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Datasheet for the decision of 8 August 2007

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| IPC: | C09C 1/36 |
| Publication Number: | 0748359 |
| Application Number: | 95912062.7 |
| Case Number: | T 0538/02 - 3.3.06 |

Language of the proceedings: EN

Title of invention:

Improved processibility and lacing resistance when silanized pigments are incorporated in polymers

Patentee:

E.I. DU PONT DE NEMOURS AND COMPANY

Opponent:

KRONOS INTERNATIONAL, INC.

Headword:

Silanized titanium dioxide/DUPONT

Relevant legal provisions:

EPC Art. 56

Keyword:

"Inventive step (main request; auxiliary requests 1 to 3): no - hint in the prior art to improve dispersibility" "Inventive step (auxiliary request 4): yes - adding silanes to polydimethylsiloxane for silanizing titanium dioxide not derivable form prior art; effect proven"

Decisions cited:

-

Catchword:

-



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Beschwerdekammern

Boards of Appeal

Chambres de recours

Case Number: T 0538/02 - 3.3.06

DECISION of the Technical Board of Appeal 3.3.06 of 8 August 2007

| Appellant: | E.I. DU PONT DE NEMOURS AND COMPANY | | | | |
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Representative:

Decision under appeal: Decision of the Opposition Division of the European Patent Office posted 26 March 2002 revoking European patent No. 0748359 pursuant to Article 102(1) EPC.

Composition of the Board:

| Chairman: | Ρ | -P. | Bracke |
|-----------|----|-----|--------|
| Members: | G. | Rat | ths |
| | U. | Tro | onser |

Summary of Facts and Submissions

- I. This appeal is from the Opposition Division's decision to revoke European patent No. 0 748 359 relating to "[i]mproved processibility and lacing resistance when silanized pigments are incorporated in polymers".
- II. The patent as granted contained 40 claims of which, inter alia, the independent claims 1, 2 and 10 read as follows:

"1. A polymer matrix comprising polymer and about 0.01 to about 87% by weight silanized TiO₂ pigment, based on the weight of the polymer matrix, exhibiting enhanced processibility, wherein the silanized TiO₂ pigment has a coating of about 0.1 to about 5% by weight, based on the weight of the silanized TiO₂, of at least one organosilicon compound having the formula

R_xSi(R')_{4-x}

wherein

R is a nonhydrolyzable aliphatic, cycloaliphatic or aromatic group having 8-20 carbon atoms; R' is a hydrolyzable group selected from alkoxy, halogen, acetoxy or hydroxy or mixtures thereof; and x = 1 to 3."

"2. A polymer matrix comprising polymer and about 0.01 to about 87% by weight silanized TiO_2 pigment, based on the weight of the polymer matrix, wherein the silanized TiO_2 pigment has a coating of about 0.1 to about 5% by weight based on the weight of the silanized TiO_2 , of an organosilicon compound comprising a mixture of (a) and (b) wherein

(a) is at least one silane having the formula: $R_{x}\text{Si}\left(R^{\,\prime}\right)_{4-x}$

wherein
R is a nonhydrolyzable aliphatic, cycloaliphatic or
aromatic group having 1-20 carbon atoms;
R' is a hydrolyzable group selected from alkoxy,
halogen, acetoxy or hydroxy or mixtures thereof; and
x = 1 to 3; and

(b) is at least one polysiloxane having the formula



wherein

R is an organic or inorganic group; n = 0-3; and $m \ge 2.$ "

"10. The composition of Claim 1, Claim 2, Claim 8 or Claim 9 wherein the silanized pigment is present in the amount of about 70 to about 82% by weight and the organosilicon compound is selected from the group consisting of octyltriethoxysilane, nonyltriethoxysilane, decyltriethoxysilane, dodecyltriethoxysilane, tridecyltriethoxysilane, tetradecyltriethoxysilane, pentadecyltriethoxysilane, hexadecyltriethoxysilane, heptadecyltriethoxysilane, octadecyltriethoxysilane, mixtures thereof; and mixtures of butyltrimethoxysilane and polydimethylsiloxane, and mixtures of octyltriethoxysilane and polydimethylsiloxane."

