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D E C I S I O N
of 15 June 2004

Case Number: T 1048/02 - 3.3.6

Application Number: 93870059.8

Publication Number: 0618290

IPC: C11D 17/06

Language of the proceedings: EN

Title of invention:

Flow aids for detergent powders comprising sodium aluminosilicate and hydrophobic silica

Patentee:

THE PROCTER & GAMBLE COMPANY

Opponent:

Henkel KGaA

Headword:

Flow aids/PROCTER

Relevant legal provisions:

EPC Art. 56

Keyword:

"Inventive step (main request) - no; obvious alternative"
"Inventive step (first auxiliary request) - yes: non-obvious improvement"

Decisions cited:

-

Catchword:

-



Case Number: T 1048/02 - 3.3.6

D E C I S I O N
of the Technical Board of Appeal 3.3.6
of 15 June 2004

Appellant: Henkel KGaA
(Opponent) VTP (Patente)
D-40191 Düsseldorf (DE)

Representative: -

Respondent: THE PROCTER & GAMBLE COMPANY
(Proprietor of the patent) One Procter & Gamble Plaza
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Decision under appeal: Decision of the Opposition Division of the
European Patent Office posted 16 August 2002
rejecting the opposition filed against European
patent No. 0618290 pursuant to Article 102(2)
EPC.

Composition of the Board:

Chairman: P. Krasa
Members: G. Dischinger-Höppler
U. J. Tronser

Summary of Facts and Submissions

I. This appeal is from the decision of the Opposition Division to reject the opposition and to maintain the European patent No. 0 618 290 on the basis of the claims as granted, the independent Claims 1 and 9 reading:

"1. A granular detergent component or composition having a bulk density of at least 700 g/l which comprises:

- i) a detergent powder which comprises at least one nonionic surfactant which is liquid at temperatures below 40°C; and
- ii) from 0.5 to 15% by weight of a powdery flow aid characterised in that the flow aid comprises sodium aluminosilicate and hydrophobic silica wherein the ratio of the sodium aluminosilicate to hydrophobic silica in component ii) is from 100:1 to 3:1.

9. A process for making a free-flowing detergent powder having a bulk density of at least 700 g/l which comprises the steps of:

- i) making a nonionic surfactant system which comprises at least one nonionic surfactant which is liquid at temperatures below 40°C;
- ii) making a granular detergent powder having a bulk density of at least 650 g/l;
- iii) spraying on a part of, or all of the nonionic surfactant system of step i) on to the granular detergent powder of step ii);

iv) mixing the product of step iii) with a premixed powder, said premixed powder comprising sodium aluminosilicate and hydrophobic silica, wherein the premixed powder is used at a level of from 3% to 15% by weight of the finished detergent component or composition and having a ratio of the sodium aluminosilicate to hydrophobic silica is from 100:1 to 3:1."

II. A notice of opposition had been filed against the granted patent, wherein the Appellant (Opponent) sought revocation of the patent on the grounds of Article 100(a) EPC for lack of novelty and lack of inventive step (Articles 52(1), 54(2) and 56 EPC). The opposition was based inter alia on the following documents

D1: EP-A-0 477 974 and

D5: EP-A-0 513 824.

III. In its decision, the Opposition Division held that the subject-matter of Claim 1 as granted was novel over the cited prior art and - considering the data provided in Table 2 of the patent in suit and with the letter of 17 November 1997 - based on an inventive step.

Consequently, the Opposition Division did not consider the six auxiliary requests filed by the Respondent (Patent Proprietor) during the opposition proceedings.

IV. With its letter of response dated 6 June 2003 to the Appellant's statement of grounds of appeal, the Respondent filed amended claims in four auxiliary

requests which are identical to those filed in the first to fourth auxiliary request during opposition proceedings under cover of the letter dated 23 May 2002.

The claims of the first auxiliary request differ from those of the main request only in that the term "and the particles of detergent powder (i) are coated with the powdery flow aid (ii)" is added at the end of Claim 1.

V. Upon requests made by both parties, oral proceedings before the Board of Appeal were held on 15 June 2004, in the course of which the Appellant dropped its novelty objection.

