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# Datasheet for the decision of 8 March 2013

| Case Number:        | т 1831/10 - 3.3.06 |
|---------------------|--------------------|
| Application Number: | 00944734.3         |
| Publication Number: | 1194219            |
| IPC:                | B01F 17/00         |
|                     |                    |

Language of the proceedings: EN

## Title of invention:

Hydrolyzable silane emulsions and method for preparing the same

#### Patent Proprietor:

Momentive Performance Materials Inc.

## Opponent:

Evonik Degussa GmbH

#### Headword:

Silane emulsions/MOMENTIVE PERFORMANCE MATERIALS

# Relevant legal provisions (EPC 1973):

EPC Art. 83

#### Keyword:

"Sufficiency of disclosure (all requests): no - patent in suit not containing teaching enabling a skilled person to carry out all essential process steps of the claimed invention"

Decisions cited:

-

# Catchword:

-



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Boards of Appeal

Chambres de recours

**Case Number:** T 1831/10 - 3.3.06

## D E C I S I O N of the Technical Board of Appeal 3.3.06 of 8 March 2013

| Appellant: | Evonik  | Degussa   | GmbH   |   |
|------------|---------|-----------|--------|---|
| (Opponent) | Paul-Ba | aumann-St | crasse | 1 |
|            | D-45764 | 4 Marl    | (DE)   |   |

Representative:

Bendele, Tanja RUHR-IP Postfach 23 01 44 D-45069 Essen (DE)

Respondent: (Patent Proprietor) Momentive Performance Materials Inc. 187 Danbury Road Wilton, CT 06897-4122 (US)

Representative:

Roos, Peter Gille Hrabal Patentanwälte Brucknerstrasse 20 D-40593 Düsseldorf (DE)

Decision under appeal: Interlocutory decision of the Opposition Division of the European Patent Office posted 1 July 2010 concerning maintenance of European patent No. 1194219 in amended form.

Composition of the Board:

| Chairman: | Ρ  | -P.       | Bracke |
|-----------|----|-----------|--------|
| Members:  | L. | Li        | Voti   |
|           | J. | Geschwind |        |

## Summary of Facts and Submissions

- I. The present appeal is from the decision of the Opposition Division to maintain in amended form the European patent no. 1 194 219, concerning a process for preparing an oil-in-water emulsion.
- II. In its notice of opposition the Opponent sought the revocation of the patent on the grounds of Articles 100(a),(b) and (c) EPC 1973.
- III. The Opposition Division found in its decision, in particular, that the claims according to the main request filed during oral proceedings complied with all the requirements of the EPC.
- IV. An appeal was filed against this decision by the Opponent (Appellant).

The Appellant submitted two experimental reports by Dr. M. Friedel, dated 28 October 2010 and 9 November 2010, respectively (referred to hereinafter as EXP1 and EXP2). Moreover, it cited *inter alia* documents

(13): Römpp Lexikon Chemie, 10th edition (1997), Georg Thieme Verlag, page 1151, keyword "Emulsionen", and

(18): Ullmann's Encyclopedia of Industrial Chemistry, fifth edition (1987), vol. A9, pages 298 to 303 and 310 to 311.

The Respondent (Patent Proprietor) submitted with the letter of 15 June 2011 *inter alia* ten auxiliary requests.

Oral proceedings were held before the Board on 8 March 2013.

V. The independent claim 1 of the set of claims according to the Respondent's main request, which corresponds with the set of claims found by the Opposition Division to comply with all the requirements of the EPC, reads as follows:

> "1. A process for preparing an oil-in-water emulsion comprising at least one hydrolyzable silane, at least one emulsifier, and water, comprising the steps of:

I) dispersing at least one emulsifier in water at a weight ratio of 1.5:1.0 to about 1.0:4.0;

II) preparing a water-in-oil concentrate by mixing said emulsifier and water blend with at least one hydrolyzable water insoluble or only slightly soluble silane having the general formula:

R<sup>1</sup><sub>a</sub>R<sup>2</sup><sub>b</sub>Si(OR<sup>3</sup>)<sub>4-a-b</sub>

wherein

 $R^1$  is a hetero atom substituted hydrocarbon group;  $R^2$  is independently an unsubstituted hydrocarbon group;  $R^3$  is alkyl, alkoxyalkyl, aryl or aralkyl radicals having from 2 to 10 carbon atoms; and a is 0 to 3, b is 0 to 2;

with the proviso that a+b = 1, 2 or 3;

such that said oil concentrate comprises 1 part to 28 parts of said emulsifier and 1 part to 55 parts water, per 100 parts of said at least one hydrolyzable silane; III) slowly adding water to said oil concentrate until inversion of said emulsion occurs."