III. A notice of opposition to the European granted patent was given in accordance with Article 99(1) EPC. The opponent (now the respondent) based its opposition on lack of sufficiency of disclosure (Articles 100 (b), 83 EPC), novelty and inventive step (Articles 100 (a), 54 and 56 EPC).

Inter alia, the following documents were filed during the opposition proceedings:

- (1) AU-A-88 180/91 and
- (3) Derwent Abstract AN 84-103778 of JP-A-59045906, patent application 57-155849.
- IV. During the oral proceedings before the Opposition Division, novelty and sufficiency of disclosure were no longer disputed by the respondent. In its decision the Opposition Division held that the subject-matter of Claim 1 of the then pending main request and auxiliary request did not involve an inventive step (Article 56 EPC), both requests having been submitted during the oral proceedings.
- V. On 23 May 2002 the proprietor of the patent (now the appellant) filed an appeal against this decision. On 30 July 2002, it filed the grounds of appeal, document

(Y) Tiona RCL-69, a sales brochure of SCM Chemicals, and requested the reimbursement of the appeal fee.

In its letter dated 26 October 2005 the respondent withdrew the opposition, accepted the arguments of the appellant and acknowledged the validity and enforceability of the claims of the main request dated 25 July 2002. VI. With the letter dated 6 July 2007 the appellant withdrew the request for the reimbursement of the appeal fee and submitted a new main request, and new first and second auxiliary requests as well as document

(Z) annex 1, a test report.

Under cover of the letter dated 2 August 2007, the appellant submitted a translation of the whole examined patent application corresponding to document (3), namely

(X) JP-B2-57 155849.

VII. During the oral proceedings before the Board which took place on 8 August 2007, the appellant filed additionally auxiliary requests 3 and 4.

VIII. Claim 1 of the main request reads:

"1. A polyolefin matrix comprising polyolefin and about 50 to about 87% by weight silanized TiO₂ pigment, based on the weight of the polyolefin matrix, exhibiting enhanced processability, wherein the silanized TiO₂ pigment has a coating of about 0.1 to about 5% by weight, based on the weight of the silanized TiO₂, of at least one organosilicon compound selected from octyltriethoxysilane, nonyltriethoxysilane, decyltriethoxysilane, dodecyltriethoxysilane, tridecyltriethoxysilane, tetradecyltriethoxysilane, pentadecyltriethoxysilane, hexadecyltriethoxysilane, heptadecyltriethoxysilane, octadecyltriethoxysilane, and mixtures thereof." Claim 1 of <u>auxiliary request 1</u> differs from Claim 1 of the main request in that the range "about 50 to 87% by weight" was replaced by "about 70 to about 87% by weight".

Claim 1 of <u>auxiliary request 2</u> differs from Claim 1 of the main request in that the range "about 50 to 87% by weight" was replaced by "about 70 to about 82% by weight".

Claim 1 of a<u>uxiliary request 3</u> differs from Claim 1 of the main request in that the words "at least one organosilicon compound selected from" and the passage "nonyltriethoxysilane, decyltriethoxysilane, dodecyltriethoxysilane, tridecyltriethoxysilane, tetradecyltriethoxysilane, pentadecyltriethoxysilane, hexadecyltriethoxysilane, heptadecyltriethoxysilane, octadecyltriethoxysilane and, mixtures thereof" were deleted.

Claim 1 of auxiliary request 4 reads:

"1. A polyolefin matrix comprising polyolefin and about 50 to about 87% by weight silanized TiO₂ pigment, based on the weight of the polyolefin matrix, wherein the silanized TiO₂ pigment has a coating of about 0.1 to about 5% by weight, based on the weight of the silanized TiO₂, of an organosilicon compound comprising a mixture of (a) and (b) wherein (a) is selected from octyltriethoxysilane, nonyltriethoxysilane, decyltriethoxysilane, dodecyltriethoxysilane, tridecyltriethoxysilane, tetradecyltriethoxysilane, pentadecyltriethoxysilane,

