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D E C I S I O N
of 4 February 2000

Case Number: T 1048/97 - 3.3.3

Application Number: 92116495.0

Publication Number: 0534480

IPC: C08F 6/10

Language of the proceedings: EN

Title of invention:
Steam purging of granular EPDM resins

Patentee:
UNION CARBIDE CHEMICALS & PLASTICS TECHNOLOGY CORPORATION

Opponent:
Bayer AG Konzernbereich RP Patente und Lizenzen

Headword:
-

Relevant legal provisions:
EPC Art. 56

Keyword:
"Inventive step (yes) - remote technical field"

Decisions cited:
T 0150/82

Catchword:
-

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Case Number: T 1048/97 - 3.3.3

D E C I S I O N
of the Technical Board of Appeal 3.3.3
of 4 February 2000

Appellant: (Opponent)
Bayer AG
Konzernbereich RP
Patente und Lizenzen
D-51368 Leverkusen (DE)

Representative:
-

Respondent: (Proprietor of the patent)
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Decision under appeal:
Decision of the Opposition Division of the European Patent Office posted 17 September 1997 rejecting the opposition filed against European patent No. 0 534 480 pursuant to Article 102(2) EPC.

Composition of the Board:

Chairman: C. Gérardin
Members: B. ter Laan
A. Lindqvist

Summary of Facts and Submissions

I. Mention of the grant of European patent No. 0 534 480 in respect of European patent application No. 92 116 495.0, filed on 25 September 1992, claiming priority from an earlier application in the United States (766405 of 27 September 1991), was published on 11 October 1995 on the basis of ten claims, Claim 1 reading:

"A process for substantially removing and recovering unreacted diene monomer from ethylene propylene diene monomer (EPDM) resin produced in a fluidized bed reactor which comprises:
(a) directing said resin into a purging zone in contacting relationship with steam in an amount and at a velocity sufficient to remove substantially all unreacted diene monomer from said EPDM resin said resin being introduced at a temperature above the temperature of the steam in said purging zone;
(b) discharging said EPDM resin having substantially all of the diene monomer removed from said purging zone;
(c) discharging a stream including steam and diene monomer from said purging zone and thereafter introducing said stream into a first steam and diene monomer separation zone to condense said steam into water and to separate diene monomer from said water leaving trace amounts of diene monomer in said water;
(d) discharging and recovering said diene monomer removed from said water from said separation zone; and
(e) discharging said water containing trace amounts of diene monomer from said separation zone."

Claims 2 to 10 referred to preferred embodiments of the process according to Claim 1.

II. On 28 May 1996 a Notice of Opposition against the granted patent was filed, in which the revocation of the patent in its entirety was requested on the grounds set out in Article 100(a) EPC.

The opposition was, *inter alia*, supported by the following documents:

A EP-A-0 047 077,

B DE-A-1 795 396 and

C US-A-4 332 933.

III. By a decision issued in writing on 17 September 1997, the Opposition Division rejected the opposition. It held that

(a) Novelty had not been contested and it was acknowledged.

(b) Regarding inventive step, none of the cited documents was considered to form an appropriate starting point for a discussion on the basis of the problem-solution approach, since none of them referred to the same problem as the patent in suit. No combination of those documents was possible nor would any such combination result in the claimed subject-matter. Therefore, the presence of an inventive step was accepted.

IV. On 13 October 1997 the Appellant (Opponent) lodged an appeal against the above decision and paid the prescribed fee simultaneously. The Statement of Grounds of Appeal was filed on 9 January 1998.

The Appellant, in writing and during oral proceedings held on 2 February 2000, argued essentially as follows:

Document E (GB-A-1 104 740), which had not been previously mentioned during the proceedings, but which was referred to in the patent specification in suit, was considered to be the closest state of the art. It should be admitted to the proceedings in the light of standard jurisprudence of the boards of appeal.

