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Bezeichnung der Erfindung: Process for the manufacture of detergent
Title of invention: compositions containing sodium aluminosilicate
Titre de l'invention :

Klassifikation / Classification / Classement : C11D 3/12

ENTSCHEIDUNG / DECISION

vom / of / du 19 September 1990

Anmelder / Applicant / Demandeur :

Patentinhaber / Proprietor of the patent /
Titulaire du brevet :

Unilever PLC/Unilever NV

Einsprechender / Opponent / Opposant :

Henkel Kommanditgesellschaft auf Aktien

Stichwort / Headword / Référence : Detergent compositions/UNILEVER

EPU / EPC / CBE Articles 56 and 114(2)

Schlagwort / Keyword / Mot clé : "Inventive step (yes) - non-obvious alternative"

Leitsatz / Headnote / Sommaire

Summary of Facts and Submissions

- I. European patent No. 61 296 was granted with four claims on European patent application No. 82 301 373.5.

Claim 1, the only independent claim, reads as follows:

"1. A process for manufacturing washing powder comprising a synthetic aluminosilicate as a detergency builder, or part of the builder, which comprises the steps of

- (a) spray-drying a slurry comprising an anionic detergent active compound and sodium silicate to form a spray-dried powder;
- (b) binding the spray-dried powder and a detergency builder compound at least partly comprising a synthetic aluminosilicate with a liquid binder to form granules or agglomerates; and
- (c) drying the granules or agglomerates.

- II. The Appellants (Opponents) filed a notice of opposition against the European patent requesting revocation of the patent on the grounds that the claimed process was lacking novelty and inventive step (Article 100(a) EPC) having regard to the following two prior art documents:

- (1) US-A-4 096 081
- (2) DE-B-2 529 685.

- III. The Opposition Division rejected the opposition and maintained the patent as granted.

The reasons for maintaining the patent were in essence the following:

- (i) Document (1) related to a laundering composition comprising two discrete types of particle, the first of which was an intimate mixture comprising an aluminosilicate, an organic agglomerating agent and an inorganic salt and the second of which was a spray-dried detergent granule containing a surfactant. According to Example II the aluminosilicate-containing particles might also be spray-dried granules. According to Example III the granules of Example II were mixed with detergent granules containing a nonionic surfactant and sodium silicate.

Document (2) related to a method of making detergent compositions comprising an aluminosilicate, wherein at least the portion of the aluminosilicate in the form of a powder was blown into a spray-drying tower at about the same height as the spray jet. According to Examples 1 and 2, the aqueous slurry being spray-dried included an anionic surfactant (ABS) and sodium silicate. Drying of the product in the tower resulted in agglomerated particles containing the aluminosilicate as well as the spray-dried slurry constituents.

Since neither of documents (1) and (2) disclosed a process as claimed the subject-matter of Claim 1 was new.

- (ii) The problem to be solved in the patent in suit was that of poor powder properties in washing powders containing synthetic aluminosilicates.

According to document (1), which was concerned with this problem, it was not only known that sodium silicate is a component which resulted in crisp, free-flowing granules, but also that in a slurry containing both aluminosilicate and a silicate, a chemical reaction takes place leading to cross-polymerisation of the aluminosilicate molecules

through bridging by the silicate, thus negating the beneficial effects of the silicate. It was found in this prior document that if the aluminosilicate was added after the slurry had been spray-dried, free-flowing non-friable compositions were obtained.

The subject-matter of Claim 1 was thus considered not to be obvious in the light of document (1).

Document (2) was not related with the problem of the interaction between sodium silicate and aluminosilicate and thus already differed in its aim from the patent in suit. Thus the products produced according to Example 1 of document (2) would be subject to the interaction reaction between silicate and aluminosilicate on account of the elevated temperature in the spray-drying tower and the fact that the residence time would be considerable since the aluminosilicate was introduced at the same height as the jets. It was concluded that a skilled person wishing to make a product free from interaction between silicate and aluminosilicate would have been led away from the teaching of document (2) and, therefore, the subject-matter of Claim 1 of the patent in suit was also not obvious in the light of document (2).

IV. The Appellants filed a notice of appeal against this decision and submitted a statement of grounds.

(i) During the appeal proceedings they filed the following three documents:

(3) Firmenschrift "Natriumaluminiumsilikat HAB A 40"
Degussa, 1979;

(4) Manufacturing Chemist & Aerosol News, October 1978,
pages 51 and 60;

(5) Tenside Detergents 17 (1980) 4, O. Pfrengle,
pages 197 to 200.

