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**Datasheet for the decision
of 23 October 2015**

Case Number: T 1649/12 - 3.3.06

Application Number: 01938063.3

Publication Number: 1274827

IPC: C11D11/00, C11D1/38, C11D1/88

Language of the proceedings: EN

Title of invention:
GRANULAR DETERGENT COMPONENT AND PROCESS FOR ITS PREPARATION

Patent Proprietor:
Unilever PLC / Unilever N.V.

Opponent:
Henkel AG & Co. KGaA

Headword:
Granular detergent component/Unilever

Relevant legal provisions:
EPC Art. 52(1), 54, 56, 83
RPBA Art. 12(4), 13(3)

Keyword:

Sufficiency of disclosure - (yes)

Late-filed argument - admitted (no) - new line of arguments
presented at the oral proceedings

Novelty - (yes) - Features not disclosed in claimed
combination

Inventive step - (yes) - non-obvious solution

Decisions cited:

Catchword:



Beschwerdekammern
Boards of Appeal
Chambres de recours

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Case Number: T 1649/12 - 3.3.06

D E C I S I O N
of Technical Board of Appeal 3.3.06
of 23 October 2015

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Decision under appeal: **Interlocutory decision of the Opposition**
Division of the European Patent Office posted on
30 July 2012 concerning maintenance of the
European Patent No. 1274827 in amended form.

Composition of the Board:

Chairman B. Czech
Members: G. Santavicca
 C. Heath

Summary of Facts and Submissions

I. The appeal by the Opponent lies from the interlocutory decision of the Opposition Division concerning maintenance of European Patent No. 1 274 827 in amended form.

II. The patent had been opposed in its entirety on the grounds of Articles 100 (a) and (b) EPC. The evidence cited during the opposition proceedings includes the following documents:

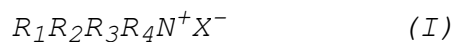
D1a: GB 1 557 568 A;
D2: DE 4 127 323 A1;
D3: WO 98/53037 A1;
D4: EP 0 714 976 A1;
D5: DE 2 235 891 A; and
D6: WO 98/14556 A1.

III. In the decision under appeal, the Opposition Division held that the patent in the amended version with the claims according to the Second Auxiliary Request filed with letter dated 22 January 2009 and a description adapted thereto met the requirements of the EPC.

IV. Claim 1 according to the Second Auxiliary Request held allowable by the Opposition Division reads as follows:

"1. A process for the preparation of a granular detergent component comprising

a) at least 20wt% of cationic surfactant which is a water-soluble quaternary ammonium salt of the general Formula I



wherein R_1 is a C_8 - C_{18} hydrocarbon group, optionally interrupted with a heteroatom or an

ester or amide group; each of R_2 , R_3 and R_4 (which may be the same or different) is a C_1 - C_3 alkyl or substituted alkyl group; and X is a solubilising anion,

b) an inorganic carrier material,

the process being characterised in that it is carried out in a fluidised bed and comprises the steps of

i) fluidising granules of the inorganic carrier material (b) using a drying gas having a temperature within the range of from 100 to 150°C,

ii) spraying an aqueous solution of the surfactant (a) onto the mass of fluidised moving granules of the inorganic carrier material in the presence of the drying gas,

iii) cooling the resulting granular detergent component by mixing in the presence of a gas having a temperature not exceeding 50°C,

the aqueous solution of the surfactant (a) being free of anionic surfactants."

Dependent claims 2-22 concern specific embodiments of the process defined in Claim 1.

V. In its statement setting out the grounds of appeal, the Appellant (Opponent) maintained that the invention was not sufficiently disclosed, that the subject-matter of Claim 1 held allowable by the Opposition Division lacked novelty over document D1a and that it was obvious over D1a taken alone, or over document D3 in combination with any of documents D2 and D5, and even over D5 taken alone. The Appellant also invoked document D6, allegedly illustrating common general knowledge and disclosing the features of several dependent claims.

