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File Number: T 600/90 - 3.3.1
Application No.: 85 200 742.6
Publication No.: 0 164 778
Title of invention: Detergent powder compositions containing sodium perborate monohydrate

Classification: C11D 3/39

D E C I S I O N
of 18 February 1992

Proprietor of the patent: UNILEVER NV AND UNILEVER PLC

Opponents: 01) Henkel Kommanditgesellschaft auf Aktien
02) Procter + Gamble E.T.C.
03) Colgate-Palmolive Company

Headword: Perborate/UNILEVER

EPC Articles 54, 56 and 83

Keyword: "Sufficiency - well-known parameters"
"Prior use - not established"
"Inventive step (affirmed)"

Headnote



Case Number : T 600/90 - 3.3.1

D E C I S I O N
of the Technical Board of Appeal 3.3.1
of 18 February 1992

Appellant :
(Opponent 01)

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Decision under appeal :

Decision of the Opposition Division of the European Patent Office of 21 November 1989, with written reasons posted on 29 May 1990, concerning maintenance of European patent No. 0 164 778 in amended form.

Composition of the Board :

Chairman : K.J.A. Jahn
Members : R.W. Andrews
E.M.C. Holtz

Summary of Facts and Submissions

- I. European patent No. 0 164 778 in respect of European patent application No. 85 200 742.6 which was filed on 10 May 1985, was granted on 9 September 1987 (cf. Bulletin 87/37).
- II. Notices of opposition, which were filed on 3, 7 and 9 June 1988, requested the revocation of the patent on the grounds of insufficiency and lack of novelty and inventive step. The oppositions were supported, inter alia, by the following documents:
- (3) GB-A-1 520 127,
 - (4) EP-A-0 122 763,
 - (9) Letter from Henkel France s.a. dated 25 May 1988,
 - (11) Declaration of Nigel John Kermode filed on 7 June 1988,
 - (12) EP-A-0 098 108,
 - (13) GB-A-1 337 858, and
 - (14) Declaration of Jeffrey D. Painter and James R. McLeod dated 23 January 1987.
- III. By a decision delivered orally on 21 November 1989, with the corresponding interlocutory decision being issued on 29 May 1990, the Opposition Division maintained the patent on the basis of Claims 1 to 11 filed on 26 April 1989. The only independent claim of this set of claims reads as follows:
- "Detergent powder composition containing at least a detergent-active material, a detergency builder and sodium perborate monohydrate in particulate form, characterised in that the sodium perborate monohydrate incorporated therein has physical characteristics of specific surface

area (SA in m^2/g) and pore volume (PV in cm^3/g) such that the formula $(SA + 31.25PV - 16.25)$ is greater than 1."

The Opposition Division held that the disclosure of the invention was sufficient and that the claimed subject-matter was novel and involved an inventive step.

The Opposition Division decided that the evidence of Opponent 01 was not sufficient to prove the allegation of prior use and that none of the cited prior art documents disclosed detergent powder compositions containing sodium perborate monohydrate (PBM) having a perborate caking index (PCI) greater than 1.

The Opposition Division also considered that the proposed solution to the technical problem of providing detergent powder compositions containing PBM which, during storage under conditions of high humidity, remain free flowing with less tendency to cake and retain a rapid rate of dissolution was not obvious in the light of the teaching of documents (4) or (12).

IV. Appeals were lodged against this decision on 21, 23 and 31 July 1990 and the prescribed fees duly paid. Statements of Grounds of Appeal were filed on 22 September 1990 and 1 and 5 October 1990. In these Statements and during the oral proceedings held on 18 February 1992, the Appellants advanced essentially the following arguments:

Appellant 01 contended that the claimed subject-matter lacked novelty because of prior use and the disclosure of document (4).

This Appellant also argued that the subject-matter of the disputed patent did not involve an inventive step having regard to the combined teaching of documents (3) and (4).

Appellant 02 maintained that the disclosure of the patent was insufficient and that the alleged prior use had been made credible by the available evidence.

This Appellant also contended that the present subject-matter lacked novelty in the light of the disclosure of document (12). In his opinion, the present patent represented a thinly disguised attempt to re-patent the invention claimed in document (12).

With respect to inventive step, this Appellant argued that the thesis that the contested patent represents a selection of PBM's having superior properties remains unproven in the absence of any comparative data on the caking and dissolution performance of the PBM having a surface area of $7.85 \text{ m}^2/\text{g}$ of document (12) versus the material claimed in the patent in suit. Furthermore, the alleged superior properties are not substantiated by Table 1 of the disputed patent. Finally, the Appellant contended that the claimed advantages are, in fact, disclosed in documents (13), (12) and (3).