- (ii) Oral proceedings took place, during which an auxiliary request was submitted wherein Claim 1 was amended by limiting step (c) as follows:

"(c) drying the granules or agglomerates in a fluidized bed."

- (iii) The Appellants argued essentially as follows:

The submission of documents (3) to (5) should not be considered as being too late with regard to Article 114(2) EPC, for the reasons that in particular document (5) should have been known to the Respondents since the aluminosilicate mentioned in the document is that used by the Respondents according to the description of the patent in suit.

In Example III of document (1) comparative tests were described which presented precisely the same kind of mixture used as a slurry which had to be spray-dried as that described in the patent in suit. A separate powder was mixed to the spray-dried slurry which had the same particle size. It was not necessary to agglomerate the powders since the same particle size of both powders ensured a satisfying mixture. The difference between the process described in Example III and that of the patent in suit was that no final drying step was carried out in said example. This difference was, however, not relevant. Although the mixture in Example III was used for the purpose of comparison this disclosure had nevertheless to be considered as state of the art.

In addition, document (2) disclosed in its Example 5 a process almost identical to the one claimed in the patent in suit.

~~Among the newly submitted documents, in particular~~ document (5) disclosed in its Example 1 on page 199 a typical spray-drying process containing the aluminosilicate. In particular a combination of document (1) with document (5) led directly and obviously to the process as claimed in the patent in suit.

The same conclusion held true for a combination of the disclosure of document (1) and the document (4), the latter disclosing exactly the same aluminosilicate as described in the disputed patent as new builder for detergents and which was, according to this same document, produced by spray mixing, whereby the aluminosilicate could be fed into the production system together with other solid components of the detergent and sprayed with the tenside solution (see page 60). Literally it was mentioned there: "Which particular working method should be recommended depends on the technical preconditions, the formulation of the detergent, and the nature of the other solid components."

It was thus a simple step for a skilled person to adopt the necessary process to the conditions given by the particular choice of the components.

V. The Respondents submitted essentially the following arguments:

The newly filed documents (3) to (5) should be rejected as being submitted too late within the meaning of Article 114(2) EPC.

Example III of document (1) disclosed a washing powder prepared in a way which could be compared to that of the patent in suit (see column 17, lines 53 to 57). However, this product was used there for comparison purposes and it was stated further that "the product containing the aluminosilicate prepared in accordance with the present invention (A) cleaned the fabric swatches significantly better than the comparative product" (see column 17, lines 62 to 65), indicating that products according to the comparison example should be avoided. This clearly led away from the invention claimed in the patent in suit. In any event it was not even described for the comparison product that the binder worked at room temperature, since the binder mentioned in document (1) had a melting point of between 30 - 200°C. This feature, however, was important, since in the claimed process the degree of the moisture was important for the quantitative binding of the aluminosilicate, whereby the final drying step could be carried out in a conventional way. The process of Claim 1 therefore differed completely from that disclosed in the examples of document (1).

With regard to document (2), it was apparent that there a complicated spray-drying equipment was necessary. Therefore, the skilled person could not find any incentive in document (2) to resort to the described process, let alone to modify it. Consequently the process according to the patent in suit could not be considered as being obvious in the light of document (2).

Furthermore, no combination of document (1) or (2) with any of the newly-filed documents would lead in an obvious manner to the claimed process. None of the newly-filed documents (3) to (5) provided any teaching which would (not: could) have led the man skilled in the art to modify the process according to documents (1) or (2) to arrive at a process within the claims of the present patent.

- VI. The Appellants request that the decision under appeal be set aside and that the patent be revoked.

The Respondents request that the appeal be dismissed and ~~the patent be maintained, and, as an auxiliary request~~ that the patent be maintained on the basis of the claims filed during oral proceedings.

Reasons for the Decision

1. The appeal is admissible.
2. The question of novelty was not at issue during the appeal proceedings and the Board sees no reason to raise this question on its own motion. The process of Claim 1, therefore, is novel.
3. Closest prior art and the problem
 - 3.1 After consideration of the prior art documents cited during the proceedings, the Board considers document (1) to be the closest prior art. This document is directed to a process for preparing a laundering composition comprising two discrete types of particle, the first of which is an intimate mixture comprising an aluminosilicate, an organic agglomerating agent and an inorganic salt and the second of which is a spray-dried detergent granule containing a surfactant (see Claim 1). According to Example II the aluminosilicate-containing particles may also be spray-dried granules. According to Example III the granules of Example II are mixed with detergent granules containing a nonionic surfactant and sodium silicate.

