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D E C I S I O N
of 29 March 2006

Case Number: T 1099/04 - 3.3.06

Application Number: 96929808.2

Publication Number: 0850293

IPC: C11D 3/33

Language of the proceedings: EN

Title of invention:

Succinic acid derivative degradable chelants, uses and compositions thereof

Patentee:

THE DOW CHEMICAL COMPANY

Opponent:

Mitsubishi Rayon Co. Ltd.

Headword:

Succinic acid derivatives/DOW CHEMICAL

Relevant legal provisions:

EPC Art. 54, 56

Keyword:

"Novelty (yes): method of preparation not leading unavoidably to a product - containing detectable amounts of a by-product"

"Inventive step (no): no synergistic effect proved over the whole scope"

Decisions cited:

-

Catchword:

-



Case Number: T 1099/04 - 3.3.06

D E C I S I O N
of the Technical Board of Appeal 3.3.06
of 29 March 2006

Appellant:
(Opponent)

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Decision under appeal:

Interlocutory decision of the Opposition
Division of the European Patent Office posted
21 July 2004 concerning maintenance of European
patent No. 0850293 in amended form.

Composition of the Board:

Chairman: P. Krasa
Members: L. Li Voti
A. Pignatelli

Summary of Facts and Submissions

I. The present appeal is from the decision of the Opposition Division concerning the maintenance in amended form of the European patent no. 0 850 293, relating to compositions comprising a combination of succinic acid derivatives as chelants.

II. In its notice of opposition the Opponent sought revocation of the patent *inter alia* on the grounds of Articles 100(a), because of lack of novelty and inventive step of the claimed subject-matter.

The following documents were referred to *inter alia* in support of the opposition:

(1): EP-B-0267653

(2): WO-A-9512570.

III. In its decision the Opposition Division found *inter alia* that

- the claims according to the second auxiliary request filed during oral proceedings complied with the requirements of Article 123(2) EPC;
- document (1) disclosed laundry detergent compositions comprising ethylenediamino disuccinic acid (**EDDS**) as chelating agent;
- the method of preparation of EDDS disclosed in this document could lead to the formation of minor

quantities of ethylenediamino monosuccinic acid
(**EDMS**);

- however, document (1) did not contain any unambiguous disclosure of the use of EDDS in combination with EDMS in a laundry detergent composition;
- the claimed subject-matter was thus novel over the cited prior art;
- moreover, the patent in suit showed that combinations of EDDS and EDMS had a better sequestering activity and a better biodegradability than it could have been expected from the known single activities of EDDS and EDMS;
- since the prior art did not suggest to replace part of the EDDS used in document (1) with EDMS for improving the sequestering activity and the biodegradability of the disclosed compositions, the claimed subject-matter involved also an inventive step.

IV. The set of 26 claims according to said second auxiliary request contained an independent claim 1 reading as follows:

"1. A laundry detergent composition comprising (a) from 1% to 80% by weight of a detergent surfactant selected from nonionic, anionic, cationic, zwitterionic, and ampholytic surfactants and mixtures thereof; (b) from 5% to 80% by weight of at least one detergent builder; and (c) from 0.1 % to 15% by weight of a combination of

chelants comprising at least one polyamino disuccinic acid and one or more polyamino monosuccinic acids, or salts thereof."

This set of claims contained also independent claim 4 relating to an automatic dishwashing composition comprising the same mixture of chelants, independent claims 22 to 25 relating to various methods involving the use of this mixture and, precisely a method of laundering fabrics, a process for removing H₂S from a fluid, a process for removing NO_x from a fluid and a method of electroless deposition of copper, respectively, and independent claim 26 directed to the use a composition comprising such a mixture of chelants.

Dependent claims 2 to 3 and 5 to 21 related to particular embodiments of the claimed laundry and dishwashing compositions.

V. An appeal was filed against this decision by the Opponent (Appellant).

In the statement of the grounds of appeal the Appellant referred additionally *inter alia* to the following documents

(11): US-A-3158635;

(12): GB-A-757704;

(13): Mr. Ansai's Test Report of 15 November 2004;

and in the letter of 26 January 2006 also to

(15): US-A-2761874.

The statement of the grounds of appeal contained also experimental data obtained by following the instructions of example 3 of the patent in suit.

Oral proceedings were held before the Board on 29 March 2006.

During oral proceedings the Respondent and Patent Proprietor filed two amended sets of claims to be considered as main and auxiliary request, respectively.

The set of claims according to the main request consisted of claims 1 to 25 of the second auxiliary request found by the Opposition Division to comply with the requirements of the EPC (see point IV above).

