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Case Number: T 0489/00 - 3.3.3

Decision of 28 March 2003
correcting an error in the
D E C I S I O N
of the Technical Board of Appeal 3.3.3
of 24 September 2002

Appellant: DSM N.V.
Het Overloon 1
NL-6411 TE Heerlen (NL)

Representative:

Decision under appeal: Decision of the Examining Division of the
European Patent Office posted 8 February 2000
refusing European patent application
No. 96 935 590.8 pursuant to Article 97(1) EPC.

Composition of the Board:

Chairman: R. Young
Members: A. Däweritz
J. De Preter

In application of Rule 89 EPC, the Decision given on 24 September is hereby corrected as follows:

Page 7, Point V, and

Page 15, Order, Point 2:

The reference to page "4A" is deleted from the list of pages of the Description.

The Registrar:

The Chairman:


E. Görgmaier


R. Young



Internal distribution code:

- (A) Publication in OJ
(B) To Chairmen and Members
(C) To Chairmen
(D) No distribution

D E C I S I O N
of 24 September 2002

Case Number: T 0489/00 - 3.3.3

Application Number: 96935590.8

Publication Number: 0861289

IPC: C08K 3/26

Language of the proceedings: EN

Title of invention:

Process for the preparation of a thermoplastic elastomer

Applicant:

DSM N.V.

Opponent:

-

Headword:

-

Relevant legal provisions:

EPC Art. 54, 56

Keyword:

"Novelty (yes) - prior disclosure"

"Inventive step (yes) - problem and solution"

Decisions cited:

-

Catchword:

-



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Summary of Facts and Submissions

I. European patent application No. 96 935 590.8, based on International application No. PCT/NL96/00433, filed on 6 November 1996, claiming the priority of 17 November 1995 of an earlier European patent application No. 95 203 152.4 and published under No. WO/97/19130 on 29 May 1997 including 15 claims, was refused by a decision of the Examining Division, issued in writing on 8 February 2000, for lack of novelty on the basis of

D1: US-A-4 311 628

and for lack of inventive step on the basis of the combination of D1 and

D2: EP-A-0 097 330.

II. The decision was based on a set of 16 claims submitted with a letter dated 26 January 1999. The claims of this set of claims relevant to these appeal proceedings read as follows:

"1. Process for the preparation of a thermoplastic elastomer, comprising preparing a blend of a rubber and a thermoplastic resin, in which the rubber is at least partially vulcanized by using a phenolic curative, characterised in that an effective amount of a Lewis base is added after the desired degree of vulcanization of the rubber, to the extent that not more than 50% of the rubber is extractable in boiling xylene, is obtained."

- "2. Process according to claim 1, characterized in that the Lewis base used is a compound selected from the group of hydrotalcites."
- "11. Process according to anyone of claims 1-10, characterized in that after the desired degree of vulcanization is obtained also an effective amount of a HALS-compound is added."
- "16. Article, obtainable by a process according to claims 11 to 15."

Claims 3 to 10 and 12 to 15 concerned further specific elaborations of the process according to Claim 1 and were appendant to at least one preceding claim.

This set of claims differed from the set of claims as originally filed by the insertion ", to the extent that not more than 50% of the rubber is extractable in boiling xylene," in Claim 1, and by the presence of new Claim 16.

Based on the argument that zinc oxide was a Lewis base, it was decided that the subject-matter of Claims 1 and 3 to 10 was anticipated by D1, because in that document a polypropylene/EPDM-rubber blend was vulcanised with the help of a phenolic curative, and a Lewis base, eg ZnO, was added at the end of the reaction (cf. examples, tables and column 15, lines 42 to 65) to provide compositions, which are referred to as thermoplastic elastomeric compositions ("TPE") or thermoplastic vulcanizates ("TPV") in D1 and the application in suit, respectively.

The other claims were deemed novel, because hydrotalcite was used as a Lewis base and because of the presence of a HALS (hindered amine light stabiliser), respectively. However, the claimed subject-matter was held not to involve an inventive step, because it was not apparent from the application documents that any Lewis base other than hydrotalcite would contribute to the solution to the problems addressed in the application (ie to reduce or prevent surface roughness of the products and surface cracking occurring within a short period of time, and to enhance the efficiency of HALS).

