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Bezeichnung der Erfindung: Bleaching compositions

Title of invention:

Titre de l'invention :

Klassifikation / Classification / Classement : C11D 3/39

ENTSCHEIDUNG / DECISION

vom / of / du 23 February 1989

Anmelder / Applicant / Demandeur : The Procter & Gamble Company

Patentinhaber / Proprietor of the patent /
Titulaire du brevet :

Einsprechender / Opponent / Opposant :

Stichwort / Headword / Référence :

EPÜ / EPC / CBE Articles 56, 111

Schlagwort / Keyword / Mot clé : "Inventive step - decision not supported by
the reasoning" - "amended claim having
formal deficiencies" - "remittal"

Leitsatz / Headnote / Sommaire

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Beschwerdekammern

Boards of Appeal

Chambres de recours

Case Number : T 298/87 - 3.3.2



D E C I S I O N
of the Technical Board of Appeal 3.3.2
of 23 February 1989

Appellant : THE PROCTER & GAMBLE COMPANY
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Decision under appeal : Decision of Examining Division 023 of the
European Patent Office dated 24 March 1987
refusing European patent application
No. 83 200 938.5 pursuant to Article 97(1) EPC

Composition of the Board :

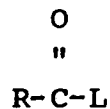
Chairman : P. Lançon
Members : S. Schödel
E. Persson

Summary of Facts and Submissions:

I. European patent application No. 83 200 938.5, published on 11 January 1984 under publication No. 98 021 was refused by a decision of the Examining Division dated 24 March 1987. The decision was based on Claims 1 to 10 as filed with the letter of 3 October 1985. Claim 1 after correction of three obvious errors was as follows:

"A laundry detergent composition for use in domestic automatic washing machines comprising:

- a) from 1% to 30% by weight of the composition of a surfactant system comprising a mixture of anionic, ethoxylated nonionic and optionally cationic surfactants;
- b) from 1% to 60% of a peroxygen bleaching compound capable of yielding hydrogen peroxide in an aqueous solution; and
- c) from 0.5% to 40% of a bleach activator having the general formula:



wherein R is an alkyl group containing from 5 to 18 carbon atoms wherein the longest linear carbon chain extending from and including the carbonyl carbon contains from 6 to 10 carbon atoms and L is a leaving group, the conjugate acid of which has a pK_a in the range of from 6 to 13; wherein the molar ratio of hydrogen peroxide yielded by (b) to bleach activator (c) is greater than 1.5."

II. According to the reasons for the decision, the subject-matter of Claim 1 did not involve an inventive step having regard to the prior art as represented by

- (A) GB-A-864 798 and
- (B) EP-A-43 173.

The technical problem underlying the application in suit was that of selecting particularly efficacious bleaching compositions from the broad scope of document (A).

The compositions of the illustrative examples 9 and 10 of that document differed from those of the application insofar as (i) an ethoxylated nonionic surfactant was not comprised in the surfactant system and (ii) the molar ratio of hydrogen peroxide yielded to bleach activator was about 1:1, i.e below 1.5.

The mere addition of an ethoxylated nonionic surfactant would have been an obvious measure in view of its well known reduced sudsing behaviour. As to the molar ratio of the components of the bleaching system document (A) did not teach that a variation of their molar proportions could affect the bleaching efficiency of the selected activators. Although there were no hints to use the indicated upper limits, the preferred range of 1/4 to 4 was to be taken as a direct indication that any molar ratio within this narrow range might be used. Thus the selected ratio for the bleaching composition was directly derivable from that prior art. A molar ratio of 4:1 would have been obvious taking into account also the possible activator decomposition during storage.

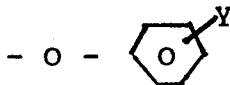
Example II of the application in suit, relating to the % conversion of bleach activator to percarboxylic acid at various molar ratios clearly showed that complete conversion was already achieved at molar ratios of the bleaching system of 1:1 with activators falling within the scope of document (A).

The bleach effect on dingy soils of the activators having a longer linear carbon chain had in any case been recognised in document (B) which disclosed bleach activators within Claim 1 of both the application in suit and document (A).

III. A Notice of Appeal was lodged by the Appellant on 1 June 1987 with payment of the fee on the same day. A Statement of Grounds was filed on 24 July 1987 together with a new set of Claims 1 to 6.

