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D E C I S I O N
of 21 June 2002

Case Number: T 0479/98 - 3.3.6

Application Number: 88201175.2

Publication Number: 0294904

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Language of the proceedings: EN

Title of invention:

Process for making an aqueous liquid detergent composition
containing a perborate bleach

Patentee:

THE PROCTER & GAMBLE COMPANY, et al

Opponent:

UNILEVER N.V. / UNILEVER PLC

Headword:

Stirring/PROCTER & GAMBLE

Relevant legal provisions:

EPC Art. 84, 54, 56

Keyword:

"Novelty (yes)"
"Inventive step (yes) - alternative not suggested in the prior
art document"

Decisions cited:

-

Catchword:

-



Case Number: T 0479/98 - 3.3.6

D E C I S I O N
of the Technical Board of Appeal 3.3.6
of 21 June 2002

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Decision under appeal: Interlocutory decision of the Opposition Division
of the European Patent Office posted 4 March 1998
concerning maintenance of European patent
No. 0 294 904 in amended form.

Composition of the Board:

Chairman: P. Krasa
Members: G. N. C. Rath

Summary of Facts and Submissions

I. This appeal is against the interlocutory decision of the Opposition Division to maintain in amended form European patent 0 294 904 relating to a liquid detergent containing perborate bleach.

II. The patent as amended contained a set of 11 claims, of which the independent Claims 1, 3 and 6 read as follows:

"1. A process for making an aqueous liquid detergent composition having a pH of at least 8, comprising at least 5% of an organic, non-soap anionic surfactant, at least 5% of a builder and from 1%-40% of a solid perborate bleach, wherein sodium perborate tetrahydrate or monohydrate is added to an aqueous liquid comprising the anionic surfactant and the builder, wherein the resulting slurry is stirred and wherein the perborate particles having a weight average particle diameter of from 0.5 to 20 micrometers are formed by in situ crystallization of the perborate.

3. A process for making an aqueous liquid detergent composition having a pH of at least 8, comprising at least 5% of an organic, non-soap anionic surfactant, at least 5% of a builder and from 1%-40% of a solid perborate bleach, wherein sodium metaborate is added to an aqueous liquid comprising the anionic surfactant and the builder, wherein a stoichiometric amount of peroxide is added, while stirring until completion of the reaction and wherein the perborate particles having a weight average particle diameter of from 0.5 to 20 micrometers are formed by in situ crystallization of the perborate.

6. A process for making an aqueous liquid detergent composition having a pH of at least 8, comprising at least 5% of an organic, non-soap anionic surfactant, at least 5% of a builder and from 1%-40% of a solid perborate bleach, wherein boric acid is added to an aqueous liquid comprising the anionic surfactant and the builder, wherein a stoichiometric amount of hydrogen or sodium peroxide is added, while stirring until completion of the reaction and wherein the perborate particles having a weight average particle diameter of from 0.5 to 20 micrometers are formed by in situ crystallization of the perborate."

The dependent Claims can be summarized as follows:

Dependent Claim 2 specified the perborate. Dependent Claims 4 and 5 specified the peroxide and the formation of sodium metaborate, respectively. Dependent Claims 7 and 8 specified the aqueous liquid, dependent Claim 9 the builder, and dependent Claims 10 and 11 a preferred diameter of the perborate particles.

III. The notice of opposition based on lack of novelty and inventive step (Articles 100(a), 54, 56 EPC) cited, inter alia, the following document:

(6) GB-A-943 271.

IV. In its decision the Opposition Division held that the subject-matter as defined in the set of 11 Claims submitted as the "main auxiliary request" by the respondents (proprietors) during oral proceedings before the Opposition Division complied with the relevant requirements of the EPC, namely, that it was novel and involved an inventive step over the cited

prior art, in particular over document (6).

V. The appellant (opponent 01) lodged an appeal against this decision; its arguments are summarized as follows:

- monohydrate perborate was available at the priority date of the patent in suit and was part of the disclosure of document (6) mentioning sodium perborate;
- the homogenisation step disclosed by document (6) comprised "stirring"; so, Example 1 of document (6) was novelty destroying with respect to claim 1;
- the problem of the patent in suit was similar to the problem stated in document (6); the goal of document (6) was to obtain an improved bleaching activity and stability of concentrated liquid bleaching and detergent compositions;
- even if in situ crystallization was not mentioned in document (6), this process step implicitly occurred in the preparation of Example 1 of document (6); therefore the whole process was obvious.