Document E disclosed a process for the removal of dienes from elastomeric polymers, for example terpolymers of ethylene/propylene/diene, by steam treatment of the polymer. This process was especially suitable for polymers with the particle size such as produced in fluid bed processes. Heating the polymer prior to the steam treatment to a temperature higher than that of the steam to prevent condensation of water onto the polymer particles was described in Document C. Document A disclosed a similar process for the treatment of polyolefins with an inert gas such as steam. The recovery of the removed diene from the steam was a process which the skilled person would envisage without hesitation. Therefore, the claimed process was not inventive.

In case document E would not be admitted to the proceedings, document C was regarded as the closest state of the art since it concerned the removal of

volatile matters from olefin polymers. It taught to heat the polymer by superheated steam under fluid bed conditions. Since steam stripping of EPDM was generally known, it was obvious to apply the process described in document C also to EPDM polymer.

V. The Respondent (Proprietor), in its written and oral submissions, argued essentially as follows:

The patent in suit concerned the removal of residual diene monomer from an EPDM resin produced in a solvent-free polymerization process. In view of the sticky nature of EPDM resin, this was a difficult process and it was not unproblematic to heat the polymer even further in order to remove the residual diene, which heating was an essential feature of the claimed invention. The purging step, too, was carried out in the absence of solvents. Therefore, and also in the light of the patent specification, the steam treatment was a dry process.

Document E disclosed a classical steam distillation process, which was not a dry process. It did not suggest to heat the polymer prior to steam treatment. Since the other documents all referred to crystalline polyolefins, which were of a different nature than EPDM, no combination of document E with any of them would be envisaged by the skilled person. For the same reason, if document E would be left out of consideration, the other documents could not serve to teach the solution of any problem regarding EPDM polymers. Therefore, the claimed process was inventive.

VI. The Appellant requested that the decision under appeal

be set aside and the patent be revoked.

The Respondent requested that the appeal be dismissed and that the patent be maintained.

Reasons for the Decision

1. The appeal is admissible.

The wording of the claims

2. According to Claim 1, the patent in suit concerns a process for substantially removing and recovering unreacted diene monomer from ethylene propylene diene monomer (EPDM) resin **produced** in a fluid bed reactor (emphasis added). A claim to a product produced or obtained by a certain process is usually referred to as a product-by-process claim. In such a case, the product itself must be patentable, independent from the process by which it is made, even if the process itself is patentable (see Decision T 150/82, OJ 1984, 309).

2.1 Present Claim 1 however, is a process claim directed to the treatment of a previously prepared product. Hence it is not a product-by process claim. The question therefore arises whether the claim should be read as including the process steps employed to obtain the product to be treated, or whether it should be regarded as embracing the treatment of all possible products that may be obtained by the indicated process.

2.2 In the patent specification, reference is first made to "current production facilities for producing EPDM resin". They are based on slurry or solution processes, in which the solvent, not the polymer, forms the major part of the reactor effluent (column 1, lines 22 to 29). The removal in three steps of the unreacted monomers and solvent from the polymer is described (column 1, line 30 to column 2, line 20).

Then, a more recent development of producing EPDM by gas phase polymerization is described, which results in larger particles than in the other processes, rendering inefficient the steam stripping process described earlier.

In the gas phase process, residue removal is based on contacting the polymer with an inert gas (column 2, lines 21 to 45).

The additional problems of using ethylidene norbornene (ENB) as the diene are mentioned in column 2, line 54 to column 3, line 39.

After a short discussion of document E (column 3, lines 40 to 47), the objects of the invention are defined (column 3, lines 48 to 54) and the proposed solution (column 3, line 55 to column 4, line 56) is given. Special emphasis is laid on the absence of a liquid phase in the purging process and its particular amenability to purging EPDM polymer particles which are produced in a gas phase fluid bed process (column 4, lines 41 to 51).

In the detailed description of the invention, making

reference to the sole drawing present in the patent specification, it is stated that the "EPDM resin from the reactor containing unreacted ENB monomer is introduced into [the] heater..." of the purging system (column 5, lines 22 to 31).

In the four examples further illustrating the invention an EPDM resin is prepared by the gas phase process disclosed in US-A-4 994 534. "After polymerization, the granular resin is transferred from the reactor to the heater and heated..." (column 8, lines 10 to 11).