VI. The Appellant, orally and in writing, maintained that the claimed subject-matter was not based on an inventive step for the following reasons:

- It followed from the description of the patent in suit that the subject-matter of Claim 1 was not restricted to granular detergent material having the flow aid only on the surface of the granules.
- The technical problem of reducing nonionic leakage from detergent granules underlying the patent in suit was already addressed in document (5) and solved by a coating with fine particles selected from aluminasilicates and amorphous silica derivatives, the latter including hydrophobic silica.

- The subject-matter of Claim 1 differed from that of document (5) only in that the detergent granules comprised a mixture of aluminosilicates and amorphous silica in a ratio of 100:1 to 3:1 and was to be regarded as a selection from the detergent compounds disclosed in document (5).

- The advantages set out in the examples of the patent in suit were not surprising since it was known from document (1) that the tendency of nonionic surfactants to leak out from a detergent powder can be prevented by the addition of an oil absorbing carrier such as Sipernat D10 (Degussa) which was a hydrophobic silica. It was therefore obvious to use hydrophobic silica as an additive either within the granules of document (5) and/or in their coating.

- Finally, the remaining feature relating to the identification of the optimum ratios of aluminosilicate and hydrophobic silica was merely the result of routine experimentation and economy considerations.

VII. The Respondent submitted the following arguments:

- It was apparent from the patent in suit that the flow aid was present only as a coating on the surface of the particles of the detergent powder.

- It was shown in the examples of the patent in suit that the claimed detergent composition provided benefits over that of document (5) with respect to the flow properties, nonionic leakage, bulk density on storage and particle size distribution.
- There was no suggestion in the art that the particular combination of characteristics of the flow aid would lead to those benefits.

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

The Respondent requested that the appeal be dismissed and that the patent be maintained or the decision under appeal be set aside and the patent be maintained on the basis of one of the four auxiliary requests submitted under cover of the letter dated 6 June 2003.

Reasons for the Decision

1. *Main request*

Novelty no longer being contested, the only issue to be decided is whether the subject-matter of the claims, having regard to the state of the art according to Article 54(2) EPC, was obvious to a person skilled in the art or whether it involved an inventive step in accordance with Article 56 EPC.

1.1 The patent in suit relates to the use of flow aids for high bulk granular detergent compositions comprising nonionic surfactants which are liquid at ambient

temperatures, in order to reduce the tendency of the liquid nonionics to leak out from the detergent powder (page 2, lines 5 to 12). According to the patent in suit, such use of flow aids is known in the art, e.g. from document (1) by disclosing nonionic powdery detergent compositions comprising 10 to 60% by weight of crystalline aluminosilicate and an oil absorbent carrier, and from document (5) disclosing a process in which nonionic detergent granules are surface coated with particles of less than 10 μm in size (page 2, paragraph [0003] in combination with paragraphs [0007] and [0008]).

1.2 However, in view of this prior art it is still an objective of the patent in suit to reduce the leakage problem. Further objectives consist in the reduction of changes in bulk density upon storage and achieving of improved control over particle size distribution of the finished product (page 2, paragraphs [0010] to [0012]).

1.3 According to the Appellant all these objectives relate to one and the same technical problem of avoiding caking of the composition upon storage, since it was within the common general knowledge of those skilled in the art that powdery detergents having a broad particle size distribution tend to be more sticky due to the increased number of points of contact between the particles as compared to a powder having a small particle size distribution, and that this tendency was increased by any surfactant leakage resulting in an undesired bulking and caking of the compositions.

The Appellant further argued that document (5) was concerned with the same technical problem and represented the closest prior art for the assessment of inventive step.

1.4 The Respondent did not contest the Appellant's arguments with regard to the technical problem and the Board also does not have any reasons for doubts in this respect. Further, document (5) deals in fact with the technical problem of producing a granular detergent composition of high bulk density containing a nonionic surfactant and having non-caking properties and excellent powder fluidity (page 2, lines 3 to 6). Thus, under the above considerations of the Appellant, document (5) indeed qualifies as a suitable starting point for the evaluation of inventive step.