VI. The respondents refuted the arguments of the appellant as follows:

- the monohydrate was not commercially available in 1961, the date on which document (6) was filed;
- the monohydrate form of perborate was more expensive than the tetrahydrated form; monohydrate

would immediately transform into tetrahydrate perborate in a liquid matrix;

- for reasons of stability, the objective of the patent in suit was to formulate an aqueous liquid detergent composition having suspended therein perborate bleach articles of a weight average diameter of 0.5 to 20 μm (page 2, lines 46 to 50), a feature not highlighted by document (6);
- according to document (6), the composition was not submitted to a crystallization process but to an oxidation process (page 2, lines 103 to 106); in Example 1, acetic anhydride was added (page 3, lines 37 to 39) to promote the oxidising action of the hydrogen peroxide released by the persalt (see page 2, lines 106 to 111).

VII. The appellant requested that the decision under appeal be set aside and the patent be revoked.

The respondents requested that the appeal be dismissed.

VIII. Oral proceedings took place on 21 June 2002.

As announced in their letter of 7 November 2001, the respondents did not attend the oral proceedings.

IX. The other party (opponent 02) took no part in the appeal proceedings.

Reasons for the Decision

1. *Articles 84 and 123 EPC*

1.1 With respect to the subject-matter of Claim 1, the Board is satisfied that the requirements of Articles 84 and 123 EPC are met. Since no objections have been raised in this respect, no further reasons need to be given.

1.2 However, since the appellant based its reasoning on the allegation that the "stirring" feature of the claimed process has to be understood as "homogenisation", a term used in Example 1 of document (6) (page 3, line 42; see appellant's letter of 3 May 1998, page 2, lines 30 and 31), the Board finds it appropriate, in this case, to establish the technical meaning of this feature in the light of the patent in suit.

Stirring is a mechanical process and suggests, in a very general manner, a movement or an agitation of a predominantly liquid medium aimed at a uniform distribution of components, temperature etc. in the said medium. The equalization of concentration and temperature in the medium in question is the essential task of a stirring process which implies the use of a stirring device and can be called homogenisation.

However, depending on the particular context, the term "homogenisation" may connote not just a process of rendering the relevant medium uniform as mentioned above, but also a process the objective of which is a reduction in the size of the particles of the dispersed system.

While stirring, as already said, calls for the use of a stirring device, a skilled person would not, in the Board's judgement, have considered a size-reducing device such as a colloid mill as an apparatus to be used in a stirring process such as that of Claim 1 and especially not when the patent in suit taught to avoid grinding (page 2, lines 51 and 53), even if one accepts that, as it certainly does, a colloid mill generates movement and a mixing effect as does any stirring device.

Example 1 of document (6) discloses a process for the preparation of a liquid detergent composition containing "sodium perborate" as bleaching agent which was readily pourable and showed no separation into layers or sedimentation after storage for 7 weeks (page 3, lines 1 to 48, in particular line 14 and lines 43 to 46).

For this process, the use of a colloid mill is mandatory. The relevant passages in document (6) read:

"The method of preparation adopted was as follows: half the condensed phosphate was added over a periode of some minutes to water in a collod mill. A small amount of detergent B was added followed by..."

"The pH was adjusted to about 8.5 by means of sulfuric acid. Detergents A and B were successively added to the suspension..."

and

"...Finally the optical brightener and the stabiliser for the per-salt were added and the product was

homogenised for 15 minutes."

(page 3, lines 19 to 23, lines 27 to 29, and lines 39 to 46).

It is clear to the skilled person that "homogenised for 15 minutes" relates to a process involving size reduction, since otherwise the use of a colloid mill would not make any sense.

2. Novelty

2.1 Claim 1

Claim 1 relates to a process for making an aqueous liquid detergent composition comprising, inter alia, from 1% to 40% of a solid perborate bleach, wherein sodium perborate tetrahydrate or monohydrate was added and wherein the resulting slurry was stirred and wherein the perborate particles having a weight average particle diameter of from 0.5 to 20 micrometers were formed by in situ crystallization of the perborate.

Since the term "sodium perborate", as used in Example 1 of document (6), does not relate to a specific chemical entity but encompasses several perborate forms, it is not clear which "sodium perborate" was actually meant. In particular, document (6) does not disclose directly and unambiguously which of the two forms specified in Claim 1 of the patent in suit, namely the monohydrate and the tetrahydrate form, were used as the "sodium perborate" in its Example 1. Apart from these two forms, there existed also e.g. the trihydrate form. Whether or not one of these "sodium perborates" were commercially available at the publication date of

document (6) is of no importance in this context. It should be mentioned, however, that the trihydrate form is thermodynamically more stable than the tetrahydrate and cakes less in detergent formulations than the tetrahydrate (see e.g. Encyclopaedia of Chemical Technology, vol.17, 3rd edn., John Wiley & Sons, 1982, page 8, lines 5 to 6 and 16 to 18). These considerations certainly make the trihydrate form a possible candidate for the "sodium perborate" of Example 1 of document (6). Since document (6) disclosed neither sodium perborate tetrahydrate nor sodium perborate monohydrate as the bleaching component to be used in the process of Claim 1 of the patent in suit, that process is, if only for this reason, novel.