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hexadecyltriethoxysilane, heptadecyltriethoxysilane, octadecyltriethoxysilane and, mixtures thereof; and (b) is at least one polysiloxane having the formula $(R_n SiO_{(4-n)/2})_m$

wherein R is an organic or inorganic group n = 0-3; and m ≥ 2; or wherein (a) is butyltrimethoxysilane and (b) is polydimethylsiloxane; and wherein the weight ratio of silane (a) to polysiloxane (b) is from 1:2 to 2:1." The dependent claims 2 to 6 represent particular embodiments of the subject-matter of Claim 1. Independent Claim 7 reads: "7. A process for preparing a concentrate of a silanized TiO₂ pigment in a polyolefin comprising the steps of: (a) treating TiO₂ pigment with a mixture of a silane

compound selected from octyltriethoxysilane, nonyltriethoxysilane, decyltriethoxysilane, dodecyltriethoxysilane, tridecyltriethoxysilane, tetradecyltriethoxysilane, pentadecyltriethoxysilane, hexadecyltriethoxysilane, heptadecyltriethoxysilane, octadecyltriethoxysilane, and mixtures thereof; and a polysiloxane compound having the formula

(R_nSiO_{(4-n)/2})_m

wherein R is an organic or inorganic group n = 0-3; and m \ge 2; wherein the weight ratio of silane to polysiloxane is from 1:2 to 2:1; to form silanized TiO₂ pigment, and (b) mixing the silanized TiO₂ pigment with a polyolefin resin to form a highly loaded polyolefin concentrate comprising about 50 to about 87% by weight silanized TiO₂ pigment."

The dependent claims 8 to 13 represent particular embodiments of the subject-matter of claim 7.

IX. In writing and orally, the appellant submitted in essence the following arguments:

> The Opposition Division did not assess correctly inventive step when applying the problem solution approach. Document (1) would not be a suitable starting point because the purpose of this document would not concern masterbatches (called polymer matrices in Claim 1 i.e. polyolefin compositions containing high amounts of TiO₂) and their processability.

Commercially available masterbatches containing Tiona RCL-69 would represent a suitable starting point since Tiona RCL-69, even if its precise composition was not made available to the public, is titanium dioxide treated with polydimethylsiloxane. It is used in high concentration masterbatches of polyethylene and polypropylene.

When starting from commercially available masterbatches it would not be possible to rely on document (1) because incorporation of silanized titanium dioxide according to document (1) could only be done at conventional concentrations.

The commercial success of the polymer matrices as claimed - called also masterbatches in this case would be indicative of the superior masterbatch performance.

Document (3) disclosing mixtures of silanes and polydimethylsiloxane would not be relevant for the subject-matter of the patent in suit because the type of silanes would differ from that of the patent in suit.

X. The appellant requested that the decision under appeal be set aside and that the patent be maintained on the basis of the main request or auxiliary request 1 or 2 respectively filed under cover of the letter dated 6 July 2007 or on the basis of auxiliary request 3 or 4 both of them filed during oral proceedings before the Board.

Reasons for the Decision

Main request

1. The subject matter of Claim 1 of the main request is directed to a polyolefin matrix comprising polyolefin and about 50% to about 87% by weight of silanized TiO₂ pigment having a coating of an organosilicon compound selected from the silanes as defined in said claim (see point VIII).

1.1 Article 56 EPC

1.2 The problem to be solved according to the patent in suit was

(a) to improve processability in compounding of whitepigmented polymers i.e. dispersibility of TiO_2 pigment in a polymeric matrix;

(b) to improve performance properties such as lacing resistance in a polyolefin matrix. Lacing occurs as a result of volatiles released from the pigment during high temperature polyolefin fabrication processes; and
(c) to avoid slower processing rates due to higher loadings of TiO₂ pigment (page 2, lines 14 to 19).

- 1.3 During oral proceedings before the Board, the appellant did not mention the problem of lacing but focused only on the problems identified under points 2.2 (a) and 2.2 (c) i.e. in summary, the improvement of processability.
- 1.4 The appellant argued that document (1) would not be a suitable starting point since it did not concern masterbatches and did not address the problem of processability but of yellowing. It would be more appropriate to take commercially available masterbatches as a starting point.