VI. In its reply, dated 20 February 2013, the Respondent

(Patent Proprietor) maintained that the subject-matter of the claims held allowable by the Opposition Division was sufficiently disclosed, novel over D1a and inventive over D3 and D5.

VII. Oral proceedings were held on 23 October 2015.

The debate first focused on the issue of sufficiency. After deliberation, the Board indicated that it did not intend to take into account those allegations of insufficiency that had not been previously presented in writing and that it considered that the claimed invention was sufficiently disclosed.

Then novelty over document D1a was discussed and, after deliberation, acknowledged by the Board.

As regards inventive step, the parties were first heard regarding the combination of D3 as the closest prior art, taken alone or in combination with D5. The Board indicated that the claimed invention did not appear to be obvious in view of D3, taken alone or in combination with D5, and that D5 did not look like a promising starting point, as it did not disclose a fluidised bed as claimed. Inventive step was then also debated in the light of the combinations D1a with D2, D6 with D3, and D3 with D2. However, after the debate of inventive step in the light of the combination of D3 with D2, the Appellant withdrew the inventive step attacks based on either D1a or D6 as the closest prior art. It did no longer rely on any further documents or combinations thereof.

VIII. Requests

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.

- IX. The arguments of the Appellant of relevance here can be summarised as follows:

Insufficiency of the disclosure

The feature "*solubilising anion*" of Claim 1 was not defined in the patent in suit. The description merely mentioned three examples of solubilising anions [chloride (Cl^-), bromide (Br^-) and methosulfate ion (methyl-sulfate anion, $\text{CH}_3\text{-O-SO}_3^-$)] and no indication was given on how to determine which other anions were "*solubilising*". Since the skilled person could not reproduce the invention across its whole breadth as claimed, the claimed subject-matter of the patent in suit was not sufficiently disclosed.

At the oral proceedings, the Appellant supplemented this objection with further arguments relating to the potential bearing of this feature on the properties of the granules produced.

Lack of novelty

The claimed subject-matter lacked novelty over D1a because it was the result of a single choice within the teaching of D1a, namely that of the cationic surfactant from the list disclosed in D1a, in which two cationic surfactants falling under the Markush formula defined in Claim 1 at issue were mentioned. D1a also disclosed that the cationic surfactant particles were obtainable using a fluidised bed, which technique was also used according to its examples. The slurry with the cationic surfactant mentioned in D1a was aqueous. If there were any doubts

about the aqueous character of the slurry mentioned in D1a in its fluidised bed technique, D3 might be used as evidence therefor. Amounts of more than 20% by weight (up to 50 %) of cationic surfactant were also disclosed in D1a, as well as the use of an inorganic salt as carrier fluidised from an aqueous slurry not containing any anionic surfactants. A cooling step was not expressly mentioned as a process step in D1a, but a granulation in a fluidised bed was usually followed by a cooling step at ambient temperature. Finally, the temperature range of 100-150°C to be used in the fluidised bed according to Claim 1 at issue, was also usual, i.e. a known option, as evidenced e.g. by D2 (page 5, lines 20-22).

Lack of inventive step

D3 addressed the same problems as the patent in suit, i.e. the thermolability of the cationic surfactants, the formation of sticky viscous gels upon contact with water and their effects on dispensing property and high bulk density. Hence, D3 was the closest prior art for assessing inventive step. Like the patent in suit, D3 taught the use of low drying temperatures in order to preserve the cationic surfactant from thermal degradation. The closest embodiments of D3 were those of Examples 1 and 2, in which mixtures containing 20% by weight or more of a cationic surfactant, as required by Claim 1 at issue, were atomized within a co-current spray-drying tower operating at 200°C. The process according to Claim 1 at issue only differed from the one of examples 1 and 2 of D3 in that it expressly required the use of a fluidised bed technique comprising a step of cooling and mixing using a cool gas. There was, however, no particular effect whatsoever attributable to this measure, as apparent from paragraph [0004] of the

patent in suit.