Appellant 03 maintained that the disclosure of the invention was insufficient in the absence of any details regarding the conditions under which the surface area of the PBM is to be measured. This Appellant also contended that SA and PV are not independent parameters and, therefore, a formula based on them is meaningless. Moreover, since it is well-known that when a powder is dried water is removed preferentially from the larger pores, during drying there will be a small increase in PV but a large increase in SA due to the relationship of SA to PV. In this Appellant's opinion, this was clearly illustrated by examples 4, 10 and 16 in Table 1 of the disputed patent.

- V. With respect to insufficiency, the Respondents maintained that both pore volume and surface area are well known concepts for particulate materials and therefore, a detailed description of their determination was unnecessary.

The Respondents also contended that, since it was not permissible to combine documents (9) and (11), prior use was not proved. The Respondents maintained that the claimed subject-matter was novel with respect to document (4) and that there could be no question of re-patenting the invention of document (12) since there were many PBM's having surface areas of more than 5 m²/g which are unsuitable for the purpose of the present invention.

The Respondents finally argued that none of the cited documents mentioned the morphology of the PBM's to be incorporated in powder detergent compositions and, therefore, did not provide any indication pointing in the direction of the present invention.

- VI. The Appellants requested that the decision under appeal be set aside and that the patent be revoked. The Respondent requested that the appeal be dismissed.
- VII. At the conclusion of the oral proceedings, the Board's decision to dismiss the appeal was announced.

Reasons for the Decision

1. The appeal is admissible.
2. There are no objections under Article 123 EPC to the present version of the claims. In particular, present

Claim 1 represents a combination of Claims 1 and 2 as filed and granted. Claims 2 to 11 correspond to Claims 3 to 12 as filed and granted.

3. The patent in suit relates to detergent powder compositions comprising surfactants, detergency builders and PBM in particulate form. Document (12), which is considered to represent the closest state of the art, discloses solid detergent compositions comprising surfactants, alkali metal aluminosilicate materials as detergency builders and PBM in particulate form having a specific surface area of at least 5 m²/g (cf. Claim 1).
- 3.1 A known disadvantage of PBM is that, particularly under conditions of high humidity, as it takes up water it tends to cake, resulting in a reduction in the free flowing properties of detergent compositions containing it. This water uptake also affects the bleach delivery of PBM since it results in the PBM dissolving less rapidly in the wash liquor (cf. patent specification, page 2, lines 11 to 18).
- 3.2 Although the stability of PBM with respect to the alkali metal aluminosilicate builder material in the compositions of document (12) was considered to be satisfactory, nevertheless it was impossible to predict with any degree of confidence whether, under storage conditions of high humidity, the PBM would lead to caking or not and whether its ratio of dissolution would be reduced or not.
- 3.3 Therefore, in the light of this closest prior art, the technical problem underlying the disputed patent is to be seen in providing detergent powder compositions containing PBM for which it is possible to predict with some confidence whether, after storage under high humidity

conditions, their caking profiles and rate of dissolution will be satisfactory.

According to the patent in suit, this technical problem is solved by ensuring that the PBM incorporated into the detergent powder compositions has physical characteristics of specific surface area (SA in m^2/g) and pore volume (PV in cm^3/g in pores lying between 0.1 and 1.0 μm) such that the formula $(SA + 31.25PV - 16.25)$ is greater than 1 (referred to as Perborate Caking Index, PCI).

In the light of the examples in the disputed patent which demonstrate that compositions containing PBM with PCI's greater than 1 have better caking profiles and superior bleach release than those containing PBM with PCI's less than 1, the Board considers it plausible that the above-defined technical problem has been solved.

4. From the above it is clear that in order to be able to carry out the invention, the skilled person must be in a position to determine the specific surface area and the volume in pores of diameter lying between 0.1 and 1.0 μm . According to page 2, lines 33 to 36, PV is measured by mercury intrusion using a Quantachrome Scanning Porosimeter and SA is determined by nitrogen gas adsorption, commonly known as the BET method. It is admitted by all the parties that the BET method is well known. The question is whether it is possible to obtain meaningful and reproducible results in the absence of any indication of the conditions under which the measurement of SA is carried out, particular, since the observed surface area of PBM decreases if it is stored under conditions that allow continual or periodic air exposure (cf. the last paragraph on page 3 of document (14)).

- 4.1 However, the skilled person is aware that PBM is a desiccant and would take steps to minimise its exposure to moist air. Moreover, in view of the nature of the sample, the skilled person would take measures to ensure that the conditions under which the measurements are carried out are such as to give reproducible results within the limits of the experimental error for the particular instrument used.

The present case is distinguished from that decided in the appeal T 241/90 of 14 August 1990 (unpublished) of this Board since the parameter (not more than 3 mg/kg of reactive titanium (IV)) relied on to differentiate the claimed compositions from prior art ones was new, whereas specific surface area and pore volume are well-known parameters of particulate solids.

