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Datasheet for the decision of 20 April 2012

Case Number: T 1565/10 - 3.3.09

Application Number: 00963852.9

Publication Number: 1262510

IPC: C08J 3/12, C08J 3/24, C08J 3/26, C08J 3/28

Language of the proceedings: EN

Title of invention:
Full vulcanized powdered rubber with controllable particle diameter, preparing method and uses thereof

Patentee:
CHINA PETRO-CHEMICAL CORPORATION
Beijing Research Institute of Chemical Industry, SINOPEC

Opponent:
LANXESS Deutschland GmbH

Headword:
-

Relevant legal provisions:
EPC Art. 56

Keyword:
"Inventive step (yes) - new experimental results"

Decisions cited:
-

Catchword:
-

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Case Number: T 1565/10 - 3.3.09

D E C I S I O N
of the Technical Board of Appeal 3.3.09
of 20 April 2011

Appellant I: (Patent Proprietor 1)
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Decision under appeal:
Decision of the Opposition Division of the European Patent Office posted 11 May 2010 revoking European patent No. 1262510 pursuant to Article 101(2) EPC.

Composition of the Board:

Chairman: W. Sieber
Members: J. Jardón Álvarez
R. Menapace

Summary of Facts and Submissions

I. The grant of European patent No. 1 262 510 in respect of European patent application No. 00963852.9, in the name of CHINA PETRO-CHEMICAL CORPORATION and Beijing Research Institute of Chemical Industry, SINOPEC, which had been filed on 18 September 2000 as international application PCT/CN2000/000281, was announced on 7 February 2007 (Bulletin 2007/06). The patent was granted with 25 claims, claims 1 and 23 reading as follows:

"1. A fully vulcanized powdery rubber obtained by vulcanizing with irradiation having a gel content of 60% by weight or more and an average particle size of from 20 to 2,000 nm, with each of the particles present in the powdery rubber being homogeneous, said fully vulcanized powdery rubber not including powdery silicone rubber, wherein said rubber is selected from the group consisting of natural rubber, styrene-butadiene rubber, carboxylic styrene-butadiene rubber, nitrile rubber, carboxylic nitrile rubber, chloroprene rubber, polybutadiene, acrylic rubber, butadiene-styrene-vinylpiridine rubber, isoprene rubber, butyl rubber, polysulfide rubber, acrylate-butadiene rubber, urethane rubber, and fluorine rubber."

"23. A process for preparing a vulcanized rubber powder having a gel content of at least 60% and an average particle size of from 20-2000nm, said process consisting essentially of the following steps in the following sequence:

(a) providing a rubber latex comprising rubber in the form of particles having an average particle size in a range of from 20 to 2000 nm,
(b) optionally adding a cross-linking agent to said rubber latex to form a rubber latex composition;
(c) irradiating the rubber latex composition to cause cross-linking of the rubber with formation of a particulate rubber having a gel content of at least 60% by weight; and
(d) drying the irradiated rubber latex composition and obtaining the vulcanized rubber powder,

wherein said rubber latex composition comprises a rubber latex selected from the group consisting of: natural rubber, styrene-butadiene rubber, carboxylic styrene-butadiene rubber, nitrile rubber, carboxylic nitrile rubber, chloroprene rubber, polybutadiene, acrylic rubber, butadiene-styrene-vinylpiridine rubber, isoprene rubber, butyl rubber, polysulfide rubber, acrylate-butadiene rubber, urethane rubber, and fluorine rubber."

Claims 2 to 22, 24 and 25 were directly or indirectly dependent on claims 1 and 23 respectively.

II. A notice of opposition was filed by LANXESS Deutschland GmbH (opponent) on 7 November 2007 requesting revocation of the patent in its entirety on the grounds pursuant to Article 100(a) EPC (lack of novelty and lack of inventive step).

The documents cited during the opposition proceedings included the following:

D1: EP 0 215 959 A1;

D2: US 5 082 732;

D3: EP 0 575 851 A1;

D4: EP 0 854 170 A1;

D5: EP 1 048 692 A2; and

D6: EP 1 063 259 A1.

III. By its decision announced orally on 25 March 2010 and issued in writing on 11 May 2010, the opposition division revoked the patent.

The opposition division found that none of documents D1 to D6 anticipated the subject-matter of claim 1 of the patent. Concerning inventive step, the opposition division considered D1 to represent the closest prior art document. The patent in suit did not contain any indication or evidence that the process of irradiation had any influence on the uniformity and/or size of the rubber particles. In the absence of such improvement, the opposition division defined the problem to be solved over D1 as being to provide an alternative fully vulcanized powdery rubber. In its opinion, this problem was already solved by D1 alone or by the combination of D1 with any of D2 to D6.