2.3 Thus, the information contained in the patent specification boils down to the statement that the steam stripping process, which was satisfactory for the removal of residues from EPDM resins produced in solution or slurry processes, could not be applied to EPDM resins produced with the more recently developed gas phase fluid bed process, for which the process now being claimed was meant. From the description of the polymerisation system as well as the examples, it is clear that the resin is produced in a gas phase fluid bed reactor and as such transferred to the purging system, without any addition of solvents or the like. This information leaves no room for any other interpretation than that the polymerization of EPDM in a gas phase fluid bed process is an integral part of the invention. Therefore, the subject matter of Claim 1 should be read as a process in which first EPDM is gas phase polymerized in a fluidized bed reactor and then it is purged according to the steps indicated as a) to e) (Article 69(1)EPC).

The issues of novelty and inventive step will be

evaluated in this light.

The documents

3. Three documents were cited within the nine months opposition period.

3.1 Document A describes a process for removing unpolymerized gaseous monomers from a solid polyolefin polymer containing said gaseous monomers which comprises:

- conveying said polymer to a purge vessel in a first gas stream, said gas being inert to said polymer and monomers and containing substantially no oxygen;

- feeding purge gas to said purge vessel, said purge gas being inert to said polymer and monomers and containing substantially no oxygen;

- countercurrently contacting said polymer and said purge gas in said purge vessel to produce a second gas stream having a reduced amount of said gaseous monomers;

- and recycling a portion of said second gas stream to said purge vessel (Claim 1).

The polymers are specified as being low density ethylene (co)polymers, the comonomer(s) being at least one á-olefin having 3 to 8 carbon atoms (page 1, line 18 to page 2, line 18; page 5, lines 1 to 22), and may be produced by means of a fluid bed gas phase

process (page 2, line 31 to page 3, line 3) with a catalyst based upon a titanium compound, a magnesium compound and an electron donor supported on a carrier (page 2, lines 19 to 24). All examples disclose the treatment of an ethylene/1-butene copolymer, which is not an EPDM resin.

The purging process involves countercurrently contacting the solid polymer particles containing the unreacted monomer gases with an inert gas stream, whereby the monomer gases are stripped away from the polymer particles (page 6, lines 14 to 20; description of drawings on page 8, line 5 to page 9, line 16). As the purge gas, preferably nitrogen is used (page 12, lines 13 to 17). The purge gas containing unreacted monomer is partially flared off, the rest being either used as the conveying gas for the resin particles (Figure 1) or reintroduced into the purge vessel as the purge gas (Figure 2). The temperature of the resin in the purge vessel is said not to be critical. It may be obtained directly from the polymerization reaction at a temperature of about 80 to 85°C. It is economically not desirable to add heat to the resin before feeding it to the purge vessel; the temperature should be kept below the softening point of the polymer (page 10, lines 1 to 21). There is no mention of separating the purge gas and the monomers.

3.2 Document B discloses a process for the removal of volatile constituents from a particulate polyolefin by treating a powdery layer of the polymer particles, which have an average diameter of 100 to 1000 µm, with an inert gas stream at a temperature between 80°C and at least 5°C below the crystalline melting point of the

polymer, while keeping the polymer layer in vigorous motion (Claim).

The polyolefin can be polyethylene, polypropylene, ethylene/propylene copolymer, or a copolymer of ethylene and/or propylene with butene-1. It can be produced by gas phase polymerisation in the absence of any solvents (page 2, first full paragraph). In the sole example polypropylene produced in a gas phase process is subjected to a treatment for the removal of volatile constituents.

The treatment involves leading a gas stream through the polymer particles with a velocity high enough to keep those particles in motion, in particular in a fluidized bed (paragraph bridging pages 2 and 3). As purging gas, nitrogen, carbon dioxide and steam are mentioned. The temperature of the gas is between 80°C and at least 5°C below the crystalline melting point of the polymer (page 2, second full paragraph). Recycling of the gas or recovery of the removed volatile constituents is not suggested.

