Publication in the Official Journal ~~Yes~~ / No

File Number: T 33/88 - 3.3.1

Application No.: 82 300 309.0

Publication No.: 0 057 088

Title of invention: Detergent compositions

Classification: C11D 3/06

D E C I S I O N
of 16 January 1992

Proprietor of the patent: The Procter & Gamble Company

Opponent: Unilever N.V.

Headword: Porphine-speckles/PROCTER

EPC Article 54, 56

Keyword: "Novelty (yes, after amendment)" - "Inventive step (yes, after amendment)"

Headnote

Case Number : T 33/88 - 3.3.1

D E C I S I O N
of the Technical Board of Appeal 3.3.1
of 16 January 1992

Appellant : (Proprietor of the patent)
The Procter & Gamble Company
301 East Sixth Street
Cincinnati
Ohio 45202 (US)

Representative :
Brooks, Maxim Courtney
Procter & Gamble (NTC) Limited
Whitley Road
Longbenton
Newcastle-upon-Tyne NE12 9TS (GB)

Respondent : (Opponent)
Unilever N.V.
Burgemeester's Jacobplein 1
NL-3015 CA Rotterdam (NL)

Representative :
Tan, Bian An, Ir.
Unilever N.V.
Patent Division
P.O. Box 137
NL-3130 AC Vlaardingen (NL)

Decision under appeal :
Decision of Opposition Division of the European Patent Office of 13.10.87, posted on 17.11.87, revoking European patent No. 0 057 008 pursuant to Article 102(1) EPC.

Composition of the Board :

Chairman : K.J.A. Jahn
Members : P.K.J. Krasa
J.C. Saisset

Summary of Facts and Submissions

I. The mention of the grant of patent No. 0 057 088 in respect of European patent application No. 82 300 309.0 filed on 21 January 1982 was published on 21 August 1985 (c.f. Bulletin 85/34) on the basis of twelve claims.

II. A notice of opposition was filed in due time by the Respondent (Opponent) requesting the revocation of the European patent on the grounds of lack of novelty and inventive step (Article 100(a) EPC) and, furthermore, on the ground of insufficient disclosure (Article 100(b) EPC). The opposition was based, inter alia, on the following document:

(2) DE-A-2 733 935.

III. Claim 1 of the disputed patent was amended in the course of the opposition proceedings. By a decision delivered orally on 13 October 1987, with written reasons posted on 17 November 1987, the Opposition Division revoked the patent for lack of novelty of the said amended Claim 1 in view of document (2).

IV. An appeal was lodged against this decision on 18 January 1988 and a statement of grounds of appeal was filed on 22 March 1988. During the oral proceedings, which took place on 16 January 1992, the Appellant (Patentee) submitted a first set of five claims together with an amended description (main request) and a second set of five claims (auxiliary request). Claim 1 of the main request reads:

"A granular detergent composition comprising (all percentages being by weight of the granular detergent composition):

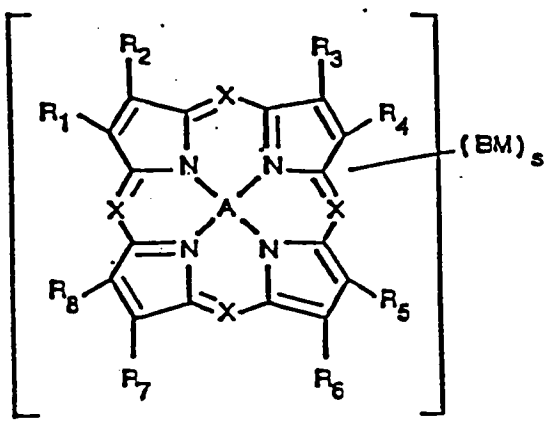
a) from 40% to 87.9% of spray-dried base powder comprising

i) from 1% to 20% of organic surfactant selected from anionic, zwitterionic and ampholytic surfactants and mixtures thereof,

ii) from 5% to 81.9% of detergency builder, and

iii) from 5% to 18% moisture,

b) from 0.1% to 20% of a detergent additive composition in particulate form comprising a storage-sensitive detergent additive material, the storage-sensitive detergent additive material being a multifunctional photoactivator/dye which is a porphine having the general formula II:



II

wherein each X is (=N-) or (=CY-), and the total number of (=N-) groups is 0,1,2,3 or 4; wherein each Y, independently, is hydrogen or meso substituted alkyl, cycloalkyl, aralkyl, aryl, alkaryl or heteroaryl; wherein each R, independently, is hydrogen or pyrrole substituted alkyl, cycloalkyl, aralkyl, aryl, alkaryl or heteroaryl, or wherein adjacent pairs of R's are joined together with ortho-arylene groups to form pyrrole substituted alicyclic or heterocyclic rings; wherein A is 2(H) atoms bonded to diagonally opposite nitrogen atoms, or Zn(II), Cd(II), Mg(II), Ca(II), Al (III), Sc(III), or Sn(IV);

wherein B is an anionic, nonionic or cationic solubilizing group substituted into Y or R; wherein M is a counterion to the solubilizing groups; and wherein s is the number of solubilizing groups, wherein when B is cationic, M is an anion and s is from 1 to 8; when B is nonionic, B is polyethoxylate, M is absent, s is from 1 to 8, and the number of condensed ethylene oxide molecules per porphine molecule is from 8 to 50; when B is anionic and attached to a carbon atom displaced 5 or less atoms away from the porphine core, M is cationic and s is from 3 to 8; when B is anionic and attached to carbon atom displaced more than 5 atoms away from the porphine core, M is cationic and s is from 2 to 8; and when B is sulphonate the number of sulphonate groups is no greater than the number of aromatic and heterocyclic substituent groups, and

c) from 2% to 25% of ethoxylated nonionic surfactant in intimate mixture with spray-dried base powder and detergent additive composition,

characterised in that the detergent additive composition comprises an intimate mixture of the photoactivator/dye with a hydratable water-soluble crystalline salt, the intimate mixture being releasably enclosed within a substantially continuous glassy matrix of amorphous phosphate material having the general formula I:

$$(M_2O)_x(P_2O_5)_y \qquad I$$

wherein M is hydrogen, alkali metal, ammonium or a substituted ammonium groups, y has a value in the range of 2 to 50, and the ratio x:y is from 0.7:1 to 1.7:1."

The claims according to the auxiliary request relate to a method of manufacturing such granular detergent compositions.

V. The Appellant submitted that the subject-matter of the new claims was novel and inventive as none of the citations taught or made obvious the technical problem to which the invention relates, i.e. to prevent "bleeding" of porphine photoactivator speckles in detergent compositions comprising nonionic surfactants, let alone the solution of this problem. The function of the amorphous polyphosphate used in (2) was said to differ from that according to the patent in suit especially insofar as it acts as a sequestering and, thus, a chemically stabilising agent for the percarbonate particles of document (2) in contrast to forming a continuous physical barrier enrobing the photoactivator according to the disputed patent.

Furthermore, the Appellant stressed the function of the hydratable water-soluble crystalline salt as a promoter for the formation of the glassy enrobing matrix. This function was not disclosed in any of the citations.

VI. While conceding novelty for the subject-matter of the new claims, the Respondent (Opponent) contested inventive step:

To replace the storage-sensitive sodium percarbonate of (2) by another storage-sensitive agent, i.e. the porphine photoactivator/dye, was deemed to be neither surprising nor inventive and, relying on two fresh documents

(4) GB-A-1 031 645 and
(5) GB-A-811 658,

he further argued that it was common practice to co-granulate adjuncts in intimate admixture with hydratable water-soluble crystalline salts.

The Respondent further submitted that the process of document (2) was, apart from the ingredients to be stabilised, substantially the same as the one of the disputed patent and he pointed out that both processes made use of the same amorphous phosphate, i.e. sodium hexametaphosphate, which was used in both processes in amounts of the same order of magnitude. Therefore, he concluded that the respective products obtained must also be basically the same.

In respect to the homogeneity of the particles aimed at in document (2), the Respondent commented that granules according to the disputed patent would unavoidably be infiltrated by the aqueous solution of amorphous polyphosphate and, thus, would be pervaded by the glassy polyphosphate. The Respondent deduced therefrom that the degree of homogeneity of a product according to the patent in suit was about the same as that of a product according to citation (2), taking into account that the latter was also not really a homogeneous product in view of the use of seed crystals in its manufacture.

VII. The Appellant requested that the impugned decision be set aside and that the patent be maintained in amended form either on the basis of the main request or of the auxiliary request. The Respondent requested that the appeal be dismissed. At the end of the oral proceedings the Chairman announced the decision of the Board to allow the appeal.