IV. On 7 July 2010 the patent proprietors (appellants) lodged an appeal against the decision of the opposition division and paid the prescribed fee on the same day. In the statement setting out the grounds of appeal,

filed on 9 September 2010, the appellants requested that the decision under appeal be set aside and that the patent be maintained as granted (main request); on an auxiliary basis, that the patent be maintained in amended form in accordance with the claims according to the auxiliary request also submitted with the grounds of appeal.

The appellants also filed an experimental report.

V. By letter dated 19 January 2011 the opponent withdrew the opposition against the patent.

VI. The arguments presented by the appellants, insofar as they are relevant for this decision, may be summarised as follows:

– The patent in suit was directed to a fully vulcanized powdery rubber obtained by vulcanizing selected rubber latexes by irradiation, wherein each of the particles present in the powdery rubber was homogeneous. The fully vulcanized rubbers consisted of free-flowing particles that could be easily dispersed into plastics. The addition of the particles to plastics resulted in a significant improvement in their properties, such as the Izod impact strength, which could be improved by a factor of more than 10, as could be inferred from table 2 of the patent.

– This fully vulcanized rubber could only be obtained by an irradiation process. The report filed with the grounds of appeal showed that a rubber latex vulcanized using peroxide exhibited a

poor dispersibility. There was no hint of this advantageous property of the claimed vulcanized rubber in the documents cited in the decision under appeal. In particular, care had to be taken to prevent enlargement of rubber particles in the process of D1. This was made by using specific copolymers in particular amounts.

VII. The appellants requested that the decision of the opposition division revoking European patent No. 1 262 510 be set aside and that the patent be maintained with the claims as granted (main request), or with the amended claims filed as an auxiliary request with the statement of grounds of appeal.

Reasons for the Decision

1. The appeal is admissible.

MAIN REQUEST (claims as granted)

2. *Novelty*

In its decision the opposition division considered that the subject-matter of the granted claims was novel over the cited prior art. The board sees no reason to depart from this view.

3. *Inventive step*

3.1 The patent in suit relates to a fully vulcanized powdery rubber obtained by irradiating a rubber latex and having a uniform and controllable particle size

which is substantially the same as the particle size of the rubber particles in the latex. The rubber can be easily dispersed to produce toughened plastics (paragraph [0009]).

In particular, the subject-matter of claim 1 is directed to a fully vulcanized powdery rubber having the following features:

a) the powdery rubber is obtained by vulcanizing with irradiation a rubber selected from the group consisting of natural rubber, styrene-butadiene rubber, carboxylic styrene-butadiene rubber, nitrile rubber, carboxylic nitrile rubber, chloroprene rubber, polybutadiene, acrylic rubber, butadiene-styrene-vinylpiridine rubber, isoprene rubber, butyl rubber, polysulfide rubber, acrylate-butadiene rubber, urethane rubber, and fluorine rubber;
b) the powdery rubber has a gel content of 60% by weight or more;
c) has an average particle size of from 20 to 2 000 nm; and
d) each of the particles present in the powdery rubber is homogeneous.

3.2 The opposition division and the parties regarded document D1 as closest prior art.

Document D1 relates to a fine particulate cross-linked amorphous copolymer having constituent units derived from at least ethylene and α-olefin, having an average particle diameter of 0.2 to 50 µm and containing hot toluene insolubles of 15% by weight or more (see claim 1). The latex cross-linking is carried out by

means known in the art, such as ionizing radiation cross-linking and organic peroxide cross-linking (page 13, lines 8-12; see also claim 6). The vulcanized rubber is used as a modifier of plastics (paragraph bridging pages 3 and 4).

Thus, D1 is in the same technical field as the invention and the board is also of the opinion that D1 is indeed the closest prior art.

3.3 According to the appellants, the technical problem underlying the present invention in the light of the closest prior art (D1) is the provision of a fully vulcanized powdery rubber which provides a significant toughening effect on a plastic. In particular, the fully vulcanized powdery rubber should have particles of controlled size which do not agglomerate, and can be readily dispersed in a plastic.

3.4 As a solution to this problem the patent proposes the fully vulcanized powdery rubber of claim 1, which is derived from rubbers not disclosed in D1 and obtained by vulcanization by irradiation.

3.5 The board is satisfied that this problem has been credibly solved by the claimed rubber. Examples 1 to 11 in the patent in suit show that fully vulcanized powder rubbers having the desired properties can be obtained from different rubber types by irradiation. Examples 12 to 14 further show that the powdery rubber can be easily dispersed into plastics to prepare toughened plastics.