The problem of preventing thermal degradation of the cationic surfactant was already solved by D3, and the prevention of the formation of gel phases could not be part of the formulation of the problem. Hence, the technical problem in the light of D3 could only be seen in the provision of an alternative process for the production of a free-flowing granular detergent component with cationic surfactants of Formula I.

The granular detergent of D3 was free-flowing too and could, alternatively, be obtained by a "spray granulation" process, which was advantageous considering that the temperature could be kept even below 150°C. Thus, the skilled person got a hint from D3 to alternatively use spray-granulation if low operating temperatures were sought-for. The terms spray-granulation and fluidised bed granulation were equivalent and the feature "*fluidised bed*" of Claim 1 at issue meant any "moving set of particles". Hence, a Lödige mixer in which hot air was injected for drying the moving granules was also a "fluidised bed" within the meaning of Claim 1. The feature "*inorganic carrier material*" of Claim 1 merely meant that the carrier contained (not necessarily consisted of) inorganic material. Hence, it could contain other components, as apparent from e.g. Claim 12 of the patent in suit. With reference to the use of a "Lödige mixer", D5 mentioned (page 15, last paragraph) the term "bed" in connection with moving carrier particles, and also mentioned (paragraph bridging pages 17 and 18) that the drying gas moved the particles. Using the Lödige mixer as disclosed in D5 thus represented one alternative way of carrying out "spray-granulation" in a fluidised bed. Implementing the "spray-granulation" suggested in D3 in this manner,

the skilled person would arrive at a process according to Claim 1 at issue in an obvious manner.

If the fluidised bed of granules of inorganic carrier material, the lower drying temperature and the cooling/mixing step using a cool gas were seen as distinguishing features compared to the "spray-granulation" disclosed by D3, the process of Claim 1 was nevertheless obvious in view of a combination of D3 with D2.

D2 disclosed a process for preparing free-flowing granules with a high surfactant loading, which were stable on storage and powder-free. This non-spray-drying process was carried out at relatively low temperature and comprised mixing the detergent solution or paste with an organic or inorganic solid particle and then simultaneously granulating and drying the mixture in a fluidised bed ("Wirbelschicht"). According to Examples 1 and 2 of D2, the temperature of the fluidising air at the bottom of the granulating-drying apparatus used was as low as 100 or 126°C, and the cooling step was carried out at 20°C ("Sichterluft"). The process was suitable also for cationic surfactants. In any case, the skilled person knew from D5 that cationic surfactants were thermolabile and could be granulated and dried at low temperatures. So he would have been motivated to try to apply the process features of D2, thereby arriving at a process as claimed in an obvious manner.

- X. The arguments of the Respondent of relevance here can be summarised as follows:

Sufficiency of the disclosure

In Claim 1, the term "solubilising" merely repeated that the cationic surfactant of Formula I was water-soluble. The patent mentioned three examples of solubilising

anions [chloride (Cl^-), bromide (Br^-) and methosulfate ion (methyl-sulfate anion, $\text{CH}_3\text{-O-SO}_3^-$)]. Its example used a commercially available aqueous solution of a cationic surfactant with chloride anion as solubilising anion. The skilled person, relying merely on common general knowledge, would be able to prepare further cationic surfactant solutions, i.e. find which anions could be used to make water-soluble cationic surfactants. The Appellant had not brought any evidence to the contrary, nor challenged the examples of the patent or the further guidance given therein. The claimed invention was sufficiently disclosed. Those arguments that were presented by the Appellant for the first time at the oral proceedings should be disregarded.

Novelty

D1a was not novelty-destroying. Already the assumption that the slurry mentioned in Examples I, V and VII of D1a was an aqueous solution was contradicted by the further disclosure of D1a, which mentioned prilling, i.e. the spraying of a molten agglomerating product, thus not an aqueous solution as claimed. The amount of quaternary ammonium cationic surfactant in each of Examples I, V and VII of D1a was below the lower limit of 20% by weight defined in Claim 1 at issue. Moreover, the claimed process was to be carried out in a fluidised bed with a drying gas having a temperature of 100 to 150 °C not disclosed in D1a. To arrive at a process falling within the terms of Claim 1 at issue, it was necessary to purposively pick temperature values falling within the claimed ranges. Since a number of choices had to be made to arrive at the claimed process, the claimed process was novel over that disclosed by D1a.