- 4.2 Since the SA of PBM changes due to the uptake of water it was also alleged that the disclosure was insufficient insofar as there was no indication in the disputed patent when the SA of the PBM was to be measured. Moreover, the expression "incorporated therein" could imply that the physical parameters of the PBM are measured after it has been added to the other ingredients of the composition.

However, in the Board's judgment, the skilled person would realise in the absence of any indication to the contrary and Examples II to V, that, in order to obtain the benefits of the invention, the PCI of the PBM must be greater than 1 shortly before it is added to the other detergent ingredients.

- 4.3 Therefore, in the Board's judgment, the disclosure of the invention is sufficient to enable the skilled person to reduce it to practice.

5. In view of the Respondents' declaration submitted during oral proceedings in which it was stated that the PBM referred to in the disputed patent relates to PBM, with or without adsorbed liquid, so long as the physical characteristics of SA and PV is such that the PCI is greater than 1, Appellant 01 withdrew his objection of lack of novelty based on document (4). This document discloses a detergent composition comprising one or more surfactants to a total weight of 5 to 95% of the composition, one or more detergency builders to a total weight of 0 to 90% and a particulate PBM having adsorbed therein one or more activators (cf. Claims 1 and 24). Therefore, in view of the adsorbed liquid it is impossible for the PCI of the PBM to be greater than 1.

5.1 In accordance with the jurisprudence of the Boards of Appeal (see, for example, T 194/86 of 17 May 1988, unpublished), in order to decide whether an alleged prior use is comprised in the state of the art it is necessary to establish on the balance of probabilities (cf. for example, T 182/89, OJ EPO 1991, 391) the following:

- (i) the date on which the alleged prior use occurred;
- (ii) exactly what was used; and
- (iii) the circumstances relating to the use by which it was made available to the public.

5.2 Document (9), which is a letter signed by two employees of Henkel France s.a., discloses that two detergent powder compositions were marketed by Henkel France s.a. under the brand names Purblan and Pursol in France from May 1976 and September 1983 respectively. According to this letter both products comprised surfactants, detergency builder and PBM which was supplied by Air Liquide.

In the absence of any evidence which could cast doubt on the correctness of those facts, the Board finds that the date and circumstances of the alleged prior use has been established.

- 5.3 It still remains to be decided whether the PBM incorporated in the above-mentioned detergent powder compositions had the physical characteristics of SA and PV such that the PCI was greater than 1.

According to N.J. Kermode's declaration (document (11)), a sample of PBM produced by Air Liquide sometime in late 1985/early 1986 had a SA value of $10.3 \pm 0.6 \text{ m}^2/\text{g}$ (cf. letter dated 15 November 1985 from S.R. Daish of Harwell's Applied Chemistry Group) and a PV volume of $0.3 \text{ cm}^3/\text{g}$, resulting in a PCI of 3.43 ± 0.6 . It is clear from document (20) (a letter dated 22 April 1986 from J.W. Dimery of the University of Bristol submitted by Appellant 02 with his Grounds of Appeal) that the value $0.3 \text{ cm}^3/\text{g}$ is the volume in pores of diameter lying between 0.1 to $1.0 \text{ }\mu\text{m}$. Therefore, the Board is satisfied that this particular sample of PBM supplied by Air Liquide satisfies the requirement of the present Claim 1 with regard to its PCI.

However, since these measurements were made on samples of PBM produced after the application date of the disputed patent, in the Board's judgment it is not permissible to combine documents (9) and (11) and arrive at the conclusion that the sale of the detergent powder compositions, Purblan and Pursol, in France before this date constitutes prior use.

Document (11) also contains the statement that Mr Kermode had ascertained from representatives of Air Liquide that there had been no significant changes in their production

process for making PBM since 1982 or even earlier. Thus, it is on the basis of hearsay evidence amounting to no more than an unsubstantiated assertion that the Appellants contended that information in documents (9) and (11) should be combined. In the Board's judgment, this reported assertion by an unnamed employee of Air Liquide cannot be regarded as sufficient to establish, on the balance of probabilities, that prior use had occurred.

Even if it were accepted that Air Liquide had made no changes to their process for producing PBM in the relevant time period, there is no evidence before the Board that would prove that, during this period, the SA and PV did not vary from batch to batch. On the contrary, the Respondents have contended that the mere fact that no changes have been made to the process is no guarantee of constant physical characteristics of the PBM produced. Since it is the established jurisprudence of the Boards of Appeal that, if parties to opposition proceedings make contrary assertions which they cannot substantiate and the European Patent Office is unable to establish the facts of its own motion, the patent proprietor is given the benefit of the doubt, the Board considers that the prior use has not occurred even taking into account the unsubstantiated statement mentioned above.