Reasons for the Decision

1. The appeal is admissible.

2. Amendments

Claim 1 of the main request is in fact a combination of Claims 1, 3, 7, and 12 as granted. Dependent Claims 2 to 5 correspond to Claims 4, 5, 6, and 8 as granted. The claims of the auxiliary request differ from those of the main request basically only in that they are drafted as process claims. They are supported especially by Claims 9 and 10 as granted in combination with page 4, lines 35 to 38 of the patent specification. The claims as granted were in substance those claims as originally filed. Thus, there are no objections under Article 123 EPC to any of the claims in accordance with Appellant's two requests.

During the oral proceedings the Respondent for the first time objected to the definition of M in formula I of Claim 1. He alleged that, because in this formula M was, inter alia, hydrogen, polyphosphoric acids which would not form the desired glassy matrix, were also covered. The Board cannot share this opinion. It was admitted by the Respondent that the expression " amorphous phosphate material" unambiguously designates a salt. Thus, in the Board's judgement, the skilled person would understand immediately that the said phosphate material has to be a salt with the possibility that some hydrogen ions are present.

3. Novelty

After examination of the cited prior art, the Board has reached the conclusion that the claimed subject-matter is novel. Since novelty of the present claims was conceded by the Respondent, it is not necessary to give detailed reasons for this finding.

4. Inventive step

4.1 The patent in suit relates to granular detergent compositions comprising a photoactivator/dye of formula II as a detergent additive composition in particulate form (page 2, lines 3 to 5 and Claim 1). These photoactivators can provide fabric bleaching effects in built detergent compositions in the presence of visible light and atmospheric oxygen and can also synergistically enhance the bleaching effect of conventional bleaching agents such as sodium perborate (cf. the sentence bridging pages 3 and 4 of the patent in suit). Such detergent compositions comprising a water-soluble dye in the form of speckles are uncontestedly state of the art. However, they can lose their aesthetic appeal on storage because the dye may migrate into the detergent base powder in the presence of a nonionic surfactant (page 2, lines 11 to 15).

Thus, the technical problem to be solved according to the patent in suit was the avoidance of this "bleeding" effect (page 3, lines 35 to 37).

Taking into account that no prior art document was cited which would require a modification of this technical problem, the Board accepts it as the basis for investigating inventiveness. It is also noted that the existence of this problem was not seriously contested by the Respondent. It was only during the oral proceedings that the Respondent questioned whether the above technical problem would also arise with low amounts of nonionic surfactant. In the absence of any supporting evidence the Board disregards this late submission.

4.2 According to the patent in suit the above technical problem is essentially solved by releasably enclosing an intimate mixture of the photoactivator/dye of formula II

with a hydratable water-soluble crystalline salt within a substantially continuous glassy matrix of amorphous phosphate material, including sodium hexametaphosphate, of the formula $(M_2O)_x(P_2O_5)_y$. The enrobing of the porphine is achieved by spraying an aqueous solution comprising the amorphous phosphate onto moving granules of the said intimate mixture of the photoactivator/dye with the hydratable salt (page 3, line 44 to 47). According to the Appellant's plausible and uncontested submission during the oral proceedings, the presence of this hydratable water-soluble crystalline salt promotes the formation of the enclosing glassy phosphate matrix.

The Board is satisfied that the above technical problem is effectively solved.

4.3 It still remains to be decided whether the subject-matter of the disputed patent involves an inventive step.

There is no document before the Board which addresses the said problem of "bleeding" of photoactivator/dye from speckles in the presence of a nonionic surfactant.

4.3.1 Document (2) relates to a process for the manufacture of sodium percarbonate granules with improved storage stability and wear-resistance, especially for the use in particulate laundry detergent compositions (Claim 1; page 4, lines 4 to 6, and page 5, lines 16 to 20). Citation (2) suggests a process for the manufacture of the stabilised percarbonate granules whereby seed particles, which are smaller than the granules to be prepared, are impregnated with an aqueous sodium percarbonate phase, water is evaporated in a fluidised bed drier (Wirbelschichttrockner) and condensed alkali metal or ammonium phosphate is introduced into the fluidised bed drier (page 5, lines 21 to 29). The phosphate is normally

introduced as an aqueous solution. One possibility is to dissolve such phosphate in water and to spray the resulting solution into the fluidised bed drier; a second method is to introduce into the drier the phosphate dissolved in the aqueous phase which is used for impregnating the seed particles (page 6, lines 18 to 28). The best results are obtained with a hexametaphosphate, e.g. the sodium hexametaphosphate (page 6, lines 10 to 12).