Inventive step

In view of the objectives set out in paragraphs [0003] and [0023] of the patent in suit, D3 was indeed the closest prior art.

As acknowledged in the decision under appeal (Reasons, 22.4), the problem solved over D3 was to provide an alternative process for the production of granules having a high load of cationic surfactants, without the formation of gel phases.

At the oral proceedings, in response to a comment by the Board that the prevention of gel-phase formation appeared to be achieved by D3, the Respondent countered that Examples 1 and 3 of D3 used fillers and did not expressly teach the prevention of gel-phase formation. Reference was made to D4 (comparative compositions A and B) where gel-phase formation was mentioned. But even considering that the formation of gel-phases was prevented in the processes of D3, the claimed process was still to be considered as another non-obvious way of solving the same problem. It was apparent from paragraph [0023] as well as from the examples of the patent in suit that the technical problem invoked was effectively solved by the claimed subject-matter.

Contrary to the argument of the Appellant, the claimed use of fluidised bed spray granulation with cooling/mixing using cool gas did not differ from the co-current spray-drying exemplified in D3 only in the cooling and mixing step using cool gas. In fact, D3 disclosed in examples 1 and 2 a co-current spray-drying process, wherein the aqueous solution was not sprayed onto moving particles of the inorganic carrier, and which was not carried out at a temperature of 100 to 150°C, nor followed by a cooling step as defined. "Spray-granulation" was mentioned only once in D3 (page 4), without any detailed description thereof, and without

any reference to a fluidised bed, let alone with any reference to the prevention of gel-phase formation. From known examples of spray-granulation, e.g. D5, Figure 1, or Example 1 of D4, it was however apparent that the expression "spray-granulation" encompassed the use of different means, such as Lödige Mixers. Example 1 of D4 showed that granulation of an aqueous surfactant composition did not necessarily comprise "spraying". In the apparatuses of D5, granulation took place first, sequentially followed by drying. Not even Figure 2 of D5, showing drying gas injection into the Lödige mixer, taught that the carrier was fluidised solely by the gas, but that this gas injection permitted the drying to be carried out within the same apparatus.

Thus, even if the skilled person started from a process according to Examples 1 and 2 of D3, where the inlet gas temperature was about 200°C, he would find no motivation to use, instead, a fluidised bed process as defined in Claim 1 at issue, in which a drying gas at a temperature of 100-150°C was used to simultaneously fluidise and dry the granules of inorganic carrier onto which the detergent solution was being sprayed, followed by a mixing/cooling step using a a gas at 50°C or less. Hence, the use of a drying gas for fluidising, thus moving, and drying the granules was not obvious from D3, not even taking into account D5.

As regards the further attack based on the combination of D3 with D2, it was to be noted that D3 exemplified only co-current spray-drying of cationic detergent compositions and contained no further details regarding the mentioned "spray-granulation". D2 concerned the granulation of heavy detergent components by simultaneous granulation and drying. Cationic surfactants were only mentioned twice in D2, as D2 was evidently much more concerned with anionic surfactants.

In fact, only the drying of anionic surfactants was exemplified. Since D2 did not address the problem of gel-phase formation in connection with cationic surfactants, the skilled person would not have considered combining the teaching of D3 with that of D2, at least not without hindsight. In any case, even considering combining the teachings of D3 and D2, he would have not have arrived at a process according to Claim 1 in an obvious manner.

