight of an isoolefin and in the absence of solvent yielded a terpolymer, wherein the units derived from the isoolefin were incorporated along the polymer backbone. The adjustment of the amount of vinyl acetate in the terpolymer to meet the requirements of wax growth stimulators specified in document (3) cannot by itself be regarded as an inventive feature, so that the process as defined in Claim 5 does not involve an inventive step.

9. Claim 5 not being allowable, the same applies to dependent process Claims 6 to 9 according to the auxiliary request, which represent preferred embodiments of the subject-matter of the main process claim and thus fall with it.

Order

For these reasons, it is decided that:

1. The decision under appeal is set aside.

2. The patent is revoked.

The Registrar: The Chairman:

E. Görgmaier F. Antony