Claim 1 according to the first auxiliary request differs from claim 1 according to the main request insofar as step (II) of the process reads "preparing an oil concentrate..." instead of "preparing a water-inoil concentrate...".

Claim 1 according to the second auxiliary request differs from claim 1 according to the main request insofar as the weight ratio of emulsifier to water used in step (I) is of 1.5:1.0 to 1.0:3.0.

Claim 1 according to the third auxiliary request differs from claim 1 according to the main request insofar as it contains in step (II) the additional wording "and each R group is cyclic, branched or linear;" after "with the proviso that a+b= 1, 2 or 3;".

Claim 1 according to the fourth auxiliary request differs from claim 1 according to the main request insofar as it requires additionally that the pH of the total composition is 5.5 to 8.5.

Claim 1 according to the fifth auxiliary request differs from claim 1 according to the main request insofar as the hetero atom substituted hydrocarbon group  $R^1$  of the silane of given general formula is a monovalent organic radical linked to the Si atom of said silane by an Si-C bond, and which has at least one ether, ester, carbamate, isocyanate, thioether, polysulfide, blocked mercaptan, amide, cyano, epoxy, oximato group, or mixtures thereof, thereon.

Claim 1 according to the sixth auxiliary request differs from claim 1 according to the main request insofar as it requires that the silane of given general formula has at least one epoxy group or is selected from the group of 3-methyldiethoxysilylpropyl thioacetate, 3-trimethoxysilylpropyl thioacetate, 3triethoxysilylpropyl thioacetate, 3trimethoxysilylpropyl thiopropionate, 3triethoxysilylpropyl thiobenzoate, 3triethoxysilylethyl thioacetate, 3-triethoxysilylmethyl thioacetate, 3-triethoxysilylpropyl thiooctanoate, 3methacryloxypropyltriethoxysilane, 3methacryloxypropyltriisopropoxysilane, 3methacryloxypropyltriethoxysilane, 3mercaptopropyltriethoxysilane, vinylmethylbis-(isopropoxy) silane, 3methacryloxypropylmethyldibutoxysilane, 1,2 bis-(alkoxy) silyl ethanes, tris(3-triethoxysilylpropyl)isocyanurate, and bis(3-triethoxysilylpropyl)disulfide.

Claim 1 according to the seventh auxiliary request differs from claim 1 according to the main request insofar as the used silane is selected from the group of 3-methyldiethoxysilylpropyl thioacetate, 3trimethoxysilylpropyl thioacetate, 3triethoxysilylpropyl thioacetate, 3trimethoxysilylpropyl thiopropionate, 3triethoxysilylpropyl thiobenzoate; 3triethoxysilylpropyl thiobenzoate; 3triethoxysilylethyl thioacetate, 3-triethoxysilylmethyl thioacetate, 3-triethoxysilylpropyl thiooctanoate, β(3,4-epoxycyclohexyl)-ethyltriethoxysilane, 4-(methyldiethoxysilyl)-1,2-epoxycyclohexane, 3-(3,4epoxycyclohexyl)-propyltri(isobutoxy)silane, 3-(2,3epoxybutoxy)propyltriethoxysilane, [2.2.1]bicycloheptane 2,3-epoxy-5-(2-triethoxysilyl) ethyl,  $\beta$ -(3,4-epoxycyclohexyl)-ethyltriisopropoxysilane,  $\beta$ -(3,4-epoxycyclohexyl)-ethyltriisobutoxysilane, 3glycidoxypropyltriisobutoxysilane, 3methacryloxypropyltriethoxysilane, 3methacryloxypropyltriethoxysilane, 3methacryloxypropyltriethoxysilane, 3methacryloxypropyltriethoxysilane, 3methacryloxypropyltriethoxysilane, 3methacryloxypropyltriethoxysilane, 3-

methacryloxypropylmethyldibutoxysilane, 1,2 bis-(alkoxy)
silyl ethanes, tris(3-triethoxysilylpropyl)
isocyanurate, and bis(3-triethoxysilylpropyl)disulfide.

Claim 1 according to the eighth auxiliary request differs from claim 1 according to the main request insofar as the silane of given general formula has at least one epoxy group.