Reasons for the Decision

Respondent's Main Request - Claims held allowable by the Opposition Division

Sufficiency of the disclosure

1. Inadmissible new line of argument
 - 1.1 At the oral proceedings, for the first time ever, the Appellant argued that the feature "*solubilising anion*" was not clear, in the sense that it was not apparent under which measuring conditions solubilisation was supposed to occur or be determined. Also, any limiting effect this feature may have on the high detergent loading, gel-phase formation, dynamic flow rate, compressibility and stickiness, was not apparent. In this respect, the Appellant referred
 - to Example 2 of the patent in suit, allegedly showing that the dynamic flow rate and the compressibility were important properties of the granules produced, and
 - to D5 (page 8, last paragraph; page 20, second paragraph), allegedly confirming that the counter-ion of the cationic surfactant influenced the drying speed and the flowability of the granules, as well as that almost

all (organic) surfactants were hygroscopic and, upon drying, could lead to gelatinous masses, so that an appropriate drying method was to be used.

Since the counter-ion of the cationic surfactant had an effect on flowability and compressibility of the granules, and since the patent in suit merely disclosed three examples thereof, without specifying how the solubility was to be determined, the disclosure gave no guidance at all on how to determine whether or not a given anion was "*solubilising*" within the meaning of the patent, not even for the three anions mentioned.

- 1.2 The Respondent objected that this attack was brought forward for the first time during the oral proceedings without substantiation, based on mere assertions, and rather related to clarity, without challenging the reproducibility of the examples or the further guidance given in the patent. This new attack should, therefore, not be admitted.
- 1.3 For the Board, this new line of argument of the Appellant does not represent a reaction to the decision under appeal and amounts to an unexpected amendment to its case within the meaning of Article 13(3) RPBA. It was submitted at a very late stage without any apparent justification and took both the Respondent and the Board by surprise. Moreover, it raised questions never dealt with before, which could not be considered without an adjournment of the oral proceedings.
- 1.4 Therefore, the Board decided not to admit this new line of argument into the proceedings and, hence, to disregard its substance (Articles 12(4) and 13(3) RPBA).
2. According to the objection under Article 100(b) EPC raised by the Appellant in writing, the skilled person

would not know which anions, beyond those exemplified in the patent in suit (chloride, bromide, methosulfate), could be used as "*solubilising anion*" within the meaning of Claim 1, since this term was not defined in the patent in suit. Hence, the skilled person would not be able to carry out the claimed invention across the whole breadth of Claim 1.

- 2.1 The Board firstly notes that this objection, which in fact concerns the reach of the term "*solubilising*", expressing the function to be exerted by the anion, appears to relate to the issue of clarity rather than insufficiency. At the oral proceedings the Board pointed out that the same term was used in D1a (page 5, lines 16-18) and D4 (page 4, lines 26 to 28), but the Appellant, prompted by the Board, had no further comments to make in this respect.

Hence, the Board accepts the argument brought forward by the Respondent that the feature "*solubilising*" merely confirms the water-soluble character of the quaternary ammonium surfactant to be used in the claimed process.

- 2.2 Moreover, the patent in suit expressly discloses three examples of solubilising anions, i.e. chloride, bromide, methosulfate.

- 2.2.1 The use of the first of them (chloride) as "*solubilising*" anion was known before the priority date of the patent in suit. This is apparent from e.g. D3, a document referred to in the application as filed, on which the patent in suit was granted. In fact, D3 (page 4, line 15) generally discloses water-soluble quaternary ammonium salt with a counterion X^- , and specifically illustrates the use of salts with chloride counterions in its examples (page 8, lines 12 to 13).