This process leads to a washing powder which shows satisfying powder properties (e.g. free-flowing and substantially non-friable) while avoiding a chemical reaction leading to cross-polymerisation of the aluminosilicate molecules through bridging by the silicate when a slurry contains aluminosilicate and a silicate, thus negating the beneficial effects of the silicate.

Surprisingly, very low levels of the inorganic salt substantially reduce friability (see column 1, lines 33 to 48; column 3, lines 15 to 35; column 16, lines 17 to 25 and column 18, lines 11 to 16). As further shown in Example III, mixing together all components in a single step does not lead to a satisfactory product.

3.2 Starting from document (1) the problem underlying the patent in suit thus can be seen in providing an alternative process for the production of washing powders containing synthetic aluminosilicates.

4. The solution

The proposed solution according to Claim 1 as granted comprises the following steps: first, spray-drying a slurry comprising an anionic detergent active compound and sodium silicate to form a powder; second, binding the spray-dried powder and a detergency builder compound, at least partly comprising a synthetic aluminosilicate with liquid binder to form granules or agglomerates which are subsequently dried.

There are no doubts that the indicated problem has been solved by the claimed proposal having regard to the series of experiments described in the description of the patent in suit (see the example described in column 3, lines 11 to 24 and column 4, lines 1 to 24, supported by the data

given in Tables 1 and 2 of the disputed patent). In particular, compositions A to D produced in accordance with the claimed process show crisp and free-flowing washing powders having satisfactory solubility/dispersion properties. ~~This is not contested by the Appellants.~~

5. Inventive step

5.1 As stated above, document (1) describes a specific process in which two separately prepared products are mixed together, one containing aluminosilicate and the other surfactant, whereby the preparation of the particulate aluminosilicate requires not only the presence of an organic agglomerating compound, but also of an inorganic salt mainly responsible for acceptable powder properties such as resistance to crumbling (friability). An additional information conferred by Example III is that by mixing together all components of the washing powder in a single step, no satisfactory product is obtained. An important result of the comparative test was indeed that the product containing the aluminosilicate prepared in accordance with the process of document (1) cleaned the fabric swatches significantly better than the comparative product. One may now ask whether this information has to be interpreted in the way as it was done by the Appellants, i.e. that in the case of the comparative product a process had been disclosed which apparently is analogous to that of Claim 1 of the patent in suit and thus anticipates this process; or whether the negative outcome of the comparative test is a hindrance for the skilled person to further investigate in a process of that kind at all. In the opinion of the Board, the Appellant's argument is clearly the result of an inadmissible ex post facto analysis and therefore in no way convincing. Therefore, the latter interpretation is to be retained, because it is unlikely that the comparative experiment

contained in Example III would have encouraged the man skilled in the art to further investigate this route since the comparison is a negative one.

5.2 The question therefore arises whether the further documents (2) to (5) cited by the Appellant contain additional technical information which might have rendered the claimed solution obvious.

5.3 Document (2) also relates to a quite different method of making detergent compositions comprising an aluminosilicate, wherein at least a portion of the aluminosilicate in the form of a powder is blown into a spray-drying tower at about the same height as the spray jet. According to Examples 1 and 2, the aqueous slurry being spray-dried included an anionic surfactant (ABS) and sodium silicate (column 10, components (1), (7), (8)). Drying of the product in the tower results in agglomerated particles containing the aluminosilicate as well as the spray-dried slurry constituents (column 9, lines 22 to 36).

In view of this, the Respondents are certainly right to point out that the advantage of the claimed process over the known one is that the firstly spray-dried powder may quantitatively absorb the synthetic aluminosilicate, whereby it is indeed avoided that the very fine powder of this synthetic aluminosilicate gets lost by being blown through the drying tower because it could not be bound quickly and sufficiently enough, and secondly, the mentioned disadvantage of any undesired chemical crosslinking can be circumvented at the same time.

5.4 Documents (3) to (5) relate to processes wherein it is proposed that, if the detergent is produced by spray-mixing, the synthetic aluminosilicate (HAB A 40 in this

case) can be fed into the production system together with the other solid components of the detergent and sprayed with the tenside solution. In many cases the admixture of HAB A 40 after completion of the actual spray mixing process proves to be advantageous. Which particular working method should be recommended depends on the technical preconditions, the formulation of the detergent, and the nature of the other solid components (see documents (3) and (4)). In particular, in Example 1 of document (5) it is disclosed that a spray-dried powder, comprising sodium sulphate, sodium triphosphate and sodium silicate, is mixed in a mixer with powdery sodium triphosphate and afterwards sprayed with an alkylbenzene sulphonate (ABS).

