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DECISION of 2 December 2004

T 1163/02 - 3.3.9 Case Number:

Application Number: 94118706.4

Publication Number: 0647673

IPC: C08J 9/14

Language of the proceedings: EN

Title of invention:

Expandable polyolefine compositions and preparation process utilizing isobutane blowing agent

Patentee:

THE DOW CHEMICAL COMPANY

Opponent:

NMC SA

Headword:

Relevant legal provisions:

EPC Art. 56

"Inventive step (no) - obviousness by combination of two documents"

Decisions cited:

Catchword:



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Boards of Appeal

Chambres de recours

Case Number: T 1163/02 - 3.3.9

DECISION

of the Technical Board of Appeal 3.3.9 of 2 December 2004

Appellant: NMC SA (Opponent) Rovert, 10

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Decision under appeal: Decision of the Opposition Division of the

European Patent Office orally announced on 11 September 2002 and posted 1 October 2002 rejecting the opposition filed against European patent No. 0647673 pursuant to Article 102(2)

EPC.

Composition of the Board:

Chairman: P. Kitzmantel
Members: W. P. Ehrenreich

M.-B. Tardo-Dino

Summary of Facts and Submissions

- I. Mention of the grant of European patent No. 0 647 673
 in respect of European patent application
 No. 94 118 706.4 in the name of The Dow Chemical
 Company filed on 3 October 1986 as a divisional
 application of application No. 86 113 687.7 claiming
 two US priorities of 24 December 1985 and 24 March 1986,
 was announced on 1 September 1999.
 The patent was granted with five claims, independent
 Claims 1 and 5 reading as follows:
 - "1. A process for preparing a substantially closed cell olefin polymer foam having dimensional stability characterized by the steps of:
 - (a) heat plastifying an olefin polymer resin of homopolymers of ethylene
 - admixing said heat plastified resin with (1) a (b) stability control agent selected from the group consisting, fatty acid amides, and polystyrene and (2) a blowing agent selected from the group consisting of (i) isobutane, (ii) a mixture of from 5%-95% isobutane on a molar basis with from 95%-5% of a physical blowing agent selected from the group consisting of chlorofluorocarbons and fluorocarbons having from 1 to 4 carbon atoms, boiling points between -50°C and 50°C, and a permeation rate through said olefin polymer resin modified with said stability control agent of less than about 1.2 times the permeation rate of air, and (iii) a mixture of at least 70% isobutane with a physical blowing agent selected from the group consisting of hydrocarbons, chlorocarbons and

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chlorofluorocarbons having from 1 to 5 carbon atoms, boiling points between -50°C and 50°C, and a permeation rate through said olefin polymer resin modified with said stability control agent of greater than about 1.2 times the permeation rate of air; and

- (c) activating said blowing agent to expand said admixture to a substantially closed-cell olefin polymer foam."
- "5. Expandable composition useful for preparing a substantially closed cell olefin polymer foam having dimensional stability by activation of the blowing agent comprising
- (a) a heat plastified olefin polymer resin of homopolymers of ethylene
- (b) a stability control agent selected from the group consisting, fatty acid amides, and polystyrene and (c) a blowing agent selected from the group consisting of (i) isobutane, (ii) a mixture of from 5%-95% isobutane on a molar basis with from 95%-5% of a physical blowing agent selected from the group consisting of chlorofluorocarbons and fluorocarbons having from 1 to 4 carbon atoms, boiling points between -50°C and 50°C, and a permeation rate through said olefin polymer resin modified with said stability control agent of less than about 1.2 times the permeation rate of air, and (iii) a mixture of at least 70% isobutane with a physical blowing agent selected from the group consisting of hydrocarbons, chlorocarbons, and chlorofluorocarbons having from 1 to 5 carbon atoms, boiling points between -50°C and 50°C, and

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a permeation rate through said olefin polymer resin modified with said stability control agent of greater than about 1.2 times the permeation rate of air."

