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D E C I S I O N
of 26 January 1999

Case Number: T 0345/96 - 3.3.3

Application Number: 90120556.7

Publication Number: 0426024

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Title of invention:
Process for incorporating organic fibrous fillers in elastomers

Applicant:
E.I. du Pont de Nemours and Company

Opponent:
-

Headword:
-

Relevant legal provisions:
EPC Art. 56

Keyword:
"Inventive step (yes)"

Decisions cited:
T 0246/91, T 0495/91

Catchword:
-

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Case Number: T 0345/96 - 3.3.3

D E C I S I O N
of the Technical Board of Appeal 3.3.3
of 26 January 1999

Appellant: E.I. du Pont de Nemours and Company
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Decision under appeal: **Decision of the Examining Division of the European Patent Office dated 7 November 1995 refusing European patent application No. 90 120 556.7 pursuant to Article 97(1) EPC.**

Composition of the Board:

Chairman: C. Gérardin
Members: R. Young
J. A. Stephens-Ofner

Summary of Facts and Submissions

I. European patent application No. 90 120 556.7, entitled "Process for incorporating organic fibrous fillers in elastomers", with 12 claims, filed on 26 October 1990, and published under No. 0 426 024, was refused by a decision of the Examining Division dated 7 November 1995, for lack of inventive step. The decision was based on a set of Claims 1 to 12, filed on 31 May 1995, with a letter of 29 May 1995. Claim 1 of this set reads as follows:

"A process for incorporating fibrous filler into an elastomer which comprises:
(a) feeding to a screw extruder an elastomer latex and an aqueous slurry containing 1-30 parts by weight organic fibrous filler per 100 parts elastomer having a length less than 25 mm, the slurry and latex being fed to the extruder either premixed or as separate streams and subsequently mixed in the extruder,
(b) adding a coagulant for the elastomer latex to the mixture of elastomer latex and aqueous fiber slurry to coagulate the elastomer latex in the screw extruder and form a coagulated elastomer containing the organic fibrous filler,
(c) feeding the coagulated elastomer containing organic fibrous filler into a dewatering zone,
(d) feeding the coagulated fiber-filled elastomer through a flow restriction which applies back pressure sufficient that water present in the coagulated elastomer is forced out of a vent provided in the extruder upstream from the flow restriction, and

(e) discharging and recovering the organic fiber-filled elastomer."

Claims 2 to 12 are dependent claims directed to elaborations of the process according to Claim 1.

II. According to the decision, in which the following three documents were cited:

D1: GB-A-2 138 430;
D2: US-A-4 136 251; and
D3: US-A-4 263 184

the closest prior art was D3, which related to a process for dispersing fibres into an elastomer. Whilst it was desirable to increase the concentration of fibrous material as much as possible, nevertheless the concentrations of fibrous material and elastomer latex were not critical. A homogeneous fibre dispersion could be made by co-precipitating a mixture of the fibrous material and elastomer latex with a solution of a coagulant, and drying the pre-dispersed fibre composition by suitable means. The addition of the fibrous filler in the form of an aqueous slurry and the use of an extruder for the co-precipitation and drying, by which the claimed subject-matter differed from this state of the art, had not, however, been shown to give rise to a technical effect, so the technical problem arising was simply to provide a further process for incorporating fibrous filler into an elastomer. Since, however, the addition of fibres in the form of a slurry was known from D1, and the use of an extruder from D2, it also being well-known that mixtures of polymer and fibrous filler could be extruded, the subject-matter of

Claim 1 was an obvious combination of the process described in D3, with the teachings of D1 and D2.

III. On 19 December 1995, a Notice of Appeal was filed against the above decision, the prescribed fee being paid on the same day.

In the Statement of Grounds of Appeal, filed on 5 March 1996, the Appellant argued substantially as follows:

(a) No worker of ordinary skill, starting from the disclosure of D3, would use such a low concentration of fibres as required in the process claimed, since the more specific disclosure of D3, including the examples, emphasised the desirability of maximising the fibre concentration.