1.5 Document (5) proposes to solve the above technical problem by mixing a detergent material comprising 20 to 89 parts by weight of a builder, 1 to 20 parts by weight of a porous oil absorbing carrier and from 10 to 60 parts by weight of a nonionic surfactant, granulating said mixture and mixing said granules with 0.5 to 30 parts by weight of fine particles to thereby coat the surface of the granules with the fine particles and produce nonionic detergent granules having a bulk density of from 0.6 to 1.2 g/ml (page 3, lines 6 to 8 in combination with lines 16 to 19 and Claims 1 and 2). Preferably, the builder may comprise aluminosilicate, e.g. Zeolite 4A in amounts ranging of from 10 to 25% by weight (Claim 14, page 8, lines 25 to 27 and Examples), the porous oil absorbing carrier is an amorphous silica derivative including hydrophobic silica, calcium silicate derivatives and

aluminosilicate derivatives, the latter being particularly preferred (Claims 16 and 17, page 9, line 19 to page 20, line 9 and Examples) and the nonionic surfactant is one which is a liquid or a paste at temperatures below 40°C (Claim 13; page 10, lines 10 to 12, and Examples).

The fine particles used for coating the surface of the granules consist of one or more compounds selected from aluminosilicates and the same amorphous silica derivatives mentioned above, including oil absorbing, i.e. hydrophobic silica (page 11, lines 54 to 56 in combination with page 9, lines 19 to 30, and Claim 19). However, Zeolite 4A and amorphous aluminosilicate are preferred (Examples).

- 1.6 According to the Respondent, the claimed invention was intended to cover only embodiments wherein the flow aid was comprised in the coating material. This was apparent from paragraph [0055] on page 7 of the patent in suit. Therefore, the claimed granular detergent composition differed from that obtained by the process of document (5) in that a combination of sodium aluminosilicate and hydrophobic silica in the particular ratio of from 100:1 to 3:1 is used in the coating material.

The Appellant disagreed and argued that the patent in suit also included embodiments where the flow aid was incorporated within the granules. Reference in this respect was made to paragraphs [0068] and [0074] on page 9 of the patent.

1.7 Paragraph [0055] contains the following statements:

"It is necessary to mix the flow aid with the rest of the detergent composition. In order to achieve the benefits of the invention, a level of the flow aid of from 0.5% to 15% by weight of the detergent composition is **then** mixed to coat the surfaces of the granules."
(emphasis added by the Board).

The Board concludes from the using of the term "then" in the second statement that a coating of flow aid is applied to a detergent composition which already contains flow aid.

On the other hand, paragraphs [0066] to [0068] propose that fine dispersion mixing or granulation of the liquid nonionic surfactant in the presence of a powder comprising sodium aluminosilicate and hydrophobic silica is one process for obtaining granular detergent components which comprise the nonionic surfactant. In particular it is stated in paragraph [0068] that

"One such process is to agglomerate by the following steps: i) fine dispersion mixing or granulation of at least one nonionic surfactant which is liquid at temperatures below 40°C in the presence of an effective amount of a powder which comprises sodium aluminasilicate and hydrophobic silica, wherein the ratio of the sodium aluminosilicate to silica in component ii) is from 100:1 to 3:1".

The Board agrees with the Appellant that this embodiment would not necessarily result in a product wherein the nonionic surfactant is coated with the

powder comprising sodium aluminosilicate and hydrophobic silica but in a mixture wherein the nonionic surfactant is distributed throughout the powder material.

In paragraph [0073] it is further stated that according to the process of the invention the detergent granules are prepared by the fine dispersion mixing or granulation described above, i.e. in paragraph [0068], followed by spraying with some or all of the nonionic surfactant onto the granules in a suitable mixer. This process is further explained in paragraph [0074], which reads:

"The following steps **may** be used in **this** embodiment of the invention (emphasis added by the Board):

- i) making a nonionic surfactant system which comprises at least a nonionic surfactant which is liquid at temperatures below 40°C;
- ii) making a granular detergent powder having a bulk density of at least 650g/l;
- iii) spraying on a part of, or all of the nonionic surfactant system of step i) onto the granular detergent powder of step ii);
- iv) mixing the product of step iii) with a premixed powder which comprises sodium aluminasilicate and hydrophobic silica, wherein the premixed powder is used at a level of from 3% to 15% by weight of the finished detergent component or composition and that the ratio of the sodium aluminosilicate to silica in component ii) is from 100:1 to 3:1."