3.5.1 The experimental report filed by the appellants during the appeal proceedings further shows that the method of preparation of the fully vulcanized rubber indeed has an influence on the properties of the rubber. Thus, a styrene/butadiene rubber vulcanized using peroxide in accordance with the prior art (cf. example 1 of D4) exhibits poor dispersibility when blended with polypropylene (see Figure 1 of the report). By contrast, the vulcanized powdery rubber of the invention obtained by irradiation can be dispersed as discrete primary particles in a matrix. This experimental evidence convincingly shows that the difference in the dispersibility of the vulcanized rubber is due to the different method of preparation. Rubber latex fully vulcanized by irradiation can be dried to give a powdery rubber that can be easily dispersed as discrete primary particles in a plastic matrix when it is blended with the plastic matrix, while rubber latex vulcanized using peroxide cannot.

3.5.2 Finally, it is acknowledged that the claimed rubbers show improved properties over the rubbers disclosed in D1. As indicated by the appellants on page 6 of their statement of grounds of appeal, the Izod impact strength measured in accordance with ASTM D256 for the plastic *per se* and for the plastic plus the rubber of the invention increases by a factor of almost 15 (cf. Table 2 of the patent). However, in D1 the increase in the Izod impact strength is considerably lower; it increases by a factor of at most 5 (cf. page 30, line 10 and Table 1 of D1). These results show the advantageous effect of using the claimed fully vulcanized powdery rubbers, when compared with those of

D1, in their ability to absorb energy during plastic deformation.

3.6 It remains to be decided whether, in view of the available prior-art documents, it would have been obvious for the skilled person to solve the above-defined technical problem by the means claimed, namely by the claimed fully vulcanized powdery rubber obtained by irradiating the specific rubbers.

3.7 There is no hint in the direction of this solution in the prior art cited in the appealed decision, namely D1 to D6.

3.7.1 Document D1, on which the opposition division mainly relied, does not give any hint of the claimed rubbers. As indicated above, D1 uses a different rubber as the starting material. In D1 vulcanization is carried out indistinctly by ionizing radiation or by organic peroxide cross-linking (page 13, lines 8-12). The control of the fine particles in D1 is done by the use of either an amorphous copolymer within a particular viscosity range or by the use of crystalline low molecular weight copolymers and modified copolymers (see page 9, line 10 to page 10, line 5 and page 12, line 34 to page 13, line 3).

The inventive step objection of the opposition division was in fact based on the lack of comparative evidence in the patent of the influence of the method of preparation on the particle uniformity and size of the vulcanized rubber. This objection was overcome by the appellants with the experimental report filed during

the appeal proceedings, as discussed under point 3.5.1 above.

3.7.2 Also none of D2 - D4 gives a hint to the claimed solution:

Document D2 relates to rubber particles useful in reinforcing resin compositions (abstract). The disclosed particles exhibit a micro-phase separating structure having hard segments and soft segments and are therefore not related to the homogenous particles now claimed.

Document D3 discloses mixtures of polybutadiene gel with other rubbers containing carbon-carbon double bonds and their use in the preparation of vulcanized rubber products having a good combination of low hysteresis loss and good abrasion resistance (claims and abstract), and D4 relates to rubber mixtures containing at least one styrene/butadiene rubber gel and at least one rubber with carbon-carbon double bonds, plus other fillers and rubber additives for the production of vulcanised rubber products and mouldings, especially tyre treads (claims). Neither D3 nor D4 uses irradiation to cross-link the rubber.

3.7.3 Documents D5 and D6 were published, respectively, on 2 November 2000 and 27 December 2000, that is to say after the filing date of the patent in suit. These documents are therefore not to be considered as state of the art within the meaning of Article 54(2) EPC.

3.8 Hence the board considers that the finding that the vulcanized rubbers having the features of claim 1 and

prepared by irradiation results in a significant improvement in the toughening of plastic materials is not obvious to the skilled person confronted with the task of finding a solution to the existing technical problem.

3.9 Therefore, the subject-matter of claim 1 and the subject-matter of claim 23, which is directed to a process for the preparation of the vulcanized rubber, involves an inventive step.

Claims 2 to 22, 24 and 25 are directly or indirectly dependent on claims 1 or 23 and, by the same token, also involve an inventive step.

4. As the main request is allowed, there is no need for the board to deal with the auxiliary request.

Order

For these reasons it is decided that:

1. The decision under appeal is set aside.

2. The patent is maintained as granted.

The Registrar: The Chairman:

G. Röhn W. Sieber