For comparison purposes it relied on masterbatches containing 70% by weight of titanium dioxide treated with polydimethylsiloxane which were representative for masterbatches according to document (Y). This document recommends Tiona RCL 69, i.e. a polydimethylsiloxane treated TiO₂ pigment, for high concentration masterbatches, comprising, in particular, polyethylene and polypropylene; the surface treatment is specifically designed for outstanding dispersion properties. The masterbatches contain e.g. 60% by weight of TiO₂ (page 3, applications selector, left column, lines 9 to 16; page 6, left column, lines 14 to 18; page 6, left column, lines 1 to 4 from the bottom). In view of the teaching of document (Y), the appellant proposed to take masterbatches containing 70% by weight of titanium dioxide treated with polydimethylsiloxane as a starting point for assessing inventive step.

The Board can agree with this approach.

- 1.5 In the light of the teaching of document (Y), the problem underlying the patent in suit was to improve the processability of the then commercially available masterbatches containing polydimethylsiloxane treated TiO₂ pigment.
- 1.6 Processability is evaluated in the patent in suit in terms of bulk density, total flux time and viscosity of masterbatches.
- 1.7 A masterbatch of polyethylene comprising 70 wt.% of titanium dioxide treated with 1 wt.% of octyltriethoxysilane according to invention example 7 of the patent in suit has a total flux of 30.2 seconds (abbreviated as s), whereby it should be taken into consideration that the lower the value of total flux the better the improvement; if according to invention example 8, octyltriethoxysilane is replaced with octadecyltriethoxysilane the total flux is 33 s.

For comparison purposes, a masterbatch of polyethylene comprising 70 wt% of titanium dioxide treated with

1 wt% of polydimethylsiloxane according to comparative example 7A, i.e. a masterbatch being an embodiment representative for the prior art according to document (Y), has a total flux of 35.3 s.

Hence, the total flux of the masterbatches according to invention examples 7 and 8 is better than that of the masterbatch according to comparative example 7A.

The Board concludes that the problem of improving processability of masterbatches of polyethylene comprising titanium dioxide treated with polydimethylsiloxane is plausibly solved with masterbatches of polyethylene comprising titanium dioxide treated with silanes according to Claim 1.

- 1.8 The question is whether this technical solution involves an inventive step, or in other terms, whether the use of titanium dioxide treated with octyltriethoxysilane or with octadecyltriethoxysilane or with any of the other silanes mentioned in Claim 1 for improving processability was obvious.
- 1.9 Silanes of the type defined in Claim 1 of the main request are disclosed by document (1) which also addresses the dispersibility properties of silanized titanium dioxide in polyolefins.

The appellant argued during oral proceedings - that the skilled person would not turn to document (1) when trying to improve processability since the property of processability was not addressed in this document which would not disclose any characteristics such as bulk density, total flux and viscosity which would be significant for processability, - that document (1) would further not mention masterbatches and would relate to yellowing, and - that processability according to the patent in suit is different from dispersibility according to document (1).

1.10 The Board does not accept the appellant's arguments for the following reasons:

The Board refers to the definition of processability given by the appellant itself in the patent in suit:

"processability i.e. dispersibility of TiO_2 pigment in a polymer matrix..." (page 2, lines 15 to 16).

This means that dispersibility and processability are, in this case, synonyms. As to the properties measured in the tests according to the patent in suit, there is no doubt that the data suitably quantify processability characteristics or - because of the above mentioned definition - dispersibility characteristics. Of the three physical properties (bulk density, total flux and viscosity), the total flux is the most relevant since it indicates the time necessary to process the mixture titanium dioxide/polyolefin until the titanium dioxide is dispersed into the melted resin (patent in suit, page 4, lines 57 to 58). This definition of total flux confirms that dispersibility (see 'time for dispersing titanium dioxide in the melted resin') and processability (see 'processing time of the mixture polyolefin/titanium dioxide') are interrelated. Hence,

in this case, dispersibility is synonymous with processability.

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The fact that document (1) does not mention masterbatches is not relevant because the skilled person was looking for an improvement of dispersibility properties of titanium dioxide in polyolefins. The loading of masterbatches at concentrations of at least 50% by weight is considered to be state of the art (see loading of 60% by weight (document (Y) and loading of 70% by weight for the comparative example 7A provided by the appellant itself). Said concentrations fall within the range of 50 to 87 % by weight of Claim 1.