It is not clear from the patent in suit whether or not the term "component ii)" mentioned in paragraphs [0068] and [0074] is meant to denote the product of step ii) in paragraph [0074] made by the fine dispersion mixing disclosed in paragraph [0068], nor is there any other information about the composition of the product of step ii). However, given the above somewhat contradictory statements in paragraphs [0055] and [0074] on the one hand and in paragraph [0068] on the other hand as concerns the essential process features for obtaining the claimed subject-matter, in particular with respect to the coating step, and in the absence of any further explanations, the claims must be given the broadest possible interpretation within the overall disclosure of the patent in suit. The Board concludes, therefore, that the subject-matter of Claim 1 covers embodiments wherein the flow aid composition is distributed throughout the granular detergent composition or component and/or is present as a coating on the surfaces of the granules.

It follows that, in the case where the flow aid is merely distributed within the granules, the claimed subject-matter differs from what is disclosed in document (5) in that sodium aluminosilicate and hydrophobic silica in a ratio of from 100:1 to 3:1 are incorporated within the granules in an amount of from 0.5 to 15% by weight (see 1.5 above).

- 1.8 There is no evidence on file showing that, in view of document (5), the technical problem mentioned in the patent in suit (see above 1.2) is solved by the embodiment having the flow aid incorporated within the granules. It would have been the Respondent's burden to

provide such evidence since document (5) has been cited in the application as filed as prior art relevant with respect to the invention (paragraph bridging pages 2 and 3). On the other hand, it would have been for the Appellant relying on document (5) as the closest prior art to show that this embodiment does not provide fluidity and non-caking properties comparable to the products obtained by the process disclosed in document (5).

In the absence of evidence in either respect, the technical problem to be solved in view of document (5) may, thus, be seen to consist in providing an alternative composition having also non-caking properties and excellent powder fluidity, and the Board has no reasons to doubt that this technical problem is actually solved by the claimed subject-matter.

1.9 It remains to be decided whether, in view of the available prior art documents, it was obvious for someone skilled in the art to solve this problem by the means claimed, i.e. the incorporation into the granules of the particular amounts and ratios of aluminosilicate and hydrophobic silica.

1.10 Document (5) does not disclose the use of aluminosilicate in combination with hydrophobic silica. Such a combination is, however, explicitly mentioned in document (1). This document is also concerned with the problem of providing a detergent composition which is free from leaking of the liquid nonionic surfactant, non-caking and has excellent flow (page 2, lines 3 to 6). In particular Example 5 discloses a composition comprising Zeolite 4A in combination with Sipernat D10[®],

the latter being an oil-absorbing carrier (page 3, line 55 to page 4, line 9) and, undisputed, a hydrophobic silica. It is stated in document (1) that it is the addition of the oil-absorbing carrier which prevents the leaking of the nonionic surfactant and provides the fluid and non-caking properties (page 2, lines 38 to 43).

Document (1) does neither disclose the particular amounts of flow aid nor the ratios of aluminosilicate and hydrophobic silica given in Claim 1. It is, however, evident that the optimum amount of flow aid and, in particular, the optimum amount of the oil absorbing hydrophobic silica within the flow aid largely depends on the amount of nonionic surfactant contained in the composition.

- 1.11 The Board, therefore, concludes that a person skilled in the art would have incorporated hydrophobic silica as an oil-absorbing material as suggested in document (1) in the granules of document (5) in amounts suitable to at least preserve the non-caking and fluidity properties of the product in order to provide an alternative composition, thereby arriving in an obvious manner at the subject-matter of Claim 1.

- 1.12 For these reasons, the Board finds that the subject-matter of Claim 1 does not comply with the requirements of Article 52(1) and 56 EPC.

2. *First auxiliary request*

2.1 Amendments

By the feature "and the particles of detergent powder (i) are coated with the powdery flow aid (ii)" added at the end of Claim 1, its subject-matter is limited to those embodiments wherein the flow aid mentioned under ii) is wholly included in the coating on the granules without, however, excluding the presence of further flow aid within the detergent powder (i) (see 1.7 above).

The Board is satisfied that no problems under Article 84 EPC have been introduced by the amendment made and that the claims comply with the requirements of Article 123(2) and (3) EPC. No objections have been raised by the Appellant in this respect.

2.2 Inventive step

2.2.1 The subject-matter of Claim 1 differs from the product obtained by the process of document (5) in that the coating material contains sodium aluminosilicate in combination with hydrophobic silica in a ratio of from 100:1 to 3:1 (see 1.5 and 1.6 above).