Claim 1 of the new set differs from the aforementioned version in that the leaving group L in the general formula under component (c) is further defined, following the phrase "... the range of from 6 to 13..." by the wording:

"..., the leaving group L having the formula



wherein Y is $-\text{SO}_3^- \text{M}^+$ or $-\text{COO}^- \text{M}^+$ and M is a cation which provides solubility to the bleach activator, preferably sodium, potassium or mixtures thereof;..."

IV. The Appellant has argued substantially as follows:

- (i) document (A) was only concerned with the problem of the storage stability of the compositions and not with their efficacy; the claimed invention provided a solution to the problem of enhancing the yield of the desired peroxycarboxylic acids by a surface bleaching mechanism;
- (ii) there was no recognition in document (A) of any performance difference between different members of the disclosed class of activators nor of a

requirement for a high hydrogen peroxide to activator molar ratio for certain activator structures; document (A) in any case did not recognise the concept of dingy soil clean up;

(iii) the argument of "complete conversion" of certain members of the claimed activators already at molar ratios of 1:1, corresponding to the molar ratios exemplified in document (A), was inaccurate in view of the values actually quoted in the table on page 24 of the application in suit; as the incorporation of a nonionic surfactant did not enhance the bleaching efficacy, the demand for a comparison between the compositions of document (A) and similar compositions incorporating a nonionic surfactant was unjustified;

(iv) document (B), although referring to dingy soil clean up, dealt with a different kind of activator which was unsuitable for use with conventional anionic surfactants; it also failed to recognise a requirement for high molar ratios of hydrogen peroxide to activator; equimolar ratios were preferred;

V. The Appellant requests that the decision of the Examining Division be set aside and that a patent be granted on the basis of the claims submitted with the Statement of Grounds on 24 July 1987.

Reasons for the Decision

1. The appeal complies with Articles 106 to 108 and Rule 64 EPC and is, therefore, admissible.

2. The application in suit relates to detergent bleach compositions that provide removal of stains and/or dingy soils from textiles by a peroxygen bleaching mechanism which takes place on the textile surface. In order to obtain a desirable level of bleaching performance at bleach solution temperatures below 60°C, when peroxygen bleaches normally become only partially effective or even ineffective, substances known as bleach activators are added, which render peroxygen bleaches more effective at lower temperatures.
3. Claims 1 to 10 filed on 10 October 1985 were refused by the Examining Division on the sole ground of lack of inventive step (Article 56 EPC). In arriving at this conclusion, the Examining Division used as its starting point document (A), which it considered to be the closest state of the art. The Board will also consider the matter from this point of view.
4. Document (A) discloses detergent bleaching compositions which contain:

10 to 50% (by weight) of organic detergent; for instance an alkarylsulphonate or an alkylsulphate;
from 1 to 15% of persalt, and
from 2 to 30% of a bleach activator which is a solid reactive organic ester,
the remainder being detergent adjuncts (cf. page 3, paragraph 2).

These compositions have strong bleaching properties at relatively low temperatures and are suitable for removing stains from textile materials (cf. page 1, line 10 and page 2, line 36). The bleach activator ester is derived preferably from an aliphatic carboxylic acid having not more than 10 carbon atoms and a phenolsulphonic or a

hydroxybenzoic acid respectively (cf. Claim 8; page 2, paragraph 3). The free-flowing solid compositions contain the bleach activator as particles separate from the persalt, the size of the ester particles being such that at least 70% of the ester is retained on a 60 mesh British Standard sieve (cf. Claim 1). The molar ratio of the reactive ester to each atom of available oxygen is preferably from 1:4 to 4:1 (cf. page 2, lines 100-104).

The bleach activators of the examples have the formula $p\text{-RO-C}_6\text{H}_4\text{-SO}_3\text{Na}$, R being acetyl (CH_3CO), butyryl ($\text{C}_3\text{H}_7\text{CO}$), caproyl ($\text{C}_5\text{H}_{11}\text{CO}$) or caprylyl ($\text{C}_7\text{H}_{15}\text{CO}$), or $p\text{-CH}_3\text{COOC}_6\text{H}_4\text{COOH}$ (Examples 1-7; 8; 9; 10; 11). In the examples, the aforesaid molar ratio is about 1:1.

The tendency of the ester and persalt to decompose during storage in the presence of one another is reduced by using the ester in granular form (cf. page 2, lines 2-16).