- 2.2.2 As regards the other two anions disclosed in the patent in suit, the Appellant objected that the invention was insufficiently disclosed in respect thereof. However, it invoked prior art actually making apparent that these anions were also known as counter-anions of water-soluble quaternary ammonium salts used as surfactants. In this respect, attention is further directed to D1a, page 5 lines 16 to 18, or D4, e.g. page 4, lines 27 to - 28 and 53 to 55, both mentioning *inter alia* bromide and methosulfate.
- 2.2.3 The Appellant has not proven by means of suitable evidence that, as regards the use of the anions specifically mentioned in the patent in suit, the claimed process cannot be reproduced.
- 2.2.4 Neither did the Appellant substantiate by verifiable facts that the skilled person would not be able to choose "other solubilising" anions (i.e. anions being suitable for providing water solubility to the ammonium salt making up the cationic surfactant) than the ones specifically mentioned in the patent in suit. In this connection, the Board observes that other anions than the three disclosed in the patent in suit had been known and used for the purpose, as shown proven by the prior art invoked by the Appellant, e.g. D1a and D4. As a case in point, iodide (another halide, like chloride and bromide mentioned in the patent in suit), acetate or sulfate, amide, hydroxyde or nitrate anions were known as counterions of water -soluble ammonium surfactant salts at the priority date of the patent in suit, as mentioned in the prior art invoked (D1a, page 5, lines 15 to 18; D4, page 4, lines 27 to 28).
- 2.2.5 The Board thus concludes that the Appellant did not discharge the burden of proof, resting on its side, to

show that the skilled person would not be able to carry out the invention across the full ambit of Claim 1 without undue burden.

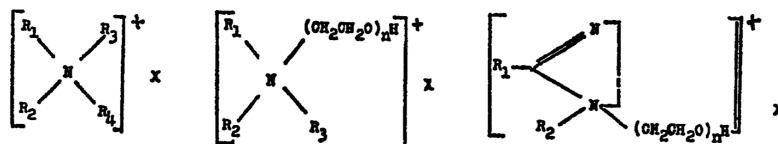
2.2.6 In the Board's judgement, the claimed process is thus sufficiently disclosed (Article 83 EPC).

Novelty

3. D1a (Claim 1) discloses a granular laundry composition comprising an agglomerate of:

- (a) from 1 to 50% by weight of the agglomerate of a quaternary ammonium surface active agent, and
- (b) from 10 to 90% by weight of the agglomerate of an organic acid precursor as activator for a persalt bleaching agent.

3.1 The quaternary ammonium surface active agent has one of the following three general formulae (see Claim 3):



in which R₁ is a straight or branched chain aliphatic with from 8 to 22 carbon atoms;

R₂ is a straight or branched chain aliphatic group with from 8 to 22 carbon atoms or a lower alkyl group with from 1 to 4 carbon atoms, or an aryl or arylalkyl group;

R₃ and R₄ are lower alkyl groups with from 1 to 4 carbon atoms; n is a number between 1 and 15; and X is an equivalent amount of a water-soluble salt-forming anion, such as (see page 5, lines 16-18) halide, e.g. chloride, bromide and iodide, or such as sulphate, acetate and methosulphate.

It is apparent that many of the compounds defined by the first of the three general formulae of D1a fall within Formula I of Claim 1 at issue (III, *supra*).

- 3.2 The agglomerate of D1a can additionally comprise an inorganic carrier salt (see Claim 7 and page 6, lines 38-41).
- 3.3 As regards the preparation of this agglomerate, D1a (page 6, lines 47-50) discloses, as one alternative, the use of a fluidised bed technique, which involves atomizing a slurry comprising the quaternary ammonium salt onto a fluidised bed of the inorganic carrier salt particles.
- 3.4 This fluidised bed technique is illustrated in Examples I, V and VII of D1a (page 9, table and lines 4 to 7). The laundry compositions according to said examples were prepared by atomizing a slurry containing, as cationic surfactant, 10% by weight of either ditallowdimethylammoniumchloride (Examples I and VII) or 1-hydroxyethyl-1-methyl-2-heptadecyl-imidazoliumchloride (Example V).
- 3.5 It was in dispute whether D1a discloses that the illustrated slurry is an aqueous solution of the cationic surfactant, as required by Claim 1 at issue. The Board, considering in particular page 6, lines 43 to 61, more particularly lines 47 to 50, and page 9, table and lines 4 to 8, holds that D1a does not directly and unambiguously disclose an aqueous solution of the cationic surfactant. In this respect, D1a cannot be supplemented by the teaching of D3, as invoked by the Appellant.
- 3.6 It is undisputed that none of the quaternary ammonium

compounds illustrated in Examples I, V and VII of D1a falls under the definition according to Formula I of Claim 1 at issue, which requires the presence of (only) one long-chain hydrocarbon group. Indeed, the compound used in Examples I and VII contains two tallow groups, i.e. two long-chain alkyl groups derived from fatty acids, whilst the compound of Example V has a heterocyclic structure.