It was concluded that Claim 1 lacked an inventive step in view of usual processes to prepare vulcanised TPEs and, furthermore, the fact that hydrotalcite was a usual additive for TPEs, and specifically known to act as an "acid acceptor", as disclosed in D2. Since hydrotalcites as well as stabilisers such as HALS were generally known additives to TPEs, these features could not support an inventive step.

III. On 5 April 2000, a Notice of Appeal, dated 4 April 2000, against the above decision was lodged by the Appellant (Applicant). The prescribed fee was paid on the same date. At the same time, the Statement of Grounds of Appeal, a new set of Claims 1 to 14 and amended pages of the description (pages 1, 1A to 7, 7A) were submitted. The claims read as follows:

"1. Process for the preparation of a thermoplastic elastomer, comprising preparing a blend of a rubber and a thermoplastic resin, in which the rubber is at least partially vulcanized by using a phenolic curative, characterised in that an amount

of 0.1 - 10 parts of a hydrotalcite per 100 parts of rubber and thermoplastic resin and an amount not exceeding 7.5 wt.% of a HALS-compound per 100 wt.% of the thermoplastic elastomer is added after the desired degree of vulcanization of the rubber, to the extent that not more than 50% of the rubber is extractable in boiling xylene, is obtained.

2. Process according to claim 1, characterized in that the hydrotalcite is used in an amount of 0.25-5 parts, per 100 parts of rubber + thermoplastic resin.
3. Process according to claim 1, characterised in that the rubber used is selected from EADM rubber and butyl rubber.
4. Process according to claim 3, characterised in that the EADM rubber used is a terpolymer of ethylene, propylene and ethylidene norbornene.
5. Process according to any one of claims 1-4, characterised in that the thermoplastic resin used is a thermoplastic polyolefin resin.
6. Process according to claim 5, characterised in that the thermoplastic resin used is selected from polyethylene and polypropylene.
7. Process according to any one of claims 1-6, characterised in that the rubber in the thermoplastic elastomer is vulcanized to the extent that not more than 15% of the vulcanizable rubber is extractable in boiling xylene.

8. Process according to claim 7, characterised in that the rubber is vulcanized to the extent, that not more than 5% of the vulcanizable rubber is extractable in boiling xylene.
9. Process according to any one of claims 1-8, characterised in that in the preparation of the elastomer 30-400 parts of rubber per 100 parts of thermoplastic resin are used.
10. Process according to claim 1, characterised in that the added HALS-compound has a molecular weight between 250 and 5000.
11. Process according to claim 10, characterised in that the added HALS-compound has a molecular weight between 300 and 1000.
12. Process according to claims 11, characterised in that the added HALS-compound is used in an amount not exceeding 5 wt.%.
13. Process according to claim 12, characterised in that the added HALS-compound is used in an amount between 0.1 and 2.5 wt.%.
14. Article, obtainable by a process according to claims 10 - 13."

The arguments provided by the Appellant can be summarised as follows:

Document D1 disclosed a process for the preparation of a thermoplastic elastomeric (elastoplastic) composition, based on polyolefin resin and fully cured

EPDM rubber. When a HALS compound was added to such a product, its efficiency was low, as evidenced by the occurrence of surface cracking. This was demonstrated in Comparative Experiment A of the application.

When, however, a combination of a HALS compound and a hydrotalcite was added after the desired degree of vulcanisation had been achieved, the UV stability was surprisingly improved to a marked extent and surface cracking occurred only after a much longer exposure to a weathering test.

Document D2 disclosed the curing under heat of a halogen containing rubber in the presence of a hydrotalcite compound, resulting in a cured product having an improved water resistance. However, D2 was silent about UV-resistance and did not give any hint that hydrotalcite would improve the efficiency of a HALS-compound in a TPE.