5. The technical problem arising in respect of this state of the art was to provide a composition of enhanced bleaching efficiency.
- 5.1 The solution to this technical problem as set out in refused Claim 1 was a detergent composition comprising, briefly:
 - a) a particular surfactant system, i.e. a mixture of anionic, ethoxylated nonionic and optionally cationic surfactants;
 - b) a peroxy compound, and
 - c) a bleach activator of the formula RC(O)L , with R being an alkyl group wherein the linear carbon chain (including the carbonyl carbon) contains from 6 to 10 carbon atoms and L being a leaving group having a conjugate acid pK_a from 6 to 13,

- d) the molar ratio of hydrogen peroxide yielded by b) to bleach activator c) being greater than 1.5 ("molar ratio" hereinafter meaning molar ratio H_2O_2 /activator).
6. In addressing the issue of the decision under appeal, it has to be considered, whether the subject-matter claimed was obvious in the light of the cited prior art.
7. Starting out from document (A), the following inferences can be drawn by the skilled person:
- (i) all the activators, whether they are derived from an aliphatic carboxylic acid with a long or a short carbon chain, are equally efficient as bleaching components; sodium p-acetoxybenzenesulphonate is preferred for economic reasons, although it is more unstable;
 - (ii) a "molar ratio" of 1:1 is optimal as illustrated in the examples;
 - (iii) the prime consideration in formulating the detergent compositions is that of storage stability by using activator granules of controlled particle size.
8. Document (B) relates to detergent products. The bleaching compositions therein comprise certain long chain acyl group-containing compounds which are said to promote or to augment the bleaching power of common peroxygen compounds thus providing highly effective, colour-safe bleaches especially suitable for use at relatively low laundering solution temperatures. Combined with detergents, the bleaching compositions are capable of concurrently delivering fabric bleaching and laundering benefits.
- 8.1 Such detergent formulations may comprise from:
1 to 50% by weight of a solubilizing surfactant and

1 to 60% by weight of a bleaching composition that is a hydrogen peroxide-yielding peroxygen compound and a bleach activator compound (Claim 10).

Detergent surfactants suitable for the above-mentioned purposes include in principle conventional nonionics (especially polyethylene oxide condensates of alkylphenols or aliphatic alcohols), ampholytic, zwitterionic and cationic surfactants and also - contrary to the Appellant's allegation - conventional anionic surfactants (page 12, para. 3, Claim 6; page 15, para. 3).

From the bleach activator compounds disclosed the preferred ones have the formula $RC(O)Z$, wherein R is a hydrocarbyl-lipophilic group containing from 5 to 13 carbon atoms (and additionally containing at least 2 oxyethylene groups if R contains more than 9 carbon atoms) and Z is a leaving group, having a pKa of from 5 to 20 and a molecular weight of less than 175, selected from certain enols, carbon acids and imidazoles. The more interesting activator compounds are those wherein R in the above formula includes no more than 9 carbon atoms and wherein Z is selected from the enols and carbon acids.

- 8.2 Activator compounds of the said structure have a sufficiently rapid rate of perhydrolysis to deliver the desired degree of peroxygen bleach activation, i.e. they are able quickly to react with the hydrogen peroxide in the laundering solution to form peroxyacids which, presumably in the form of a highly reactive oxygen - yielding radical are the species which provide enhanced bleaching activity relative to the unactivated peroxygen bleach. This rate determines the degree of bleach activation and this has been found to be highly dependent on the identity of the bleach activator compound.

In addition the relatively long chain substituent R is believed to enhance the fabric substantivity so that the bleaching species have sufficient surface activity to concentrate at the fabric surfaces where they are most effective (cf. page 10).

- 8.3 The bleaching compositions preferably contain the peroxygen compound and the bleach activator compound in approximately equimolar ratios (cf. page 11, para. 2).

The preferred bleach activator compounds are isopropenyl hexanoate $C_5H_{11}COOC(:CH_2)-CH_3$ and hexanoyl malonic acid diethylester, $C_5H_{11}COCH(COOC_2H_5)_2$, the former being most preferred. In the working examples qualitatively excellent bleaching results are secured also on dingy soils.

- 8.4 The Examining Division when referring to document (B) did not identify any indication, whether in the general description or in the examples, that a variation of the molar ratio in the bleaching compositions towards higher H_2O_2 values would result in an improved bleaching performance. Nor did they look into the various types of bleach activators disclosed therein, especially those relevant to the application in suit having a conjugate acid pKa of 6-13 (which means a relatively high degree of acid character).

Thus neither of the documents considered, taken alone or in combination, has been shown to lead in an obvious manner to the subject-matter claimed especially as regards the "molar ratio" greater than 1.5 in association with the selected bleach activators.