Claims 2 to 4 were dependent on Claim 1.

Notice of Opposition requesting revocation of the patent in its entirety on the grounds of Article 100(a),(b) and (c) EPC was filed by NMC SA on 26 May 2000.

With regard to Article 100(a) EPC the opposition was based on the following documents:

D1: EP-A 0 041 234

D2: US-A 4,214,054

D3: US-A 4,368,276

D4: US-A 4,395,510

D5: GB-A 1 170 802

In its response to the Notice of Opposition the Proprietor mentioned the following document:

D6: DE-B 1 282 918

belonging to the same patent family as D5.

III. In its decision orally announced on 11 September 2002 and issued in writing on 1 October 2002 the Opposition Division rejected the opposition.

In that decision the issues of novelty under the grounds of Article 100(a) EPC, insufficient disclosure under Article 100(b) EPC and added subject-matter under Article 100(c) EPC were no longer in dispute.

As to the question of inventive step it was held in the decision that the subject-matter of the claimed invention was non-obvious over the cited prior art, in particular over a combination of D2, representing the closest prior art, and D6.

- IV. On 26 November 2002 the Opponent (Appellant) lodged an appeal against the decision of the Opposition Division. The Statement of Grounds of Appeal was submitted on 3 February 2003.
- V. With its letter of response dated 24 October 2003, the Respondent (Proprietor) filed auxiliary requests 1 to 3 comprising the following amendments:
 - (a) Auxiliary Request 1 In the independent Claims 1 and 5 corresponding to the same claims of the main request, the stability control agent "polystyrene" has been deleted.
 - (b) Auxiliary Request 2 In the independent claims 1 and 3 corresponding to Claims 1 and 5 of auxiliary request 1, the qualification "low density" has been added to the definition "homopolymer of ethylene".

(c) Auxiliary Request 3

In the independent Claims 1 and 2 corresponding to Claims 1 and 3 of auxiliary request 2, the definition of the blowing agent has been restricted to the use of isobutane only.

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- VI. The Appellant's arguments submitted in writing and at the oral proceedings held on 02 December 2004 may be summarised as follows:
 - (a) D1 to D4 disclosed all characteristics of the invention except that <u>iso</u>butane as blowing agent was not mentioned but only "butane". In particular, example 24 of D2 showed a process for preparing a closed-cell foam by heat-plastifying low density polyethylene (LDPE), admixing it with a fatty acid amide as stability control agent and blowing the mixture with butane as blowing agent. The maximum shrinkage and the dimensional stability of the resulting foam were marked as "b" and "a", respectively, which characterise these properties as "good" and "excellent" according to col. 15 of D2.
 - (b) The term "butane" only embraced the two isomers n-butane and isobutane, ie the person skilled in the art had a choice from two alternatives only. However, in the circumstances, the use of isobutane as blowing agent was obvious because D6 indicated that branched hydrocarbon blowing agents, isobutane inclusive, improved the shrink properties of foamed olefin polymers.
 - (c) A comparison of the butane-blown foams according to examples 21, 24 and 27 of D2 with the isobutane-blown foam of example 2 (Table II) of the patent in suit showed a shrink of 10% to 15% according to D2 as compared with a shrink of 7% according to said example 2. The influence of

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isobutane on the shrink properties as compared to n-butane was therefore considered marginal.

- (d) Moreover, the advantageous influence on the shrink properties of closed-cell foams of isobutane, having a permeation rate ratio relative to air < 1, over n-butane, having a permeation rate ratio relative to air > 1, could be easily predicted because a skilled person was aware from general common knowledge that a foam would not shrink when the permeation rate of the blowing agent through the cell walls was less than that of the air replacing it, whereas otherwise a foam would tend to shrink.
- VII. The written and oral arguments of the Respondent may be summarised as follows:
 - (a) The Appellant's conclusions based on the comparison between the shrink properties of the butane-blown foams according to examples 21, 24 and 27 of D2 and the isobutane-blown foam according to example 2 of the patent were not correct. In particular, the test report submitted with the letter dated 24 October 2003 clearly demonstrated that the dimensional stability of the isobutane-blown foams according to the invention was considerably higher and their aging time at elevated temperature was lower than the corresponding properties of n-butane-blown foams.
 - (b) The dichlorodifluoromethane-blown foams according to examples 10 and 22 of D2 showed properties with regard to "surface smoothness" and "maximum