IV. Following a conversation by telephone between the Representative and the Rapporteur on 13 September 2002, Claim 1 of the said set of claims was further amended (submission by fax, received on 13 September 2002) to remove the inconsistency between the arguments provided in the Statement of Grounds of Appeal and the evidence on file, on the one hand, and the wording of the request, on the other. Claim 1 as amended reads:

"1. Process for the preparation of a thermoplastic elastomer, comprising preparing a blend of a rubber and a thermoplastic resin, in which the rubber is at least partially vulcanized by using a phenolic curative, characterised in that an amount of 0.1 - 10 parts of a hydrotalcite per 100 parts of rubber and thermoplastic resin and an amount of 0.1 - 7.5 wt.% of a HALS-compound per 100 wt.% of the thermoplastic elastomer is added after the

desired degree of vulcanization of the rubber, to the extent that not more than 50% of the rubber is extractable in boiling xylene, is obtained."

Claims 2 to 14 remained unchanged compared with the version filed with the Statement of Grounds of Appeal.

Additionally, the Appellant further adapted the description to the wording of Claim 1 on new page 1A, also filed on 13 September 2002.

V. The Appellant implicitly requested that the decision under appeal be set aside and that a patent be granted on the basis of the following documents:

- Claim 1, submitted by fax on 13 September 2002
- Claims 2 to 14, dated 4 April 2000
- Description:
Pages 1, 2, 2A, 3, 3A, 4, 4A, 5, 5A, 6, 6A, 7 and 7A, dated 4 April 2000
Page 1A, submitted by fax on 13 September 2002
Pages 8 to 11, as originally filed.

Reasons for the Decision

1. The appeal is admissible.

2. *Article 123(2) EPC*

Claim 1 as amended is based on Claims 1, 2 and 11 and on the passages on page 3, lines 32 to 35, page 4, lines 2 to 5, 18 and 19, and page 6, lines 18 to 23 of the application as originally filed. Claims 2 to 13 are

based on Claims 3 to 10 and 12 to 15 as originally filed. Claim 14 is supported by page 7, lines 35 to 37 as originally filed.

The amendments on pages 1 and 2, 2A to 7, 7A, dated 4 April 2000, and on page 1A, received by fax on 13 September 2002, are also based on these passages of the original application documents.

Consequently, the requirements of Article 123(2) EPC are met.

3. *Novelty (Articles 54(1) and (2) EPC)*

3.1 The text of the patent application in suit was drafted on the basis of Document D1, to which reference was repeatedly made for the purpose of explaining further details of the thermoplastic elastomers to be prepared in the claimed process (see page 1, line 12: TPV; page 3, lines 21 and 29: the curing system and the determination of the degree of vulcanisation; and page 7, line 5: method of blending).

3.2 Although in column 7, lines 9 to 61 of D1, reference is made to a number of possible modifications of TPE (TPV) compositions prior to or after vulcanisation, eg by incorporating additives such as particulate fillers, stabilisers, antidegradants, flame retardants, processing aids, adhesives, tackifiers, plasticisers, wax, discontinuous fibers, pigments, coupling agents and extender oils, and, *inter alia*, zinc oxide, the latter is listed as a filler, which compound is referred to elsewhere as hydrogen halide scavenger (column 6, lines 44 to 47) or cure activator

(Claims 10, 12, 34, 39, 50 and 52; column 15, lines 42 to 44; column 17, line 67, to column 18, line 1), and the document mentions neither hydrotalcite nor any HALS compounds.

3.3 Document D2 discloses neither TPE compositions nor a process for their preparation, but relates to a method for curing a curable halogen-containing rubber composition in the presence of a hydrotalcite as an acid acceptor. Moreover, there is no reference to a HALS-compound.

3.4 Consequently, the subject-matter of Claim 1 is novel.

4. *Problem and solution*

4.1 The patent application in suit concerns a process for the preparation of a TPE and articles obtainable therefrom.

4.2 Such a process is known from D1 which the Board, like the Appellant, regards as representing the closest state of the art.