(b) There was no teaching in D3 to use an extruder; on the contrary, the instruction was to decant off the serum of the coagulation and the water after washing.

(c) The disclosure of D1, which was published long after D3, and closer to the filing date of the application in suit, required the polymer latex to be added to a solution of coagulant which also contained fibres; this was contrary to the subject-matter of Claim 1, which specified that the coagulant be added to a mixture of latex and fibres. Thus, the claimed subject-matter was based on a surprising effect, since, according to D1, the use of a fibre slurry not previously dispersed

in a coagulant would lead to a non-uniform dispersion of the fibres.

(d) There was no support for the assertion that fibres might be present in the extruder which was disclosed in D2 for the recovery of coagulated polymer.

In summary, the suggested combination of references was based on a selection of elements corresponding to the claimed subject-matter, using the disclosure of the application in suit as a guide, whilst ignoring those elements which did not so correspond. Consequently, the claimed subject-matter was not obvious.

IV. The Appellant requested that the decision under appeal be set aside and a patent granted, on the basis of the set of Claims 1 to 12 filed on 31 May 1995.

Reasons for the Decision

1. The appeal is admissible.

2. The text on which this decision is based comprises:

Claims: Claims 1 to 12 as filed on 31 May 1995, with letter dated 29 May 1995;

Description: Pages 1, 3 to 5 and 7 to 23, filed on 24 November 1994, with letter dated 22 November 1994; and

pages 2 and 6, filed on 31 May 1995, with letter dated 29 May 1995;

Drawings: Sheets 1/3 to 3/3 as originally filed.

3. *Allowability of the amendments*

3.1 Claim 1 is based on Claim 1 as originally filed, read in conjunction with the description as originally filed on page 6, lines 31 to 35 (printed specification, page 4, lines 14 to 16), as well as on page 7, lines 26, 27 and page 8, lines 17 to 25 (printed specification, page 4, lines 30 and 43 to 47).

3.2 Claims 2 to 10 and 12 correspond to Claims 2 to 10 and 12, respectively, as originally filed.

3.3 Claim 11 is based on Claim 11 as originally filed, read in conjunction with page 11, lines 21 to 23 and page 12, lines 27 to 30 of the description as originally filed (printed specification, page 5, lines 39 to 40; page 6, lines 1, 2).

3.4 The description contains no amendments which, in the Board's view, would contravene the requirements of Article 123(2) EPC.

3.5 Thus, the Board confirms the finding in the decision under appeal that the requirements of Article 123(2) EPC are fulfilled.

4. *The application in suit; the closest state of the art*

The application in suit is concerned with a process for incorporating fibrous filler into elastomeric polymers (opening paragraph; Claim 1). According to the description, this has been done by heating the polymers to soften them and thoroughly mixing the polymer and filler on a mill or internal mixer (page 1, lines 14 to 16).

Such a process is illustrated by D3, which according to the decision under appeal represents the closest state of the art, a view shared by the Board.

4.1 According to D3, problems encountered in obtaining uniform dispersion of the fibres throughout the rubber matrix during a reasonable and practical mixing cycle are solved in that fibrous filler material is coprecipitated with a latex of a rubber or plastic polymer to form a homogeneous predispersion of fibres. Such predispersed fibre compositions are mechanically mixed with the rubber or plastic compound stock, whereby the greater the homogeneity of the fibre predispersion, the more rapid, uniform and thorough will be the dispersion of the fibrous material into the rubber or plastic compound stock to be reinforced (column 1, lines 27 to 30; column 2, lines 8 to 12 and 14 to 27).

Whilst the concentrations of fibrous filler material and the binder comprising the polymer latex are not critical, it is desirable to maximise the concentration of the fibrous material, firstly since the composition of the rubber latex may not be the

same as that of the rubber stock to be reinforced, and secondly to reduce the cost of a given amount of predispersed fibres to be introduced into the rubber stock (column 4, lines 4 to 38).