Moreover, the process disclosed in Example 1 of document (6) requires "homogenisation for 15 minutes in a colloid mill" which is a process not covered by Claim 1 of the patent in suit (see above point 1.2). Therefore, the Board concurs with the Opposition Division that document (6) did not anticipate the subject-matter of Claim 1. Hence, the requirements of Article 54 EPC are met.

2.2 Claim 3

Claim 3 differs from Claim 1 essentially in that sodium metaborate and a stoichiometric amount of peroxide were added to the detergent composition, two features not disclosed by document (6). Apart from this difference, neither the stirring nor the consequent crystallisation step were anticipated by document (6).

Hence, the requirements of Article 54 EPC are met.

2.3 Claim 6

Claim 6 differs from Claim 1 essentially in that boric acid and a stoichiometric amount of hydrogen or sodium peroxide were added to the detergent composition, two features not disclosed by document (6). Apart from this difference, the stirring and, hence, the crystallisation step were also not anticipated by document (6).

Hence, the requirements of Article 54 EPC are met.

3. *Inventive step*

3.1 Claim 1 relates to a process for manufacturing an aqueous liquid detergent composition comprising, inter alia, from 1% to 40% of a solid perborate bleach, wherein sodium perborate tetrahydrate or monohydrate was added and wherein the resulting slurry was stirred and wherein the perborate particles having a weight average particle diameter of from 0.5 to 20 micrometers were formed by in situ crystallization of the perborate.

A similar process was disclosed by document (6) - see point 1.2, above.

3.2 The patent in suit addressed the problem of formulating an aqueous detergent composition having suspended therein small particles of a perborate bleach. For reasons of physical stability the objective was to obtain perborate particles having a weight average particle diameter of from 0.5 to 20 micrometers (page 2, lines 47 to 50).

The objective of document (6) was to find concentrated liquid bleaching and detergent compositions with improved bleaching activity and stability (page 1, lines 44 to 46); the compositions should not contain ingredients which destroy the stability of the suspension (page 2, lines 83 to 85).

3.3 The Opposition Division and the appellant took document (6) as the starting point for evaluating inventive step. The Board sees no reason to disagree since stability was an objective of both document (6) and the patent in suit.

3.4 Hence, the problem underlying the patent in suit was to find an alternative process for making an aqueous detergent composition.

In view of the examples of the patent in suit, the technical problem as defined above was credibly solved. This was not disputed.

3.5 The question remains whether or not the claimed solution of the existing technical problem involves an inventive step.

3.6 One difference between document (6) and the patent in suit lies in the stirring step (see point 1.2) and, related thereto, the in situ crystallization of the perborate having a weight average diameter of 0.5 to 20 μm .

3.7 The appellant argued that the same components were present in both the composition according to document (6) and the composition according to the patent in suit, so that the reaction must have been the

same. Homogenisation (according to document (6), page 3, line 42) necessarily comprised a stirring process.

The Board cannot agree. Whereas according to document (6) the desired size of particles was obtained with a colloid mill by mechanical size reduction, in the patent in suit it was obtained through crystallization or recrystallization by stirring in the presence of anionic surfactants (see point 1.2).

According to the patent in suit the slurry was moved in such a way that specific crystallization conditions were to be respected, namely, those which allowed for obtaining perborate particles having a specific diameter. It would not have been obvious to the skilled person, looking for stable products, that he could do without the colloid mill which was according to Example 1 of document (6), mandatory to obtain the desired stability (see point 1.2, paragraph 6). According to that example homogenisation with a colloid mill was a compulsory process step; there is no evidence on file that the mere stirring of the surfactants would be sufficient to obtain stable products. It may be that in the process of document (6) a certain recrystallisation process, being in equilibrium with a dissolution process, took place simultaneously with the homogenisation step. However, it was not known that it was sufficient to adjust the stirring conditions so that solid perborate particles having a weight average diameter of from 0.5 to 20 μm crystallized from the slurry. The stirring of the slurry in order to obtain the desired particle size via crystallization was not obvious, since it was not suggested in document (6) and could not be deduced from

any other document.

- 3.8 The same reasoning as set out under points 3.1 to 3.7 applies mutatis mutandis to Claims 3 and 6 which both comprise the stirring and crystallization features.
- 3.9 The subject-matter of Claims 1, 3 and 6 involves an inventive step, and therefore meets the requirement of Article 56 EPC.

The dependent Claims 2, 4, 5 and 7 to 10 derive their patentability from the respective independent Claims.

Order

For these reasons it is decided that:

The appeal is dismissed

The Registrar:

The Chairman:

G. Rauh

P. Krasa