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shrinkage" which were marked with "a", ie

"excellent". The same properties of the butaneblown foam of example 24 were marked with "b" ie
only "good". Therefore, in the light of the better
properties with respect to surface smoothness and
maximum shrink, the teaching of D2 rather than the
use of butane as blowing agent suggested the use
of halogenated hydrocarbons like dichlorodifluoromethane.

- of polyethylene with isobutane. In example 2, isobutane was used for expanding an EVA copolymer. The results shown in table 3 of D6 in context with EVA were not transferable to the homopolymers of ethylene as specified in the patent in suit. Furthermore, the reference to "überwiegend geschlossene Zellstruktur" ("predominantly closed cell structure") at col. 4, ll. 11-13 of D6 was not equivalent to the closed cell foams in the sense of the invention.
- (d) Furthermore, according to D6 a stability control agent was not used. The influence on the permeation rate of isobutane of a fatty acid amide used for this purpose could therefore not be predicted. All the more so as table I of the patent in suit demonstrated a considerable variation of the influence of the fatty acid amide control agent "Kemamide S-180", on the relative permeability of n-butane, isobutane and isopentane through a polyethylene film.

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- (e) D6 did also not mention the use of a low density polyethylene in combination with isobutane as blowing agent as required according to auxiliary requests 2 and 3. The linear polyethylene exemplified in Table 2 of D6 was a high density polyethylene, and the low density high pressure polyethylene used according to example 4 was expanded with 2,2-dimethylpropane, not with isobutane.
- (f) In the light of the above, the skilled person starting from the teaching of D2 in order to solve the problem of low shrinkage, ie high dimensional stability and a low aging/curing time at elevated temperature, was not motivated to combine D2 with D6 in order to arrive at the claimed combination of features which required at least the following four selections:
 - (1) a (low density) ethylene homopolymer foam
 - (2) a closed-cell structure of the foam
 - (3) the use as stability control agent of fatty acid amides (or polystyrene) selected from a number of different stability control agents specified in D2, and altogether missing from the disclosure of D6, and
 - (4) isobutane as blowing agent which was not disclosed in D2 and not used in D6 in combination with a stability control agent.
- VIII. 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

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alternatively on the basis of Claims 1 to 5 of the first auxiliary request, or Claims 1 to 3 of the second auxiliary request, or Claims 1 and 2 of the third auxiliary request, all as filed with the letter dated 24 October 2003.

Reasons for the Decision

- 1. The appeal is admissible.
- Novelty under the Opposition Grounds of Article 100(a), and Opposition Grounds of Articles 100(b) and 100(c)
 EPC

The Opponent's objections as to lack of novelty (Article 54 EPC), insufficiency of disclosure (Article 83 EPC) and added subject-matter (Article 123(2) EPC) raised in the Notice of Opposition have been dropped in the oral proceedings before the Opposition Division.

The Board is satisfied that the subject-matter of the claims as granted as well as that of the claims of the auxiliary requests 1 to 3 is novel over the cited prior art and meets the requirements of Article 123(2) EPC. In the Board's opinion, the claims of the auxiliary requests also do not extend the protection conferred (Article 123(3) EPC); furthermore, the invention is considered to be sufficiently disclosed and meets therefore the requirements of Article 83 EPC.