One may agree to the Appellants' statement that in particular experiment No. 1 in document (5) describes a washing powder which comprises the same compounds mentioned in Claim 1 of the patent in suit and, apparently, according to the data given in the patent in suit in connection with some typical properties of the washing powder like dynamic flow rate, bulk density and water-solubility, has properties comparable in their quality to those washing powders obtainable by the claimed process. This cannot detract, however, from the fact that the difference between the process of Example 1 of document (5) and the process as claimed is that the synthetic aluminosilicate is mixed in a last step to the firstly prepared mixture. According to the Appellants' statement the reason for this process is that the synthetic aluminosilicate should not be dried frequently. Seen in combination with the process of document (1), however, a skilled person could not easily arrive at the claimed process, because there the aluminosilicate is firstly mixed with the other components after their spray-drying; secondly, this mixture is sprayed with a liquid

binder and only thereafter the complete composition is dried in a last step. As pointed out above the Board is convinced that the advantage of the latter process is that the very finely powdered aluminosilicate can be quantitatively absorbed. The sequence of steps in the claimed process represents clearly a non-obvious modification of the one disclosed in Example 1 of document (5) and any combination thereof with either document (1) or (2) does not lead to the solution claimed in the patent in suit. Since the process of document (1) relates anyhow to the preparation of two separate products which finally are mixed in a dry condition because of the similarity of the particle sizes, the Board cannot see how a combination of the teaching of the mentioned documents could lead to a modification of either one or the other process which would result in the claimed process.

The disclosure of documents (3) and (4) does not go beyond that of document (5) discussed above in detail. It is certainly true that there are many cases where the admixture of the synthetic aluminosilicate is done after completion of the actual spray mixing process and that this may be advantageous, but it is left entirely open in both documents which particular working method should be recommended since this depends on the technical preconditions, the formulation of the detergent, and the nature of the other solid components. This means actually that these documents present merely the problem and leave open the solution. There is not the slightest hint in said documents about the concrete technical steps to be taken in order to solve the problem, i.e. to provide a suitable alternative for the process known from document (1).

- 5.5 It follows from the preceding paragraphs that none of the documents considered contains a hint towards the possibility to provide an alternative to the known

processes for preparing washing powders containing synthetic aluminosilicates such that, when compared to the known processes, the result is actually a process of reduced complexity - in fact, a "simple way", as emphasised by the Appellants. These documents do not suggest to a skilled person to look for a solution of the existing problem by modification of one of the processes disclosed in the discussed prior art documents. With regard to the closest prior art, i.e. document (1), the Board cannot agree to the submissions by the Appellants that the proposed solution there leads in an obvious manner to the less complex solution proposed in Claim 1 of the patent in suit. A "simple" solution is, in the Board's opinion, not necessarily an obvious solution. On the contrary, it may be an indication for the presence of an inventive step if in the prior art only such processes are disclosed which are cumbersome, expensive, time-consuming, etc., i.e. which bear certain disadvantages and, although the problem to be solved apparently was not new and actually solved in more complex ways, the state of the art is nevertheless provided with a surprisingly "simple" solution (see T 9/86, OJ EPO 1988, 12; T 229/85, OJ EPO 1987, 237; T 106/84, OJ EPO 1985, 132).

- 5.6 The Board did not object to the admission of late-filed documents (3) to (5) into the proceedings because prima facie it could not be excluded that one of these documents in combination with the teaching of document (1) could have led in an obvious way to the process of the patent in suit. Only after having thoroughly studied the newly submitted documents and subsequent discussion of the matter during oral proceedings, the Board has come to the conclusion that the disclosure of these documents, as set out above, neither as such nor in combination with one of the documents (1) or (2) leads in an obvious way to the process as claimed in the patent in suit.

5.7 Thus, the claimed process of the patent in suit according to the main request involves an inventive step. The same applies to dependent Claims 2 to 4 which concern particular embodiments of the process according to the main claim.

Consequently, there are no grounds which prejudice the maintenance of the patent in the form as granted. Under these circumstances, there is no need to consider Respondent's auxiliary request.

6. Requests by the parties for reimbursement of the appeal fee (Appellants) and apportionment of costs (Respondents) were withdrawn during oral proceedings. The Board can see no reason which would justify to take up these matters of its own motion.

Order

For these reasons, it is decided that:

The appeal is dismissed.

The Registrar:

The Chairman:

M. Beer

A. Nuss

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