The appellant argued that document (1) would only concern low pigment concentrations. The Board however observes that document (1) does neither disclose explicitly low concentrations nor high concentrations of pigment loading. Therefore, the conclusion that document (1) would be limited to low concentrations of pigment cannot be accepted.

The problem of yellowing mentioned in document (1) (page 1, lines 21 and 28) does not distract from the fact that this document, even if it does not disclose values for the total flux time or for any other of the processability characteristics, also addresses the issue of dispersibility as is apparent from the following passage:

"The pigments coated reactively with silanes of the formula (I) have good mechanical properties (for example no tendency to form agglomerates, good dispersibility), so that their incorporation into the most diverse plastics systems presents no problems and can be carried out by conventional processes. Systems ... based on thermoplastic polymers or elastomers are preferred; plastics systems based on polyethylene, propylene ... are particularly preferred."

(page 6, line 39 to page 7, line 10).

Since document (1) teaches good dispersibility properties and easy incorporation of titanium dioxide treated with silanes of formula (I) in polyolefins, it follows that the skilled person would consider these silanes to improve dispersibility properties in masterbatches.

As to the silanes, the formula (I) of silanes according to document (1) is $SiR_1R_2R_3R_4$ (page 1, line 35). At least one of $R_1R_2R_3R_4$ may be alkoxy having 1 to 20 C atoms, and in particular 1 to 10 C atoms, e.g. methoxy, ethoxy, and at least one other of the radicals $R_1R_2R_3R_4$ is alkyl having 1 to 30, preferably 5 to 30 e.g. decyl, undecyl, dodecyl, tridecyl, tetradecyl, pentadecyl, hexadecyl, heptadecyl and octadecyl (page 3, line 36 to page 4, line 30).

Since document (1) teaches good dispersibility properties when titanium dioxide is silanized with silanes according to formula (I), the skilled person would have tried these silanes in order to improve the dispersibility in high concentration masterbatches, or in terms of claim 1 of the main request in a polyolefin matrix comprising polyolefin and pigment. It follows that the use of silanes according to Claim 1 for silanizing titanium dioxide in order to improve the dispersibility was obvious.

Consequently, the subject-matter of Claim 1 does not involve an inventive step and, therefore, does not meet the requirements of Article 56 EPC.

Auxiliary requests 1 and 2

2. Claim 1 of auxiliary request 1 differs from Claim 1 of the main request in that the range "about 50 to about 87% by weight" was replaced by "about 70 to about 87% by weight".

> Claim 1 of auxiliary request 2 differs from Claim 1 of the main request in that the range "about 50 to about 87% by weight" was replaced by "about 70 to about 82% by weight".

2.1 Inventive step

The starting point for assessing inventive step is the same as that taken for the subject-matter of Claim 1 of the main request (see point 1.4). The appellant choose a loading concentration of 70% by weight for comparison purposes (see point 1.7, paragraph 2 and point 1.10, paragraph 4), and so the masterbatch of example 7A with a loading of 70% by weight of silanized titanium dioxide is representative for high concentration masterbatches according to document (Y).

The masterbatches according to invention examples 7 and 8 comprised also 70% by weight silanized titanium

dioxide. This concentration of 70% by weight falls within the ranges of 70 to 87% by weight (Claim 1 of auxiliary request 1) and 70 to 82% by weight (Claim 1 of auxiliary request 2). Therefore, the reasoning as set out under points 1.2 to 1.10 applies mutatis mutandis to the subject-matter of each of Claim 1 of the auxiliary requests 1 and 2.

Consequently the subject-matter of Claim 1 of auxiliary requests 1 and 2 does not involve an inventive step and, therefore, Claim 1 of auxiliary requests 1 and 2 does not meet the requirements of Article 56 EPC.

Auxiliary request 3

3. The subject-matter of Claim 1 of auxiliary request 3 is limited to octyltriethoxysilane to be used as silanizing agent of titanium dioxide (see point VIII).