The set of claims according to the auxiliary request contained 24 claims and differed from that according to the main request insofar as claims 1 and 4 incorporated the feature of claim 19 of the main request that **the molar ratio of the polyamino disuccinic acid to the polyamino monosuccinic acid is from 99:1 to 5:95**, claims 20 to 25 being renumbered accordingly.

VI. The Appellant submitted in writing and orally *inter alia* that

- document (1) taught that EDDS could be prepared by the method described in document (11); as shown in Mr Ansai's report (document (13)), this method of preparation led inevitably to the formation of minor quantities of EDMS which were still present in the final product even after recrystallization;

- since document (1) did not require any specific purification step of the EDDS, it disclosed implicitly the use of an EDDS product containing inevitably minor quantities of EDMS;
- since the subject-matter of claim 1 according to the main request encompassed compositions comprising any detectable amount of EDMS, the claimed subject-matter lacked novelty;
- the alleged unexpected effect obtained by using a combination of EDDS and EDMS had not been shown for combinations comprising, for example, very low amounts of EDMS;
- as shown in the experiments submitted with the statement of the grounds of appeal, compositions comprising very small amounts of EDMS (molar ratio EDMS/EDDS of 1:99 or less) did not show any unexpected degree of chelating activity;
- moreover, claim 1 encompassed compositions comprising S,S-EDDS which was already known to have a very high biodegradability above the benchmark given in the patent in suit and high chelating activity; no evidence had been provided that a surprising effect had been achieved by using S,S-EDDS in combination with EDMS;
- since EDMS was a known chelating agent, it was obvious for the skilled person, in the attempt to provide an alternative composition, to replace minor quantities of the EDDS used in document (1) with EDMS;

- the claimed subject-matter lacked thus an inventive step.
- VII. The Respondent submitted in writing and orally *inter alia* that
- document (1) did not teach to use EDMS in combination with EDDS;
 - since it was already known how to prepare EDDS in pure form (see document (13)), the skilled person, following the teaching of document (1), would have prepared EDDS in such a way to obtain the most pure product possible;
 - therefore, document (1) did not disclose EDDS containing inevitably detectable quantities of EDMS;
 - the claimed subject-matter was thus novel over the cited prior art;
 - the tests provided in the patent in suit showed that the combination of EDDS with EDMS brought about a surprising synergistic effect in regard to chelating activity and biodegradability;
 - the tests filed by the Appellant with the statement of the grounds of appeal were not reliable;

- documents (12) and (15) taught that EDMS was a sequestering agent for heavy metals but did not suggest using it in laundry detergent compositions;
- since it was known that EDMS was not a good chelating agent at the alkaline pHs used for laundry detergent compositions, the skilled person would have not replaced part of the EDDS used in document (1) with EDMS;
- furthermore, the skilled person would have had no incentive to replace S,S-EDMS, which was already known to have a very high biodegradability and high chelating activity, with EDMS;
- the claimed subject-matter involved thus an inventive step.

VIII. The Appellant requests that the decision under appeal be set aside and that the patent be revoked.

The Respondent requests that the patent be maintained on the basis of claims 1 to 25 of the main request or of claims 1 to 24 of the auxiliary request, both filed during oral proceedings.

Reasons for the Decision

1. Respondent's main request

1.1 Article 123(2) EPC

The Board is satisfied that the claims according to the main request comply with the requirements of Article 123(2) EPC.

Since the Appellant only argued against the novelty and the inventiveness of the claimed subject-matter no further details are necessary.

1.2 Novelty

Claim 1 according to the main request relates to a laundry detergent composition comprising from 1% to 80% by weight of a detergent surfactant, from 5% to 80% by weight of at least one detergent builder and comprising from 0.1 % to 15% by weight of a combination of chelants comprising at least one polyamino disuccinic acid (**PADS**) and one or more polyamino monosuccinic acids (**PAMS**), or salts thereof.

This claim, not requiring any specific amounts of PADS and PAMS, relates thus to compositions comprising any amount of PADS and PAMS detectable by standard quantitative analytical methods normally used in preparative chemistry at the priority date of the patent in suit.

It is undisputed that document (1) discloses laundry detergent compositions comprising amounts of surfactants and builder corresponding with those of said claim 1. Furthermore, these compositions comprise 0.1 to 10% by weight of EDDS, i.e. a PADS according to the patent in suit, and, possibly other chelants; a

PAMS is not explicitly mentioned in this document (see claim 1 as well as page 9, line 56 to page 10, line 4).