3.3 Document C discloses a process for reducing the content of catalyst residues, halogenated compounds and organic substances contained in the crystalline olefinic homopolymers and copolymers prepared by (co) polymerization of olefins with catalysts obtained by reacting an organometallic Al compound with a Ti compound, in which the Ti compound is supported on a Mg halide, and an electron-donor compound is combined with the Ti compound and/or the Mg compound, said process consisting in causing a stream of superheated steam having a temperature of from 105° to 140°C and a

pressure of from 0.1 to 10 kg/cm^2 gauge, to pass over the polymer in powder form in a ratio by weight between steam and polymer ranging from 0.10 to 1, and maintaining the polymer at such a temperature so as to prevent steam condensation (Claim 1).

The process is particularly useful for polymers of crystalline polymers of propylene and, in general, of lower alpha-olefins (column 3, lines 51 to 58) made with the above mentioned catalyst. In the sole example, polypropylene produced in the presence of hexane as a solvent is subjected to such a treatment.

The treatment consists in passing, under fluidized bed conditions (column 2, lines 34 to 40), a stream of superheated steam having a temperature of 105 to 140°C over the polymer in powder form and maintaining the polymer at such a temperature as to prevent any steam condensation (column 2, lines 3 to 7). The heat required to increase the temperature of the polymer from the feeding temperature to the temperature of the fluid bed, as well as the latent vaporization of the volatile substances, are supplied to the system by suitable heat exchangers immersed in the fluid bed (column 2, lines 41 to 46). According to a preferred embodiment, the steam containing the substances separated from the polymer is condensed to water in which the substances are dissolved or undissolved, whereas an equivalent amount of fresh steam is regenerated and fed back to the purge vessel (column 2, lines 8 to 12; Figure 2; column 2, lines 52 to 68).

3.4 Document E describes a process of separating an elastomeric olefin polymer from a solution or

dispersion containing 20 to 25% by weight of solvent or dispersant wherein the average polymer particle size is reduced to below 3 mm and the solution or dispersion is stripped with steam, the ratio of fresh to recycled steam being from 1 to 5 (Claim 1). Initially, the solution or dispersion may be in the form of an aqueous suspension formed by contacting crude polymerization product with hot water so as to evaporate volatile material (Claim 2).

The polyolefins, which include homopolymers, copolymers as well as terpolymers, are generally produced in suspension or solution in a suitable solvent (page 1, lines 17 to 21); in the case of the preparation of terpolymers involving a diene as the third monomer, an excess of the latter may be conveniently used as the polymerization medium. The polymer can then be separated by means of steam stripping (page 1, lines 22 to 29). The process of document E aims at reducing the steam consumption for removing the solvent from solutions or dispersions of elastomeric polymers by means of stripping. This is done by reducing the size of the polymer particles (page 1, lines 30 to 39). In all the examples the polymer particles are first ground and then immersed in water, through which dispersion steam is blown. The used steam can be recycled and combined with fresh steam; superheating the recycle steam further reduces steam consumption (page 2, lines 1 to 58).

Novelty

4. Novelty has been recognised by the Opposition Division and the parties did not contest that part of the decision. In the light of the disclosure of the documents on file (see point 3 above), the Board also comes to the conclusion that the claimed subject-matter is novel.

Closest state of the art

5. According to established case law of the Boards of Appeal, the question of inventive step is to be decided on the basis of the problem-solution approach and, to that end, it has first to be determined which document represents the closest state of the art and which is the technical problem underlying the patent in suit.

5.1 The patent in suit concerns the steam purging of granular EPDM resins. From the above discussion of the documents (point 3) it is clear that documents A, B and C all refer to olefin polymers not containing any diene comonomer and not belonging to the class of elastomeric polymers. In view of the major differences in composition, which in turn involve major differences in properties with respect to EPDM resins, none of those citations qualifies as relevant prior art in the sense that the skilled person would consider it as an appropriate starting point for defining the technical problem. Consequently, their disclosures cannot render the claimed subject-matter obvious.