Claim 1 according to the ninth auxiliary request differs from claim 1 according to the main request insofar as the used silane is selected from the group of  $\beta$ -(3,4-epoxycyclohexyl)-ethyltriethoxysilane, 4-(methyldiethoxysilyl)-1,2-epoxycyclohexane, 3-(3,4epoxycyclohexyl)-propyltri(isobutoxy)silane, 3-(2,3epoxybutoxy)propyltriethoxysilane, [2.2.1]bicycloheptane 2,3-epoxy-5-(2-triethoxysilyl) ethyl,  $\beta$ -(3,4-epoxycyclohexyl)-ethyltriisopropoxysilane,  $\beta$ -(3,4-epoxycyclohexyl)-ethyltriisobutoxysilane, 3glycidoxypropyltriisobutoxysilane, and bis(3triethoxysilylpropyl)disulfide. Claim 1 according to the tenth auxiliary request differs from claim 1 according to the main request insofar as it specifies that the process concerns the preparation of an oil-in-water emulsion free from siloxane.

VI. The Appellant submitted in essence that

- step (III) of the claimed process required the inversion of a water-in-oil (w/o) emulsion into an oilin-water (o/w) emulsion; however, it had been shown by the repetition of example 1 of the patent in suit contained in EXP 1 and 2 as well as by the other 20 experiments contained in EXP 1 that it was not possible to obtain a w/o emulsion and an inversion of such an emulsion into the final o/w emulsion by following the teaching of the patent in suit; to the contrary, in some cases, an o/w emulsion was already obtained after step (II) or an emulsion was not obtained at all;

- since the patent in suit did not contain a teaching which would enable the skilled person to realise each step of the claimed process, the invention was not sufficiently disclosed.

VII. The Respondent submitted in writing and orally *inter* alia that

- the wording of the claims required the formation of an o/w emulsion by the steps of mixing water and emulsifiers at a given weight ratio, mixing this blend with a silane of given formula in the given proportions and adding slowly water thereto; - the patent in suit did not teach anywhere that the water-in-oil concentrate or oil concentrate obtained in step (II) should be a w/o emulsion; to the contrary, it would have been clear to the skilled person from the description of the patent in suit that the "inversion of the emulsion" occurring in step (III) of the process concerned only the thinning out of said oil concentrate with formation of an o/w emulsion and not the inversion of a w/o emulsion into an o/w emulsion; in fact, step (III) of the claimed process did not specify if and when an emulsion formed or an inversion occurred by slowly adding water;

- therefore, the objections raised by the Appellant were based on a misinterpretation of the wording of claim 1 and it was possible for the skilled person to carry out the invention by following the teaching of the patent in suit, as it had been confirmed by the test submitted as EXP 2 by the Appellant.

- VIII. The Appellant requests that the decision under appeal be set aside and the patent be revoked.
- IX. The Respondent requests that the appeal be dismissed or, in the alternative, that the patent be maintained on the basis of one of the first to tenth auxiliary requests submitted with the letter of 15 June 2011.

# Reasons for the Decision

- 1. Respondent's main request
- 1.1 Sufficiency of disclosure
- 1.1.1 The claimed process for preparing an o/w emulsion comprising at least one hydrolyzable silane of given formula (hereinafter H-silane) requires as essential features three distinct process steps; step (I) concerns the dispersion of at least one emulsifier in water at a given weight ratio; step (II) concerns the preparation of a so-called "water-in-oil concentrate" by mixing said emulsifier/water blend with at least one H-silane such that the resulting oil concentrate comprises defined parts of emulsifier, water and Hsilane; and step (III) concerns the slow addition of water to the oil concentrate obtained in step (II) until a so-called "inversion of said emulsion" occurs.

According to the Appellant the wording of step (III) of the claimed process requires that the oil concentrate of step (II) be present as a w/o emulsion and undergoes an inversion into the final o/w emulsion.

The Respondent submitted that the wording "water-in-oil concentrate" used in step (II) of the claimed process does not intend to represent a w/o emulsion and identifies only a mixture of emulsifiers, water and H-silane having the relative amounts indicated in the claim, wherein the H-silane represents the major part of the mixture.

Moreover, even though step (III) of the claimed process reads "adding water to said oil concentrate until inversion of said emulsion occurs", in the Respondent's view it would be clear to the skilled person in the light of the description (paragraphs 2, 15, 57, 59, 62) and of the examples of the patent that the wording "inversion of said emulsion" does not concern the inversion of a w/o emulsion, which is not explicitly mentioned anywhere in the patent in suit, but only the thinning out of the oil concentrate obtained in step (II).