Document (1) teaches that EDDS, depending on the selected method of preparation, can be obtained as S,S-EDDS or as a racemate mixture of the three optical isomers R,R-EDDS, S,S-EDDS and S,R-EDDS. This racemate mixture can be prepared, for example, by following the teaching of document (11) and, especially, of example 1 of this document (see document (1), page 3, lines 3 to 25 and page 7, last line to page 8, line 14).

Example 1 of document (11) reports the preparation and the recovering of the racemate EDDS in three different forms. Form (A) is a dihydrate obtained after washing the obtained crystals with 500 ml water, form (B) is an anhydrous product obtained after drying (A) at 90°C and form (C) is a pentahydrate obtained after passing boiling water through a pad of (A), the amount of boiling water used in this washing step not being specified (see column 4, lines 47 to 64).

The Appellant filed with the statement of the grounds of appeal Mr. Ansai's Test Report (document (13)) containing a reworking of example 1 of document (11).

According to this report said product (A) contains 1,7 moles% of EDMS, a PAMS according to the patent in suit, product (B) 1.2 moles% thereof and product (C) only 0.03 moles% thereof.

The Board notes that the products (A) and (B) obtained in document (13) have been prepared exactly as indicated in said example 1, whilst the product (C) has

been obtained by washing the product (A) with only 100 ml of hot water at 90°C.

Since the water used for obtaining the product (C) in document (13) was not at the boiling point and the quantity of water used could have been less than that used in document (11) which does not specify the amount used, it cannot be concluded with certainty that the product (C) obtained in document (13) is exactly the same as obtained in document (11).

Moreover, it can be derived from document (13) that the washing of the crystals with only 100 ml of hot water strongly reduces the quantity of EDMS contained in the final EDDS product from 1.7 to 0.03mole%, i.e. to very low amounts.

This result is not surprising since it was also already known how to prepare pure EDDS not containing any detectable amount of EDMS. This method, described on page 3 of document (13), involved the washing of the obtained crystals with great amounts of cool water (5 litres according to the Respondent's information) till **no EDMS could be detected.**

Therefore, it was known to the skilled person at the publication date of document (1) how to prepare an EDDS containing minor amounts of EDMS and how to easily reduce the amount of EDMS contained therein to undetectable amounts by simply washing with water and, especially, with boiling water as suggested in document (11).

Therefore, in the Board's view, the process of preparation disclosed in document (11) does not lead necessarily to an EDDS containing detectable amounts of EDMS but, depending on the selected washing conditions, could lead to pure EDDS as well.

Since the process of document (11) is only one of the processes of preparation of EDDS described in document (1) and this document does not specify which particular process of preparation according to document (11) should be used for obtaining the EDDS racemate (for example, whether the racemate should have been repeatedly washed with cold or hot water or not), the teaching of document (1) does not disclose unambiguously an EDDS product containing detectable amounts of EDMS and thus the use of a mixture of EDDS with EDMS.

Consequently, the subject-matter of claim 1 is novel over the teaching of document (1).

Since the prior art does not disclose the use of a combination of EDDS and EDMS, the subject-matter of the other claims is also novel.

1.3 Inventive step

- 1.3.1 The claimed invention and, in particular, the subject-matter of claim 1 relates to a laundry composition containing a combination of chelating agents providing an unexpected synergistic effect and, precisely, a better sequestering activity and a better biodegradability than it could have been expected from the known single activities of the single chelants (see

page 2, lines 3 and 31 to 34 and page 3, lines 21 to 26).

As explained in the description of the patent in suit, it was desirable that the used chelating agents were biodegradable to at least 60 percent within less than 28 days according to the OECD 301 B Modified Sturm Test (page 2, lines 28 to 30).

- 1.3.2 The most suitable starting point for the evaluation of inventive step is thus represented by document (1) which relates to laundry detergent compositions comprising an EDDS, e.g. S,S-EDDS, as chelating agent presenting good biodegradability and good sequestering activity (see claim 1; page 2, lines 5 to 11; page 3, lines 33 to 35; page 7, line 41 to page 8, line 37).

This document differs from the claimed subject-matter insofar as the disclosed compositions do not comprise a PAMS (see point 1.2 above).

- 1.3.3 Claim 1 according to the main request does not contain any limitation as to the type of PADS to be used and encompasses also the use of S,S-EDDS as the only PADS, which is, in fact, the subject-matter of dependent claim 10.

S,S-EDDS was already known to be a better chelating agent for heavy metals and to be more biodegradable than the racemate and, in particular, to have a high biodegradability above the benchmark of the patent in suit (see document (2), page 1, lines 8 to 11; document (1), page 8, lines 13 to 15 and the patent in suit,

page 2, lines 28 to 30 in combination with page 9, line 47).