5.2 Only document E concerns a purging process for elastomeric olefin polymers. This document was,

however, mentioned well after the nine months opposition period, in the Statement of Grounds of the Appeal. Although the Respondent first objected to the introduction of document E, he then explained in detail why that new citation, which was well-known to the Proprietor (cf. patent specification, column 2, line 3), was in fact irrelevant for the outcome of the case. At the oral proceedings the Respondent's preliminary objection was outweighed by that submission and the fact that it clearly appeared from the discussion that document E was the only citation dealing with the preparation of EPDM polymers and the elimination of the residual diene termonomer. Therefore, the Board decided to allow the Appellant to rely on document E for the presentation of his case.

5.3 According to the general teaching of document E, in order to reduce the consumption of steam during the removal of undesired substances, the elastomeric olefin polymer particles should be reduced before being purged (Claim 1; page 1, lines 30 to 39; lines 63 to 71). Preferably purging is then carried out by leading steam through an aqueous suspension of the polymer (Claim 2; page 1, lines 47 to 51; all examples). This process is applied to polymers obtained as solutions or suspensions (Claim 1; page 1, lines 9 to 29), hence not to polymers produced by a gas phase process. In fact, the process described in document E concerns the steam stripping process that, according to the patent specification, is less suitable for gas phase products. Since document E pertains to the problem of reducing steam consumption during purging of solution or suspension polymerized products and not to the purging of gas phase polymerized EPDM, it is evident that this

citation, too, does not qualify as an appropriate starting point for defining the technical problem underlying the patent in suit. For that reason, document E cannot prejudice the inventiveness of the claimed subject-matter.

Problem and solution

6. Even if, following the Appellant's line of argument, document E would be regarded as the closest document for the sole reason that, like the patent in suit, it concerns the removal of undesired substances form elastomeric polymers, one would not come to another conclusion.

6.1 In the light of the background art reported in the patent specification (column 1, line 22 to column 3, line 46) and the comments given by the Respondent during the oral proceedings, the technical problem underlying the patent in suit is to provide a process for the removal of diene monomers from EPDM resins produced by a gas phase polymerization process in an economical manner (see also point 2 above).

6.2 According to the patent in suit this problem is solved by the five step purging process as defined in Claim 1.

6.3 The examples and comparative examples in the patent show that the various aspects of the above-defined problem are effectively solved. In particular, it is demonstrated that the claimed process is effective in removing unreacted diene from EPDM polymer produced by a gas phase fluid bed process.

Obviousness

7. From the above considerations it is evident that the prior art documents, whether considered in isolation or in combination, would not provide an incentive to operate in accordance with the requirements of the claimed process.

7.1 An essential feature of the claimed process is the requirement that the polymer entering the purging zone should have a temperature above that of the steam. This feature is not disclosed in any of the documents. In document E the polymer is suspended in water before the steam is led through, so that that citation by itself cannot render the claimed subject-matter obvious.

7.2 According to document A the temperature is not critical, and it is even considered undesirable to add heat to the resin. In document B the temperature of the resin bears no relationship with the steam temperature. According to document C the resin is kept at a temperature between 105 and 140°C so as to prevent the steam from condensing upon the particles, but any heat added to the system is supplied by heat exchangers immersed in the fluid bed, that is, in the purging zone, and not before it.

Therefore, none of the cited documents hints at the possibility to have the resin, at the moment of entering the purging zone, at a temperature higher than that of the steam.

7.3 In view of the above noted differences it is also obvious that even a combination of document E with any

of documents A, B or C or any combination of the latter documents would not lead to the specific process defined in present Claim 1.

7.4 For the above reasons, the Board comes to the conclusion that the subject-matter of Claim 1 involves an inventive step.

8. As Claim 1 of the main request is allowable, the same goes for dependent Claims 2 to 10, the patentability of which is supported by that of Claim 1.

Order

For these reasons it is decided that:

The appeal is dismissed.

The Registrar: The Chairman:

E. Görgmaier C. Gérardin