2.2.2 In the examples of the patent in suit the flow aid is composed of Zeolite A and hydrophobic silica in the ratios 80:20 and 90:10 and only present as a coating on the granules in accordance with the claimed subject-matter (page 10, paragraphs [0079] to [0080]).

It is shown that the embodiments according to the invention (Tables 1 and 3, compositions 3 and 6) provide narrower particle size distribution, less nonionic leakage and less change in density upon storage as compared with embodiments representative for the prior art according to document (5) using 100% zeolite for coating (Tables 1 and 3, composition E) if the same amounts of flow aid of 10% based on the finished product are applied (page 11, paragraph [0086] to page 12, paragraph [0091] and Examples 8 to 10).

2.2.3 The Appellant contended that the advantages shown in the examples were marginal, in particular as far as the particle size distribution and change of density were concerned; nevertheless the presence of the effects was not disputed.

2.2.4 Taking into account the uncontested considerations of the Appellant in respect of how caking is influenced by particle size distribution and nonionic leakage (see 1.3 above), the technical problem credibly solved in view of document (5) can, thus, be seen in the provision of a detergent composition of further improved non-caking properties and powder fluidity.

2.2.5 The Appellant argued that it was known from document (1) that this effect is provided by the introduction of hydrophobic silica into the detergent granules. A skilled person would, therefore, have expected that the addition of hydrophobic silica onto the surfaces of the granules would also reduce nonionic leakage and consequently the undesired caking of the composition.

2.2.6 These arguments are not convincing since they are based on hind-sight considerations for the following reasons:

Document (5) itself mentions hydrophobic silica as one of several possible oil-absorbing carriers for both, the detergent granules and the coating material (page 9, lines 26 to 35). Nevertheless, aluminosilicate derivatives are preferred (Claims 16 and 17 and Examples) in both instances. Document (1) does not contain any further information. Its disclosure is even restricted to the introduction of an oil-absorbing carrier into the granules whereas any coating of the surfaces is carried out with crystalline aluminosilicate powder such as Zeolite 4A alone (page 5, lines 29 to 34 and Examples). Document (1) does, in particular, not suggest that amorphous silica, i.e. hydrophobic silica such as Sipernat D10[®] (Claim 3 and Table 2, Example 5) would perform better than an amorphous aluminosilicate (e.g. Tixolex 25[®]), sodium mordenite HSZ-640 NAA[®] or a clayey substance (e.g. Perlite 4159[®]) (Claims 4 and 5, page 4, lines 3 to 9, page 5, lines 2 to 6 and Table 4, Examples 1, 2, 5 and 6). On the contrary, document (1) explicitly prefers sodium mordenite HSZ-640 NAA[®] and amorphous aluminosilicates, from the latter in particular those having cation exchange capacity and being able to act as a builder (page 4, line 10 to page 5, line 1). Thus, there is no hint in the prior art on file towards a preference for hydrophobic silica as an oil-absorbing material in a granular nonionic detergent composition, let alone when applied to the surface of the detergent granules.

2.3 Therefore, a person skilled in the art would not have expected that the non-preferred oil-absorbing carriers of the prior art would provide better non-caking and fluidity properties to a nonionic granular detergent composition than the preferred ones, when applied within a coating composition.

The Board, therefore, concludes that it was not obvious for someone skilled in the art seeking to improve the non-caking properties of the products obtained by the process disclosed in document (5) to use for the coating of the detergent granules a flow aid which comprises hydrophobic silica in addition to the aluminosilicate in a ratio of aluminosilicate to hydrophobic silica of from 100:1 to 3:1.

2.4 The reasoning given above in respect of the detergent composition of Claim 1 applies also to the process of Claim 9 for making a free-flowing high bulk density detergent powder containing a nonionic surfactant which is liquid at temperatures below 40°C.

3. For all these reasons, the Board holds that the subject-matter of independent Claims 1 and 9 involves an inventive step (Articles 52(1) and 56 EPC).

The dependent Claims 2 to 8 and 10 refer to specific embodiments of Claims 1 and 9 and derive their patentability therefrom.

4. Since the claims of the first auxiliary request comply with the requirements of the EPC, there is no need to consider the second to fourth auxiliary requests.

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 Claims 1 to 10 of the first auxiliary request submitted under cover of the letter dated 6 June 2003, and the description to be adapted thereto as necessary.

The Registrar:

The Chairman:

A. Wallrodt

P. Krasa