In order to obtain a predispersed fibre composition having the greatest possible degree of homogeneity, a total amount of water has to be present in the mixture, prior to coagulation, which is not less than that required completely to wet the fibrous material, but not so much that the polymer is coagulated independently of the fibres (column 2, lines 28 to 43).

To ascertain the relevant solids content, a curve is plotted of the standard deviations, derived from a sufficient number of samples taken from each one of a series of coagulated test wetted fibre compositions having different percents of total solids, decreasing in equal increments from the point required completely to wet the fibrous material, against the percent of total solids in the relevant test wetted fibre composition. From this curve, a percent of total solids corresponding to the required homogeneity is selected (Claim 1).

The polymer latex is first combined with any optional ingredients and any required water of dilution, the mixture blended with the fibrous filler material, and the resulting wetted fibre mixture coagulated by mixing it with a solution of a coagulant. The serum is then decanted off. Wash water is added and then also decanted off. Finally, the predispersed fibre

composition is dried by suitable means, such as a forced air oven or partial vacuum evaporation (column 4, lines 44 to 59).

According to a typical example (Example 20), such a predispersed fibre composition comprises 79.21 wt% chopped 1/4-inch polyester fibre and 19.80 wt% rubber (column 9, 10, Table III).

Furthermore, according to an example of application (Example XXXIII), such a predispersed fibre composition is mixed into uncured rubber compound stock, in a two-roll laboratory mill, and found to be rapidly, uniformly and thoroughly dispersed into the rubber compound stock (column 11, line 54 to column 12, line 17).

4.2 As is evident from the number of sequential steps required, this method is not only cumbersome, but also both energy intensive and expensive, due to the long times required by the fabricator to incorporate fibre into the elastomer (application in suit, page 1, line 33 to page 2, line 9). Compared with this state of the art, therefore, and in line with the approach taken in the application in suit, the technical problem may be seen in the search for a simpler, cheaper and more efficient process of incorporating fibrous filler uniformly into an elastomer.

4.3 The solution proposed according to Claim 1 of the application in suit is to dispense with the predispersed fibre composition altogether, and

instead to feed the elastomer latex and an aqueous slurry of the fibrous material direct to a dewatering extruder having a downstream flow restriction, adding a coagulant in the extruder to form a coagulated elastomer containing the fibrous filler, feeding the coagulated fibre-filled elastomer through the flow restriction so that water present in the coagulated elastomer is forced out of a vent provided upstream of the flow restriction, and discharging and recovering the fibre filled elastomer.

4.4 It can be seen from the large number of examples in the application in suit that the process successfully enables an acceptably uniform dispersion of fibres for use, for instance, in making power transmission belts, to be obtained in a single step (Examples 2 to 5).

4.5 The finding in the decision under appeal, that the problem to be solved was simply to provide a further process for incorporating synthetic fibrous filler into an elastomer cannot be supported by the Board, not only since it diverges from the approach advocated in the case law of the Boards of Appeal, of normally starting from the problem actually described by the Applicant (T 246/91 of 14 September 1993, referring to T 495/91 of 20 July 1993, neither published in OJ EPO), but also because it ignores the facts that the claimed procedure not only (i) avoids the necessity of preparing a "predispersed fibre composition" having a particular water content, which has to be calculated from a standard deviation curve, itself derived from the results of a series of

iterative experiments, but also (ii) that it combines the coagulation and compounding steps in a single, quasi-continuous operation, while still giving an acceptable fibre distribution in the elastomer. Thus, the process is simpler, more efficient and consequently cheaper than that of D3.

4.6 In summary, it is credible that the claimed measures provide an effective solution of the problem as stated by the Board.

5. *Novelty*

Lack of novelty was not a ground of refusal of the application. Nor does the Board take the view that such an objection arises. Consequently, the claimed subject-matter is held to be novel.