- 8.5 As to the interpretation of the experimental data in the application in suit, the examples adequately demonstrate

the significance of the critical parameters in relation to the technical problem as stated.

Example IV, for instance, shows that the dingy soil clean up is improved by using a claimed detergent-activator system once the "molar ratio" is raised into the claimed range above 1.5. This focusses on the effect of changing the crucial parameter "molar ratio" whilst keeping the other parameters constant. In the light of this there was no need to alter further parameters, such as the detergent system. Indeed the Examining Division itself considered the addition of a nonionic surfactant to be trivial and not to affect the bleaching efficiency (cf. decision, page 6, first two paragraphs).

Example II illustrates the additional percarboxylic acid formation (percent conversion, corresponding to enhanced bleaching efficiency) at a "molar ratio" higher than 1:1 for bleach activators falling within refused Claim 1 compared with those falling outside (cf. Table on page 24). The argument of "complete conversion" already at a "molar ratio" of 1:1 for claimed bleach activators is in contradiction with the figures given in that Table. The view taken by the Examining Division in this respect was therefore unjustified.

The Examining Division also adopted the position that the claimed "molar ratio" was directly derivable from document (A) having regard to the disclosure therein of the preferred "molar ratio" of from 4:1 to 1:4. This range is, however, disclosed generally in respect of all the bleach activators covered by that document and not specifically in relation to particular ones. Indeed, the skilled person could have hit upon the claimed "molar ratio" and the selected bleach activators simultaneously by chance or by extensive experimentation. Nevertheless, there was no

incentive to do so and in the absence of any expectation of success one cannot assume that the practitioner would have made an effort in this area (cf. T 2/83 - OJ EPO 1984, 265).

Apart from this, the implication that losses of bleach activator through decomposition should be compensated by applying more, must be understood as pointing to a "molar ratio" below 1:1 and this leads rather away from what is claimed (cf. page 1, last paragraph page 2, first paragraph).

- 8.6 Accordingly the reasoning given in the decision under appeal does not support the finding under Article 56 EPC which led to refusal of the application. The decision under appeal must therefore be set aside.
9. Nevertheless, the application is to be prosecuted further on the basis of Claims 1 to 6 filed on 24 July 1987. Whilst it is appreciated that the amendments bring a further distinction over the leaving groups of document (B), and that the reasons given in the decision under appeal cannot be more effective against a narrower claim, no final position can be taken on the matter for the following reasons.
- 9.1 Claim 1 of this new set differs from Claim 1 as refused in that the bleach activator c) is more precisely defined by adding a limiting feature to the leaving group L (cf. Section III). This insertion finds support on page 10, paragraph 2 of the application as originally filed. It is therefore acceptable under Article 123(2) EPC. This is also true for the subsequent Claims 2 to 6 which correspond to original Claims 2, 3, 8, 9 and 11.

9.2 Feature a) of Claim 1, however, which relates to the particular surfactant system set out under Section 5.1 a) does not seem, on closer review, to meet the formal requirements of in Article 123(2) EPC. It was introduced during the examination procedure to remove an objection of lack of novelty and was tacitly accepted by the Examining Division.

As to the wording objected to, the Board is unable to trace any explicit disclosure at the places indicated by the Appellant in the originally filed documents. It is true that there is a list of surface active agents and mixtures thereof on page 13, third paragraph. However, no preference for any particular surface active agent or mixture of such agents appears to be expressed, let alone e.g. a mixture of anionic, ethoxylated nonionic and optionally cationic surfactants. In the relevant Examples nothing but certain detergent compositions containing some specific anionics, namely alkyl sulphates and alkyl benzene sulphonates, and specific nonionics, namely alkylpolyethoxylates (cf. Example III A) and specific cationics, namely alkyl trimethyl ammonium chloride (cf. Examples I, III B, VI) are mentioned. The general terms under consideration do not clearly arise directly from this information.

It therefore seems that feature a) has no sound basis in the original documents.

Apart from this, with features b) and c) the reference to the weight % is missing (Art. 84 EPC).

However, the amendments requested under Rule 88 EPC are clearly allowable as already indicated by the Examining Division (cf. Decision, paragraph 9).

10. It seems that a full examination of the matter of the amended claims is required, especially as regards Article 123 EPC, on which the decision under appeal is completely silent.

In these circumstances the Board deems it appropriate, making use of its powers under Article 111 EPC, to remit the case to the first instance, for further investigation.

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 for further prosecution.

The Registrar:

The Chairman:

F.Klein

P.Lançon