3.1 Inventive step

3.1.1 The starting point for assessing inventive step is the same as for the subject-matter of Claim 1 of the main request i.e. masterbatches comprising 70% of titanium dioxide treated with polydimethoxysilane representative for high concentration masterbatches according to document (Y). Therefore the reasoning as set out under points 1.2 to 1.10 applies mutatis mutandis to the subject-matter of Claim 1 of auxiliary request 3. In particular, it is the total flux of 30.2 s of the masterbatch according to invention example 7 which contains octyltriethoxysilane which proves to be better than the total flux of 35.3 s according to the masterbatch of the comparative example 7A containing polydimethoxysilane. Hence, the Board is satisfied that the problem of improving processability (or dispersibility) has been plausibly solved.

The question is whether the technical solution as proposed by Claim 1 implies an inventive step.

3.1.2 The appellant drew the attention to the passage in document (1) relating to silanes having preferably an alkyl group of more than 8 C atoms (page 4, lines 20 to 22), thus arguing that the skilled person would not have taken into consideration a silane with an octyl substituent or an alkyl group of less than 8 C atoms.

> It concluded that, therefore, the subject-matter of Claim 1 of auxiliary request 3 would involve an inventive step.

> The Board does not accept the arguments of the appellant. The crucial question to be answered is whether the skilled person would have got a hint in document (1) to use octyltriethoxysilane.

The formula (I) of silanes according to document (1) is SiR₁R₂R₃R₄ (page 1, line 35). At least one of R₁, R₂, R₃ or R₄ may be alkoxy having 1 to 20 C atoms, and in particular 1 to 10 C atoms, e.g. methoxy, ethoxy and at least one other of the radicals R₁, R₂, R₃ or R₄ is alkyl having 1 to 30, preferably 5 to 30. Hence, there was a hint to use silanes having ethoxy substituents and alkyls like methyl (C₁), pentyl (C₅) but also any other alkyl group having a number of C atoms between 5 and 30. The fact that alkyls having a number of C atoms greater than 8 are according to document (1) preferred does not exclude the use of alkyl groups having a number of C atoms lower than 8 or equal to 8, in particular because alkyls having 1 to 30, preferably 5 to 30, or more than 5, or more than 10 C atoms (page 5, line 27) are explicitly mentioned.

It may be that the performance with silanes having alkyl groups of less than 8 C atoms or equal to 8 C atoms is inferior to that with silanes having alkyl groups of more than 8 C atoms, but this is not a reason not to try these silanes, in particular because of the passage in document (1) on page 6, line 39 to page 7, line 5:

"The pigments coated reactively with silanes of the formula (I) have good mechanical properties (for example no tendency to form agglomerates, good dispersibility), so that their incorporation into the most diverse plastics systems presents no problems and can be carried out by conventional processes."

It follows that the subject-matter of Claim 1 of auxiliary request 3 does not involve an inventive step and, hence, Claim 1 of auxiliary request 3 does not meet the requirements of Article 56 EPC.

Auxiliary request 4

4. The subject matter of independent Claims 1 and 7 of auxiliary request 4 is directed to a polyolefin matrix comprising polyolefin and silanized TiO₂ pigment having a coating of an organosilicon compound comprising a mixture of silane and polysiloxane as defined in

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Claim 1, respectively, to a process for preparing a concentrate of silanized TiO_2 pigment in a polyolefin comprising the steps of treating TiO_2 pigment with a mixture of a silane compound and a polysiloxane compound as defined in claim 7 (see point VIII).

4.1 Article 123 EPC

Auxiliary request 4 comprises 13 claims which result from the deletion of previous claims of the main request and a renumbering respectively recombination of the remaining claims of the main request. The Board is satisfied that the amendments made to all the claims 1 to 13 of auxiliary request 4 meet the requirements of Article 123 EPC. It is not necessary to give detailed reasons for this finding since the respondent did not make any objections under Article 123 EPC for the main request.

4.2 Novelty

Novelty was never at issue during the proceedings. The Board is satisfied that the subject-matter of Claims 1 and 7 is novel in respect of documents (1) and (3), and that none of the other prior art documents anticipates the said subject-matter.

4.3 Inventive step

4.3.1 The problem to be solved according to the patent in suit is to improve processability in compounding of white-pigmented polymers i.e. dispersibility of TiO₂ pigment in a polymeric matrix. 4.3.2 As a starting point for evaluating inventive step the appellant proposed to take commercially available masterbatches known as masterbatches comprising Tiona RCL 69 according to document (Y) i.e. silanized TiO₂ pigment having a coating of polydimethylsiloxane.