6. *Inventive step*

To assess whether the claimed subject-matter involves an inventive step, it is necessary to consider whether the skilled person, starting from D3 and wishing to simplify and improve the efficiency of the process, would realise, in the ordinary course of his work, that the multi-stage procedure involving the preparation of an intermediate "predispersed fibre composition" according to the closest state of the art could be omitted, and a satisfactory result obtained in a single step, by using an adapted extruder for the combined operations of coagulation, de-watering and compounding.

6.1 There is no suggestion in D3 that the step of

preparing the "predispersed composition" could be omitted. On the contrary, such an intermediate product is essential to provide a source of prepared fibres which will uniformly and rapidly disperse when directly compounded with further stock using a conventional device (section 4.1, above).

6.1.1 The finding in the decision under appeal, that there was no restriction in D3 regarding the means suitable for drying fibre-filled material (Reasons for the decision, point 4.4, last sentence) ignores the fact that D3 only teaches one procedure for the mixing step, and this involves decanting off the serum (column 4, lines 44 to 59). This means that whatever kind of apparatus was envisaged according to D3, it could not have been a dewatering extruder.

6.1.2 The further finding of the decision under appeal, that it would have been obvious for the skilled person to use a dewatering extruder for this purpose, fails to recognise the nature of the mixing step in D3, which, as pointed out above, is merely a pretreatment to deposit polymer on the individual fibres preparatory to compounding them in a conventional device. It therefore need involve only relatively small amounts of water.

6.1.2.1 In the latter connection, the argument in the decision under appeal that there is no restriction on the concentration of the components of the "predispersed fibre composition" is not convincing, because the passage relied upon refers only to the starting ingredients, not to the resulting

"predispersed fibre composition" (column 4, lines 4 to 6).

6.1.2.2 On the contrary, it is evident from the relevant disclosure in D3 that the aim is that of maximising the concentration of fibrous material, none of the exemplified compositions having a fibre content less than 50 wt%, and around 80 wt% being typical (column 4, lines 12 to 18; examples).

6.1.2.3 Thus, it is evident that, in practice, the "predispersed fibre composition" consists essentially of fibrous material wetted with a small amount of coagulated elastomer latex, from which the water can therefore easily be removed.

6.1.2.4 Hence, there is no need to use an expensive and elaborate apparatus such as a dewatering extruder to dry the wet "predispersed fibre composition".

6.1.3 On the contrary, the further compounding involved in such use would tend to destroy the prepared condition of the "predispersed fibre composition", required for the subsequent conventional compounding step, and thus conflict with the purpose of preparing such an intermediate product in the first place.

6.1.4 In summary, the use of a dewatering extruder at this stage of the process according to D3 would be, at best, a completely redundant exercise in the use of expensive apparatus, and at worst, counterproductive to the point of vitiating the entire process.

6.1.5 Thus, there is no hint to the solution of the stated problem in D3.

6.2 According to D1, a process for the production of a mixture of a polymer and a fibrous material comprises agitating an aqueous solution of a coagulant for the polymer, adding an aqueous suspension of the fibrous material to the coagulant solution; adding the polymer, in aqueous latex form, to the coagulant solution; coagulating the polymer and the fibrous material; and recovering and drying the mixture of polymer and fibrous material (Claim 1). The coagulation product of the polymer and fibrous material is separated from the aqueous phase such as by mechanical separation means or by filtration, may be washed with water, is recovered and the wet particles of polymer-fibrous material mixture are dried, such as in a hot air dryer or in a dewatering-drying means. The product may be used in automotive products, such as tyres, or in mixtures with one or more compatible polymers not containing fibres (page 2, line 60 to page 3, line 10). In all such uses, the polymer-fibrous material mixture is compounded with rubber compounding ingredients, and with vulcanisation active agents using rubber mills or internal mixers (page 3, lines 18 to 21).

According to a comparative example, in which a water-wetted pulp of 4 mm aramid fibre pulp was suspended in water, added to a styrene-butadiene latex and coagulated with a 1 percent solution of calcium chloride, there was agglomeration of at least part of the polymer, which could not be recovered (page 3;

Example 1, Experiment 1). In an illustrative embodiment, however, the fibre was suspended in water and then mixed with the calcium chloride solution. On adding the latex to the stirred mixture, a uniform mixture without significant agglomeration was obtained (Example 1; Experiment 3).