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- 3. Inventive step
- 3.1 The subject-matter of the patent in suit
- 3.1.1 The patent in suit concerns a process for preparing closed-cell foams via expansion of homopolymers of ethylene (main request and auxiliary request 1), like low density polyethylene LDPE (according to the auxiliary requests 2 and 3), with a hydrocarbon blowing agent. According to Claim 1 of all requests the process is carried out in three steps:

The invention also concerns an expandable composition for preparing the closed-cell foam comprising the components as defined above; (Claim 5 of the main request and of auxiliary request 1; Claim 3 of auxiliary request 2 and Claim 2 of auxiliary request 3).

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- 3.1.2 In the patent specification it is stated in paragraphs [0012] and [0013] that the invention meets the need to provide a process and an expandable olefin polymer composition leading to a foam with a high degree of dimensional stability with minimal shrinkage during aging and curing of the polymer foam when isobutane is used as primary blowing agent.
- 3.1.3 Example 2 of the patent specification shows a comparison of a polyethylene foam blown with isobutane alone and without a stability control agent (cf. Table II, Test 1), with a polyethylene foam blown with isobutane alone but in the presence of a fatty acid amide stability control agent (Kemamide S-180; Table II, Test 2) according to the invention. The results demonstrate a significant improvement in room temperature foam stability and foam stability at 74°C, both expressed in minimum foam volume as percentage of initial volume, when a combination of isobutane blowing agent with fatty acid amide stability control agent is used over isobutane alone.
- 3.2 The closest prior art
- 3.2.1 Documents D1 to D4, lying in the same technical field as the patent in suit are representative of the closest prior art. These documents all pertain to the preparation of olefin polymer foams by expanding the polymer with a volatile blowing agent in the presence of a stability control agent.

In particular, the following essential features are disclosed in the above documents:

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- the expandable olefin polymers preferably include homopolymers of ethylene, like LDPE: D1, Claim 2 in combination with page 4, lines 14 to 18 and page 11, lines 25, 26; D2, Claim 5 in combination with col. 13, lines 17 to 19 and col. 16, lines 29 to 31 in combination with Table 7 in col. 21/22 "Base resin A"; D3, Claim 12 in combination with col. 3, line 5 and col. 6, line 40 to 42; D4, Claim 2 in combination with col. 3, line 5 and col. 6, lines 40 to 42.
- (b) the polymer is <u>heat-plastified</u>: D1, Claim 10 and page 6, lines 24 to 28; D2, col. 12, lines 37 to 49; D3, Claim 18 and col. 4, lines 12 to 16; D4, col. 4, lines 12 to 16.
- (c) the stability control agent may be selected from fatty acid amides: D1, Claim 1 in combination with page 5 lines 1 to 17; D2, formula (II) in Claim 3 in combination with col. 4, line 62 to col. 5, line 35; D3, Claim 18 in combination with col. 3, lines 23 to 37; D4, Claim 1 in combination with col. 3, lines 23 to 37.
- (d) the blowing agent may be selected from <u>butane</u>: D1, page 8, line 22; D2, col. 12, line 27 and Table 7 in col. 21/22 "blowing agent F"; D3, col. 5, line 8; D4, col. 5, line 8.
- (e) the foams have a <u>closed-cell structure</u>: D1, page 9, lines 14 to 18; D2, col. 3, lines 6 to 15; D3, col. 5, lines 35 to 39; D4, col. 5, lines 35 to 39.
- (f) all documents point to the beneficial influence of the stability control agent on the mechanical properties of the resulting foams, in particular improved dimensional stability, ie low shrink, which allows the use of relatively inexpensive

blowing agents: D1, page 3, line 27 to page 4, line 13; D2, col. 3, lines 45 to 48 in combination with col. 11, lines 57 to 66 and col. 12, lines 52 to 63; D3, col. 4, lines 48 to 60; D4, col. 4, lines 48 to 60.