1.1.2 The Board remarks that the patent in suit does not contain indeed any explicit definition for the terms "water-in-oil concentrate" used in claim 1 and "oil concentrate" used throughout the description, apart from the indication in paragraph 57 that the type of oil concentrate obtained in step (II) is sometimes referred to in the art as "grease".

> However, paragraph 62 of the patent in suit, relating to step (III) of the claimed process, reads as follows: "Water is then dispersed in the oil concentrate forming the desired oil-in-water type emulsion. Water is added slowly at first until the emulsion thins out. This is commonly called the inversion of the emulsion...".

> Therefore, this passage clearly states that the oil concentrate to which water is slowly added in said step (III) of the process is an emulsion, which thins out when its inversion occurs. This is also reflected in the wording of step (III) of claim 1 reading "slowly adding water to said oil concentrate until inversion of said emulsion occurs", wherein "said emulsion" can only refer to the "oil concentrate".

It was common general knowledge at the priority date of the patent in suit that the term "emulsion" represents a disperse system of two or more mutually insoluble or sparingly soluble liquids, wherein an external continuous phase contains an internal dispersed phase in the form of droplets. In the case of a water phase and an oil phase, depending on the nature of the external continuous phase, the emulsion thus is called w/o emulsion or o/w emulsion (see document (13), left column, and document (18), page 298, these documents being excerpts from a dictionary and an encyclopaedia representing the common general knowledge of the skilled person at the priority date of the patent in suit).

Moreover, it was also common general knowledge that the term "inversion", related to an emulsion, identifies the conversion of a w/o emulsion into an o/w emulsion, or viceversa, when the amount of the internal phase is increased (see document (13), right column, and document (18), page 302).

Therefore, in the Board's view, it would have been clear to the skilled person, by considering the whole content of the patent in suit and his common general knowledge, that the process of the invention requires the formation of an oil concentrate which is a w/o emulsion which undergoes an inversion to the final o/w emulsion in the step (III) of the claimed process.

The possibilities that an inversion could already occur in step (II), as suggested by the Respondent during oral proceedings, or that a w/o emulsion could be formed during step (III) before being inverted, concern process steps which are not part of the features of claim 1, are not mentioned anywhere in the description of the patent in suit and, in fact, are not in agreement with the essential process steps of claim 1. Therefore, such hypothetical process steps do not concern the invention upon the sufficiency of which the Board has to decide.

1.1.3 As shown in the experimental evidence submitted as EXP 1 and 2, a reworking of example 1 of the patent in suit, by using the same H-silane and the same emulsifiers, leads to a final o/w emulsion as required in the patent in suit but without formation of a w/o emulsion during step (II) of the process and, consequently, without inversion of a w/o emulsion into an o/w emulsion in step (III) of the process, as required by the claimed invention. Moreover, the other 20 experiments contained in EXP 1 show that, even varying the relative amounts of emulsifiers, water and H-silane within the broadest range indicated in step (II) of claim 1, a w/o emulsion is never formed in step (II) and, to the contrary, in some cases (experiments 1, 2, 6, 7, 8, 10, 11, 12, 15, B) an o/w emulsion is already formed in step (II). It is thus clear that in such cases an inversion of a w/o emulsion into an o/w emulsion, as required by the invention, cannot occur in step (III).

> The results of EXP 1 and 2 submitted by the Appellant were not contested by the Respondent. It was also not disputed that the patent in suit does not contain any example or general teaching explaining how it would be possible to obtain a w/o emulsion in step (II) of the process by using the amounts of H-silane, water and emulsifier indicated in the patent in suit, if the blending of such components is not sufficient for

obtaining such an emulsion, so that an inversion of such a w/o emulsion into an o/w emulsion can occur in step (III).

1.1.4 Therefore, the Board can only conclude that the patent in suit does not contain sufficient information which would enable a skilled person, with the knowledge of the patent in suit and on the basis of his common general knowledge, to carry out all the essential process steps of the claimed invention.

Therefore, the main request does not comply with the requirements of Article 83 EPC 1973.

- 2. First to tenth auxiliary requests
- 2.1 Sufficiency of disclosure
- 2.1.1 All processes claimed according to the ten auxiliary requests still require in step (III) the inversion of a w/o emulsion, which is the water-in-oil concentrate or oil concentrate of step (II), into an o/w emulsion.

Therefore, as regards sufficiency of disclosure, the Board finds that points 1.1.2 to 1.1.4 above apply *mutatis mutandis* to all auxiliary requests.

# Order

For these reasons it is decided that:

1. The decision under appeal is set aside.

2. The patent is revoked.

The Registrar:

The Chairman:

D. Magliano

P.-P. Bracke