The known process comprises (1) masticating 15 to 75 parts by weight of EPDM rubber and 85 to 25 parts by weight of thermoplastic crystalline polyolefin resin such as polypropylene (per 100 parts by weight of these two components), phenolic curing resin, in an amount sufficient to cure the rubber at a temperature sufficient to soften or melt the polyolefin resin, until a homogeneous mixture is obtained in which the rubber is in the form of small dispersed particles essentially of a size of 50 μm (number average) or below, (2) adding a cure activator and (3) continuing masticating the mixture at curing temperature until the rubber is cured to the extent that no more than five percent by weight of curable rubber is extractable in

boiling xylene (Claim 48). The term "activator" means any material which materially increases the cross-linking efficiency of the phenolic curing resin and includes metal oxides and halogen donors either used alone or conjointly. A combination of these groups of compounds is recommended, when the rubber does not readily cure with phenolic resins. Zinc oxide is a preferred cure activator. Examples for halogen donors are stannous chloride and ferric chloride, or halogen donating polymers (see section 3.2, above; D1: column 6, lines 23 to 65, in particular, lines 61 to 65, Tables V, XI, XII).

According to preferred embodiments, different orders of addition of the components are applicable. Thus, a masterbatch containing *inter alia* the rubber, filler, and zinc oxide is mixed with the polypropylene at increased temperature until the polypropylene is melted and a uniform blend is obtained (column 8, lines 61 to 68), or superior compositions, especially in the absence of a filler, are obtained when the zinc oxide is added last (column 15, lines 42 to 61).

Emphasis is put on the improvements of the compression set and the oil resistance by means of the phenolic curative system in comparison to eg sulphur-based vulcanising systems. With respect to Table III, a general reference is additionally made to smoother surfaces obtained upon extrusion or injection moulding with rubbers crosslinked by phenolic in comparison to sulphur curatives (column 10, lines 20 to 37; column 21, lines 27 to 30).

In summary, reference has been made in D1 neither to the UV-stabilisation of TPE-compositions and their weatherability, expressed in surface crack resistance, tensile strength and elongation at break, nor to further improvements of the surface smoothness. D1

contains rather a general reference to different conceivable additives which can be used in a process to prepare TPV (column 7, lines 9 to 61).

4.3 In line with the introductory statements in the patent application (page 1) and the arguments provided in the Statement of Grounds of Appeal, the technical problem underlying the patent application in suit may thus be seen as the definition of a process to prepare TPE-compositions showing improved properties as referred to in the previous paragraph (surface crack resistance, tensile strength, elongation at break and surface smoothness).

4.4 According to the patent application in suit, this problem is solved by incorporating specific amounts of hydrotalcite and a HALS-compound in combination with each other into a blend of a rubber and a thermoplastic resin at a specific moment of its preparation, ie after the desired degree of vulcanisation of the rubber by means of a phenolic curative has been achieved.

According to the examples and comparative experiments in the application, the period of time until the first surface cracks are observed can thus be significantly lengthened (Comparative experiment A: 1000 hours; Example I: more than 4000 hours). Moreover, the retentions in tensile strength (σ_b) and elongation at break (ϵ_b) are also significantly enhanced: Examples II (σ_b : 110.9%, ϵ_b : 102.1%) and III (σ_b : 102.8%, ϵ_b : 94.2%) in comparison to Comparative Experiments B (σ_b : 91.3%, ϵ_b : 84.2%) and C (σ_b : 90.7%, ϵ_b : 82.9%), respectively. Additionally, it has been shown that the smoothness of extrudates prepared from the compositions obtainable in accordance with Claim 1 can be further improved (Comparative Experiment D and Example IV).

4.5 Consequently, all the aspects of the above defined technical problem are effectively solved by the process as defined in Claim 1 of the application in suit.

5. *Obviousness*

It remains to be decided whether this solution was obvious to a person skilled in the art having regard to the state of the art relied upon in the decision under appeal.

5.1 It is evident from the above considerations that D1, by itself, does not provide any information with respect to the addition of hydrotalcite and HALS-compounds, let alone the influence of a combination of these compounds on the above properties when added after the desired degree of vulcanisation has been achieved.

5.2 Document D2 aims at improved properties such as chemical resistance (weatherability), insulation properties and, in particular, reduced water sensitivity of a product which is the result of a method for curing a curable rubber composition based on a halogen-containing rubber and a vulcanising agent or a vulcanisation accelerator, other than thiazole-type vulcanisation accelerators, under heat in the presence of an acid acceptor, wherein the acid acceptor is a hydrotalcite of a particular chemical structure or its calcination product.