- 3.2.2 Thus, the person skilled in the art is aware from D1 to D4 that thermoplastic polymers, polyethylene belonging to one of the preferred polymers, can be foamed via heat plastification and blowing with inexpensive common blowing agents (e.g. butane or other halogenated or non-halogenated hydrocarbons) to form closed-cell foams with good dimensional stability, i.e. low shrink, if a stability control agent, like a fatty acid amide, is present in the polymer composition.

 In particular, it is known from example 24 of D2 (cf. Table 7 at col. 21/22), that the ethylene homopolymer LDPE can be blown with "butane" in the presence of a fatty acid amide stability control agent to result in a foam having a "good" maximum shrinkage and an "excellent" dimensional stability.
- 3.2.3 The embodiment of the prior art coming closest to the claimed invention is disclosed in example 24 of D2, the only distinguishing feature being the use of "butane" as blowing agent in lieu of isobutane.
- 3.3 Problem and solution
- 3.3.1 With letter dated 24 October 2003 the Respondent submitted a test report demonstrating that an LDPE foam expanded with isobutane in the presence of the fatty acid amide Kemamide S-180 (stearyl stearamide) as stability control agent provides superior dimensional

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stability before and after compression (expressed as minimum volume as a percentage of initial volume at various aging temperatures) over an LDPE foam which has been blown with n-butane in the presence of Kemamide S-180.

- 3.3.2 Considering this experimental evidence, the problem to be solved in the claimed invention can be seen in the development of a method for preparing butane-blown LDPE foams with improved dimensional stability.
- 3.3.3 According to the claimed invention, the solution to this problem is the selection of isobutane as blowing agent.

In view of the aforementioned experimental evidence, the Board is satisfied that by this selection the existing technical problem is effectively solved by the subject-matter of all requests.

3.4 Obviousness

3.4.1 According to the established case law of the Boards of Appeal the question to be answered, in assessing inventive step, is whether the skilled person arrived at the claimed solution of the existing technical problem with a reasonable expectation of success (see for example decision T 149/93, Reasons, point 5.2). For the present case, the question arises whether the prior art comprises information motivating the person skilled in the art to select isobutane as blowing agent from the two existing butane isomers in the expectation of arriving at a foam with superior dimensional properties.

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In the Board's judgement, document D6 provides 3.4.2 appropriate and unambiguous information to do so. D6 pertains to a process for preparing foams having substantially closed cells and being derived from olefin polymers by heat-plastifying the polymer and foaming it with branched hydrocarbons. Isobutane, 2,2dimethylpropane, 2,2-dimethylbutane and 2,3dimethylbutane are expressly mentioned (see the sole claim in combination with col. 4, lines 11 to 13). In the general description it is stated at col. 1, lines 41 to 47 that surprisingly strongly branched hydrocarbons provide a beneficial influence on the shrink properties of foamed olefin polymerisates vis à vis non-branched hydrocarbons. In line 52 of col. 1, polyethylene is mentioned as one possible polyolefin. Example 2 provides a comparison of the foam volume after two days and the end volume after three weeks relative to the initial volume between a foam blown with isobutane and one blown with n-butane; Table 3 which summarises the results demonstrates the beneficial effect of isobutane leading to a shrink of only 12% and 5%, respectively, as compared with 38% and 25%, respectively, achieved with n-butane. Therefore, the skilled person, starting from D2 and being aware of the above disclosure in document D6, would expect that an LDPE foam, blown with butane in the presence of a fatty acid amide stability control agent, could be improved in dimensional stability and shrink if isobutane is selected as blowing agent.

Hence, the Board considers the claimed invention to be obvious in the light of a combination of D2 with D6. This conclusion is valid for the subject-matter of the

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process claims as well as for the product claim of the main request and of all three auxiliary requests because they all embrace LDPE as homopolymer of ethylene, fatty acid amide as stability control agent and isobutane alone as blowing agent.

3.4.3 Consequently, the subject-matter of all claims according to the main request and the auxiliary requests 1 to 3 does not meet the requirements of Article 56 EPC.

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:

G. Röhn P. Kitzmantel