The patent in suit does not contain any tests showing a synergistic activity of a combination of S,S-EDDS with EDMS but only tests relating to the use of the racemate EDDS which, as explained above, has by itself a lower biodegradability and a lower chelating activity than the S,S-EDDS. These tests relating to the use of a combination comprising a racemate EDDS cannot thus prove any synergistic activity of the already outstanding S,S-EDDS in combination with EDMS.

The Appellant provided with the statement of the grounds of appeal comparative tests carried out following the teaching of example 3 of the patent in suit. These tests show that the combination of S,S-EDDS with EDMS does not bring about any unexpected synergistic effect at least at low amounts of EDMS of up to 1 mole%.

Even though the Respondent contested the reliability of these tests, the Board, in the absence of any corroborating evidence, finds that the tests have been carried out correctly and that there is no reason for doubting the results obtained. Since the Appellant is the only party having brought evidence with regard to this specific embodiment of the claimed subject-matter, the burden of proof lied on the Respondent to show the contrary.

Since the combination of S,S-EDDS with EDMS does not bring about the alleged synergistic effect, the technical problem underlying the claimed invention can

only be defined in simpler terms as the provision of alternative compositions comprising S,S-EDDS and having similar chelating activity and biodegradability.

The Board is satisfied that the underlying technical problem has been solved by the claims of the patent in suit.

- 1.3.4 The Board notes that claim 1 according to the main request does not require the replacement of S,S-EDDS with other chelating agents but just the presence of 0.1 to 15% by weight of a mixture of chelating agents comprising PADS and PAMS. The claimed composition thus requires only that the total amount of chelating agents, including any PADS and PAMS be of 0.1 to 15% by weight. Moreover, the claimed compositions are not confined to compositions of a specific pH and encompass laundry detergent compositions having a neutral pH.

The question to be answered in order to evaluate the inventiveness of the claimed subject-matter is thus whether the skilled person, in the light of the teaching of the prior art and of his common general knowledge, would have envisaged the use of a PAMS, e.g. EDMS, in combination with S,S-EDDS.

EDMS was a well known biodegradable chelating agent for heavy metals (see document (12), page 3, lines 8 to 10 and document (15), column 1, lines 19 to 22 and column 2, lines 8 to 12). Therefore, in the Board's judgement, it was obvious for the skilled person to try it in the entire technical field wherein a sequestration of heavy metals is needed, thus also in a laundry detergent composition.

Moreover, the liquid laundry detergent compositions disclosed in document (1) had a pH of 6 to 10 and thus could be formulated at a neutral pH (see page 12, lines 26 to 28 and claim 11).

Since document (1) suggested the use of S,S-EDDS in combination with other sequestering agents, e.g. aminocarboxylic acids (see page 9, line 57 to page 10, line 1), it was obvious for the skilled person to try, for example, in the neutral liquid laundry detergent compositions of this document also at least small amounts of any known aminopolycarboxylic acid suitable as sequestering agent and thus also EDMS.

Therefore, the fact that EDMS had a known poor sequestering activity at alkaline pHs of at least 9, as shown in the patent in suit (page 8, table 2), would have not prevented the skilled person from trying EDMS in the neutral compositions of document (1).

It was thus obvious for the skilled person to try in a liquid neutral laundry detergent composition of document (1) at least small amounts of EDMS in combination with S,S-EDDS and to expect a similar biodegradability and chelating activity.

The Board concludes thus that the subject-matter of claim 1 according to the main request does not involve an inventive step.

2. *Respondent's auxiliary request*

Claim 1 according to the first auxiliary request differs from claim 1 according to the main request only insofar as it requires that **the molar ratio of the polyamino disuccinic acid to the polyamino monosuccinic acid is from 99:1 to 5:95.**

Since the alleged unexpected effect obtained by using a combination of EDDS and EDMS had not been shown for combinations comprising, for example, S,S-EDDS and EDMS as encompassed by claim 1 and the Appellant has convincingly shown that the combination of S,S-EDDS with EDMS does not bring about any unexpected synergistic effect at least at low amounts of EDMS of up to 1 mole%, the arguments put forward above with respect to the main request apply *mutatis mutandis* to claim 1 according to the auxiliary request.

The Board concludes thus that the subject-matter of claim 1 according to the auxiliary request does not involve an inventive step.

Order

For these reasons it is decided that:

The decision under appeal is set aside.

The patent is revoked.

The Registrar:

The Chairman:

G. Rauh

P. Krasa