Document (2) also discloses that the percarbonate granules have to be homogenous (page 4, line 27) and that this requirement is particularly well met by the disclosed process (page 5, lines 16 to 19, and especially page 19, lines 29 to 30). The percarbonate particles according to (2) show an improved stability against metal ions, such as iron ions and other components of the laundry detergent composition (page 19, lines 24 to 28). A measure of the stability is the loss in active oxygen on storage at 105°C for two hours (paragraph bridging pages 21 and 22). This disclosure, in the Board's judgement, makes it clear that the the term "stabilisation", as used in citation (2) and in the patent in suit, has two different meanings, since citation (2) is concerned with stabilising the percarbonate against chemical decomposition due to impurities and in particular due to metal ions, i.e. a technical problem which is completely different to that underlying the patent in suit.

In the Board's judgement, stabilisation of percarbonate against loss of active oxygen due to chemical decomposition, which makes homogeneity an essential feature, cannot be compared with stabilising the present coloured speckles against loss of photoactivator/dye due to "bleeding".

The Respondent's argument that the subject matter of the present patent was obvious over (2) as this document was already concerned with "stabilisation" using sodium hexametaphosphate and that it was not inventive to replace the storage-sensitive sodium percarbonate by another storage-sensitive material, i.e the photoactivator/dye of formula II is also rejected by the Board. Apart from the fact that stabilisation against chemical decomposition is of no importance in the present case, the technical problem underlying the patent in suit is the one defined above and not to find other ingredients that could be substituted for the percarbonate of document (2).

4.3.2 Moreover, document (2) discloses a process in which granules are built up from seed particles by spraying thereon appropriate aqueous solutions/phases as defined. This process simultaneously ensures the homogeneity of the obtained granules.

In contrast, the patent in suit is concerned with enrobing manufactured speckles with a continuous glassy matrix of amorphous phosphate.

The Respondent emphasised that, according to the process of the patent in suit, the glassy phosphate would by necessity infiltrate into the speckles and not be confined to an "outer shell". The Board accepts this view.

However, the Board cannot share the Respondent's opinion that, therefore, the inhomogeneity of the speckles according to the present patent as compared to the homogeneous granules of document (2) was in fact not a distinguishing feature because the Respondent's interpretation of document (2) is incompatible with its teaching. According to the present process the solution of

the glassy phosphate is sprayed onto already formed particles (speckles) and, while infiltration of these particles will occur to a certain degree, a substantially continuous encoating matrix will be formed on the speckles during evaporation which is aided by the hydratable salt. Thus, a protective layer is formed, which will not leave the porphine freely accessible at the surface of the obtained speckles. In contrast, the solution of the glassy phosphate of document (2) is not sprayed onto already formed particles but is rather used as an integral constituent to build up the granules, simultaneously with the other constituents. Thus, apart from the seed crystal, the granules of (2) will be homogeneous, without a protective "outer shell". Therefore, the solution suggested according to citation (2) is completely different compared to that of the disputed patent.

Therefore, in the Board's judgement, the skilled person would not turn to document (2) when looking for a solution to the above technical problem and, even if he had, he would have failed to find the solution suggested by the disputed patent.

4.3.3 Under these circumstances it is not necessary to further investigate the Respondent's allegation, contested by the Appellant, that percarbonate is a hydratable salt and that, consequently, the additional feature relied on by the Appellant, i.e. that the porphine is present in an intimate mixture with an hydratable water-soluble crystalline salt, was already disclosed implicitly in document (2).

4.3.4 Documents (4) and (5) were introduced by the Respondent as evidence that the use of hydratable salts is a known measure in this technical field. Both citations, however, are concerned with the problem of increasing the size of

perborate granules by agglomeration with the aid of such hydratable salts (see e.g. (4), page 1, lines 29 to 35 and lines 72 to 76; (5) page 1, lines 17 to 22 and lines 30 to 55). Hence, both documents address a completely different technical problem and give no indication of how the technical problem underlying the disputed patent may be solved.

4.4 It follows from the above that the cited references, neither on their own, nor in combination with each other, are such as to render obvious the subject matter of Claim 1 of the main request. Claims 2 to 5 cover particular embodiments of the subject-matter of Claim 1 and derive their patentability from this claim.

5. The main request being allowable, it is not necessary to deal with the auxiliary request.

Order

For these reasons, it is decided that:

1. The decision under appeal is set aside.

2. The case is remitted to the first instance with the order to maintain the patent on the basis of the description and the claims according to the main request filed during oral proceedings.

The Registrar: The Chairman:

E. Görgmaier K. Jahn

01078