6.2.1 Whilst the disclosure of D1 admittedly refers to "mechanical separation means" for removing the aqueous phase after coagulation, and to a "dewatering-drying means" as one of the options for drying the coagulated product, there is no suggestion of combining these steps in a single operation, let alone of using a dewatering extruder.

6.2.2 Even if there had been, however, it is an absolute requirement of D1 that the aqueous fibre slurry is added to a solution of coagulant. In this connection, it is evident, both from the comparative data (Experiment 1, above) and from a specific statement in D1 regarding the disadvantages of the prior art (page 1, lines 58 to 65), that it was considered impossible to form a uniform mixture with a dispersion of fibres in water as opposed to in a coagulating agent.

6.2.3 Consequently, even if the skilled person were to think of utilising a dewatering extruder in the process of D1, in spite of the absence of any hint in this direction, this would involve adding the aqueous dispersion of fibres to a coagulating agent, rather than to the latex as required by the application in suit. In other words, following the teaching of D1

would not lead the skilled person to the solution of the stated problem.

6.3 According to D2, which was published in January 1979, it was known to isolate a polymer, such as a chloroprene polymer, from its latex by introducing the latex and separately a latex coagulating agent, into a twin screw extruder (Claim 1). The latex may comprise a water-dispersible thickener, preferably hydroxyethylcellulose (column 4, lines 29 to 34).

6.3.1 There is, however, no reference to the presence of fibres, nor any indication that the extruder is suitable for processing a fibre containing mixture. Consequently, there is no support for the assertion in the decision under appeal that it was "well known that fibre containing masses could be processed in an extruder".

6.3.2 The onus of proving this assertion, which has been challenged by the Appellant, in any case lay with the Examining Division, and has not been discharged.

6.3.3 Even if the assertion were accepted at face value by the Board, however, the use of such an extruder as a drying means in the process according to D3 is practically excluded by the constellation of the latter process, since a mechanical compounding step is already envisaged in the latter, after drying has taken place (section 6.1.3, above).

6.3.4 Finally, D2 was published over ten years before the earliest priority date of the application in suit.

The fact that such use did not suggest itself to any operator, in a closely worked art such as that of reinforced polymers, for a full decade after the extruder became public knowledge, is an indication to the Board that general knowledge would not have sufficed to make available the solution of the technical problem to the skilled person.

6.3.5 Under these circumstances, the disclosure of D2 does not assist the skilled person to the solution of the technical problem, even in the light of his general technical knowledge, and whether or not considered in the light of the disclosure D1.

6.4 Thus, the solution of the stated problem does not arise in an obvious way from the state of the art.

6.5 Hence, the subject-matter of Claim 1, and, by the same token, of dependent Claims 2 to 12 involves an inventive step in the sense of Article 56 EPC.

7. Although the Board would be prepared to grant a patent on the basis of Claims 1 to 12, it is aware that certain passages of description are inconsistent with, and to this extent fall outside the scope of Claim 1. In particular, the reference to mechanical, as opposed to chemical coagulation (page 7, last line to page 8, line 8), the statement that it is preferred, as opposed to necessary, to add the coagulant (and acid) downstream of the point at which the slurry is added (page 8, lines 21 to 22) and the reference to an alternative embodiment, in which the coagulant may be mixed directly into the fibre slurry for separate injection (page 8, lines 26 to 29)

require amendment, and, in the Board's view in the cases of each "alternative" referred to, deletion, before grant can take place.

Under these circumstances, the Board has decided to make use of its powers under Article 111(1) EPC to remit the case to the Examining Division for the necessary consequential amendments to be made.

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 Claims 1 to 12 filed on 31 May 1995, after consequential amendment of the description.

The Registrar: The Chairman:

E. Görgmaier C. Gérardin