- 4.3.3 In the light of this state of the art represented by commercially available masterbatches, the problem underlying the patent in suit is to improve processability with respect to processability of masterbatches containing TiO₂ treated with polydimethylsiloxane.
- 4.3.4 According to document (Z), a masterbatch containing 70% of silanized titanium dioxide comprising a coating of 1% by weight butyl trimethoxy silane and 1% by weight polydimethylsiloxane (invention example 1) was compared with a masterbatch containing 70% of silanized titanium dioxide comprising a coating of 1% by weight polydimethylsiloxane (comparative example 1A).

The total flux time of the masterbatch according to invention example 1 was 26 s and that according to the masterbatch according to comparative example 1A 38.5 s.

The masterbatches according to the examples 2 and 3 of the patent in suit comprising octyltriethoxy silane and polydimethylsiloxane have a total flux time of 26.2 s and 24.5 and, hence, have a better performance than the masterbatch according to comparative example 1A having a total flux time of 38.5 s.

It follows that a masterbatch having titanium dioxide silanized with a mixture of silane and

polydimethylsiloxane has a better flux time than a masterbatch having titanium dioxide silanized with polydimethylsiloxane only.

In the absence of evidence showing the contrary, the Board accepts that the problem underlying the patent in suit is plausibly solved over the whole area of Claim 1.

- 4.3.5 The question is whether the technical solution as proposed by the subject-matter of Claim 1 involves an inventive step, or in other words whether other prior art documents give a hint to the skilled person to use a mixture of silane and polydimethylsiloxane according to Claim 1 in order to improve processability (or dispersibility [see point 1.10]) characteristics over a polyolefin matrix comprising only polydimethylsiloxane and no silane.
- 4.3.6 Document (1) teaches to use silanized titanium dioxide having a coating of silanes. There is no hint to add polydimethylsiloxane to the silanes. So, this document does not offer any suggestion that the dispersibility of titanium dioxide can be improved by coating it with a mixture of silanes and polydimethylsiloxane.

Document (X) teaches to treat solid inorganic particles such as titanium oxide (page 3, line 2) with a mixture of silanes and polydimethylsiloxane in order to confer outstanding reinforcing effects and dispersion promoting effects in elastomer compositions (page 3, lines 25 to 29 and line 37). However, the silane coupling agents of document (X) differ from those of Claim 1 of auxiliary request 4 in that the type of substituents is different. Document (X) nor the other cited prior art documents hint to replace these specific silanes with silanes as defined in Claim 1 of auxiliary request 4, and there is no hint to combine silanes as defined in Claim 1 of auxiliary request 4 with polydimethylsiloxane to silanize titanium dioxide in view of improving the dispersibility thereof in high loaded masterbatches.

In the light of the teaching of the prior art documents on file the skilled person would not arrive at the subject-matter of Claim 1.

It follows that the subject-matter of Claim 1 involves an inventive step, and, therefore, meets the requirements of Article 56 EPC.

The dependent claims 2 to 6 represent particular embodiments of the subject-matter of Claim 1 and, therefore, derive their patentability from Claim 1.

4.3.7 Independent Claim 7 is directed to a process for treating titanium dioxide pigment with a mixture of a polysiloxane compound and a silane and mixing the silanized titanium dioxide with a polyolefin to form a highly loaded polyolefin concentrate.

The relevant feature of the process is the mixture of silane and polysiloxane with which the titanium dioxide is treated.

This mixture is part of the subject-matter of Claim 1. Hence, the reasoning under points 4.3.1 to 4.3.6 applies mutatis mutandis to the subject-matter of claim 7. It follows that the subject-matter of Claim 7 involves an inventive step as does the subject-matter of claims 8 to 13 which depend on claim 7 from which they derive their patentability since they represent particular embodiments of claim 7.

Order

For these reasons it is decided that:

- 1. The decision under appeal is set aside.
- 2. The case is remitted to the department of first instance with the order to maintain the patent on the basis of the claims 1 to 13 according to the fourth auxiliary request submitted during oral proceedings before the Board and the description to be adapted.

Registrar:

The Chairman:

G. Rauh

P.-P. Bracke