The curing of halogen-containing rubbers requires the use of an acid acceptor in addition to a vulcanisation accelerator. The hitherto used acid acceptors, ie metal oxides or hydroxides, were converted to hygroscopic halides or basic halides of these metals in the curing

reaction and caused poor mechanical strength and low heat resistance due to low speed of vulcanisation (Pb containing acid acceptors) (Claim 1; pages 1 and 2).

The vulcanising agent and the vulcanisation accelerator may be of any known type, the latter with the exception of thiazole-type compounds. Numerous examples of these components are listed on pages 7 and 8. Further additives including fillers, reinforcing agents, plasticisers, processing aids, antioxidants, pigments and fire retardants, which are used in amounts generally known in the art, are referred to (page 8, line 31 to page 10, line 20). The presence of UV-stabilisers has, however, not been contemplated in D2.

The compositions are moulded by means of eg calendering, extrusion or intermixing, and then cured by methods such as press curing, curing in a can, curing during, injection moulding, LCM curing, oven curing and continuous curing (page 10, lines 21 to 31). Whilst the general description is completely silent in this respect, it is evident from the examples of D2 that all ingredients are mixed with each other prior to curing.

The document does not relate to a TPE composition, let alone a composition of this type stabilised by the incorporation of a HALS-compound, nor to an improvement of the efficiency of the HALS-compound in a composition of this type in order to improve the weatherability and the relevant physical properties, ie surface crack resistance, tensile strength, elongation at break and surface smoothness, mentioned above.

Hence, it relates to a completely different type of composition, exhibiting a different spectrum of properties. Consequently, D2 cannot provide an incentive to solve the relevant technical problem underlying the application in suit so as to arrive at something within the ambit of the claims under consideration.

5. It follows that the process of Claim 1 would not be obvious to a person skilled in the art with respect to the state of the art cited in the decision under appeal, and, therefore, involves an inventive step (Article 56 EPC).

6. Claims 2 to 13, which relate to particular elaborations of the process of Claim 1, are supported by the patentability of this independent claim and thus also allowable.

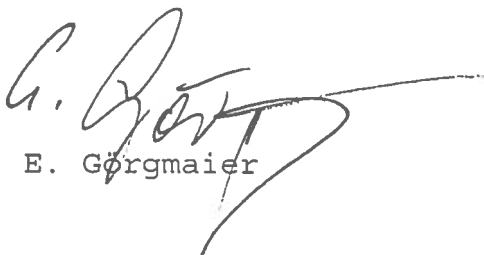
The same is true for the article according to Claim 14.

Order

For these reasons it is decided that:

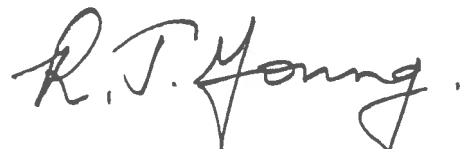
1. The decision under appeal is set aside.
2. The case is remitted to the Examining Division with the order to grant a patent on the basis of the following documents:
 - Claim 1, submitted by fax on 13 September 2002
 - Claims 2 to 14, dated 4 April 2000
 - Description:
Pages 1, 2, 2A, 3, 3A, 4, 4A, 5, 5A, 6, 6A, 7 and 7A, dated 4 April 2000,
Page 1A, submitted by fax on 13 September 2002 and
Pages 8 to 11, as originally filed

The Registrar:



E. Gorgmaier

The Chairman:



R. Young

The following information was obtained from the records of the
 Department of the Interior, Bureau of Land Management, on
 the subject of the above captioned matter.
 The land in question is situated in the County of
 [County Name], State of [State Name].
 The land is owned by [Owner Name], who is the
 [Relationship] of [Relationship Name].
 The land is being offered for sale to the public
 by the Department of the Interior, Bureau of Land
 Management, and the sale will be held at the
 [Location] on the [Date].
 The land is being offered for sale at a price of
 [Price] per acre, and the total amount of the
 sale is [Total Amount].
 The land is being offered for sale in accordance
 with the provisions of the [Act Name], and the
 sale is subject to the terms and conditions set
 forth in the [Document Name].
 The land is being offered for sale in accordance
 with the provisions of the [Act Name], and the
 sale is subject to the terms and conditions set
 forth in the [Document Name].