- 5.4 Therefore, in the Board's judgment, the claimed subject-matter has not been made available to the public by prior use or by any of the cited documents.

- 6. The Board cannot agree that the disputed patent represents an attempt to re-patent the subject-matter of document (12) since this document is wholly silent with respect to PV. To solve the technical problem underlying the disputed patent a PBM of a specific morphology is required wherein SA and PV (between 0.1 to 1.0 μm) of the particles are

interrelated such that its PCI is greater than 1. However, it is clear from Table 1 of the disputed patent and the values of SA and PV (0.1 to 1.0 μm) reported in the Respondents' letter filed in response to the notice of opposition of Opponent 02 on 26 April 1989 (cf. in particular pages 2 and 4) that SA can be from low to high and also the PV (0.1 to 1.0 μm) can be low to high. In other words a high SA does not necessarily make the morphology of the PBM suitable for solving the present technical problem and many PBM's having SA values of at least 5 m^2/g within the claims of document (12) are unsuitable for the purpose of the present invention.

7. It still remains to be decided whether the claimed subject-matter involves an inventive step.

7.1 As previously mentioned document (12) discloses detergent powder compositions comprising surfactants, alkali metal aluminosilicate detergency builders and PBM having a SA of at least 5 m^2/g (cf. Claim 1). This document is concerned with providing detergent compositions containing the above-mentioned detergency builders in which the stability of the PBM is adequate (cf. page 2, lines 4 to 8).

Nevertheless, at lines 17 to 21 of page 19 of this document, it is disclosed that a composition containing PBM having a SA of 6.8 m^2/g was still in the form of a free flowing, non-lumpy, crisp powder after storage in wax-laminated packs for 12 weeks at 37°C and 70% relative humidity. However, it is not possible to determine whether this detergent powder would remain free from caking if it were to be stored under the more severe conditions used in the disputed patent (i.e. open phials, 20°C and 81% relative humidity). Thus, a similar composition containing PBM with a SA of 6.09 m^2/g and a PCI value of -0.785 was mostly caked after storage for 7 days in open phials at

about 20°C and 81% relative humidity, whereas the same composition containing PBM having a PCI value of 3.475 showed only light caking (cf. Example IV of the disputed patent). Thus, two compositions falling within the terms of Claim 1 of document (12) have different caking profiles. Therefore, from the teaching of document (12) the skilled person would not be in the position to deduce that, in order to obtain detergent powder compositions having predictable caking profiles, it is necessary to ensure that the PCI of the PBM is greater than 1.

- 7.2 Document (3) describes a process for the preparation of abrasion resistant PBM having SA of between 7 and 9 m²/g (cf. Claim 1 in combination with page 2, lines 31 to 35). It also discloses that less abrasion resistant PBM obtained by a prior art process has a SA of 10 to 14 m²/g (cf. page 2, lines 23 to 32). However, this document is completely silent with respect to the PV or the caking of the PBM obtained in accordance with either the claimed process or the prior art one. Therefore, this document does not contain any pointer in the direction of the proposed solution to the technical problem underlying the disputed patent.
- 7.3 Document (13) discloses that disinfectant compositions containing PBM dissolve quicker than corresponding ones containing sodium perborate tetrahydrate. However, this information would be of no significance to the skilled person faced with the present technical problem since this is concerned with the rates of dissolution of different samples of PBM. This document merely points to one of the advantages of PBM over sodium perborate tetrahydrate.
- 7.4 According to Appellant 03 SA and PV are not independent parameters since, if it is assumed that the pores are cylindrical in shape, the relationship between SA and PV

is given by $SA/PV = 1/R$, where R is the average pore radius. Therefore, it is this Appellant's opinion that the equation in Claim 1 is meaningless in a mathematical sense. However, not only is this relationship based on the assumption that the pores are cylindrical in shape, it also ignores the fact that PV in the equation in question is defined as the volume in cm^3/g in pores of diameter lying between 0.1 and 1.0 μm i.e. only a certain proportion of the total pore volume. Therefore, in the present situation it is considered that the above-mentioned relationship would not be valid. On the contrary, it has been demonstrated to the Board's satisfaction that it is empirically meaningful insofar as PBM having the physical characteristics of SA and PV such that the so-called PCI is greater than one solves the technical problem underlying the disputed patent.

7.5 Therefore, in the Board's judgment, the proposed solution to the above-defined technical problem is not obvious. Thus, Claim 1 is allowable. Claims 2 to 11, which relate to preferred embodiments of the composition in accordance with Claim 1, are also acceptable.

Order

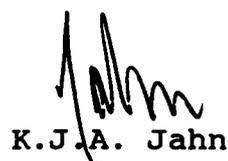
For these reasons, it is decided that:

The appeal is dismissed.

The Registrar:


E. Gorgmaier

The Chairman:


K.J.A. Jahn