In fact, in support of its novelty objection, the Appellant referred to two specific compounds mentioned in the description of D1a (page 5, lines 19-29) but only within a relatively long list of examples of suitable quaternary ammonium salt surfactants, namely trimethylstearylammoniumbromide and cetyltrimethylammoniumchloride (both falling under formula I of Claim 1 at issue).

- 3.7 Moreover, the amount of ammonium salt surfactant (10% by weight) incorporated in the agglomerated detergent component ("particulate laundry compositions") produced according to said examples I, V and VII of D1a (i.e. by means of a fluidised bed technique) is less than the minimum of 20% by weight required according to Claim 1 at issue.

Regarding the amount of cationic surfactant to be used according to D1a, the Appellant pointed out the upper limit of 50% by weight generally disclosed on page 6, lines 53 to 61. For the Appellant, the feature "*at least 20% by weight*" as claimed was thus at least implicitly disclosed in D1a in combination with a fluidised bed technique as generally referred to on page 6, lines 47 to 50, thereof.

- 3.8 The Board has strong reservations in this respect but

also notes that the description of the fluidised bed technique exemplified in D1a (Examples I, V and VII) mentions none of the following features of Claim 1 at issue:

- i) the use of an aqueous slurry of the cationic surfactant;
 - ii) the use of a fluidising drying gas having a temperature within the range of from 100°C to 150°C;
- nor,
- ii) the use of a cooling step wherein a gas having a temperature not exceeding 50°C is mixed with the resulting granular detergent.

3.9 Hence, however obvious or usual the measures i) to iii) might be, a number of choices (at least in terms of the cationic surfactant used, its minimum amount in weight-%; the specific fluidised bed technique used for granulation/drying and the drying and cooling temperatures used) must be made within the much broader disclosure of D1a in order to arrive at a process with all the features of Claim 1 in combination. Hence, for the Board, absent any hint in this particular direction, there is no direct and unambiguous disclosure of a process falling within the terms of Claim 1 at issue.

3.10 Therefore, the novelty objection based on D1a raised against Claim 1 at issue is not conclusive. In the Board's judgement, the process according to Claim 1, is thus novel over D1a (Articles 52(1) and 54 (1)(2) EPC).

Inventive step

4. The invention

The patent in suit (see paragraph [0001]) relates to a process for the preparation of a granular detergent

component containing a cationic surfactant, which is suitable for incorporation into particulate laundry detergent compositions.

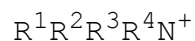
5. The closest prior art

5.1 At the oral proceedings before the Board, it was common ground between the parties that D3 represented the closest prior art for the assessment of inventive step.

5.2 Considering the similarities between the patent in suit and D3 in terms of the technical problems addressed and of the features of the processes disclosed, as indicated below, the Board has no reason to take a different stance.

5.2.1 Indeed, D3 (see page 2, first and second full paragraphs) addresses the problem of providing a process for incorporating cationic surfactants, provided in the form of aqueous solutions, into free-flowing, high bulk density granules whilst preventing the degradation of the cationic surfactants, especially those with alkyl chains of C₁₀ or less, due to high temperatures. D3 also addresses the providing of detergent granule compositions comprising cationic surfactants and displaying good dispensing properties when used as a component of a laundry product. Moreover, D3 (Claim 9) discloses granular detergent compositions with a relatively high surfactant loading, such granules being suitable for incorporation into "compact" or "concentrated" laundry powders. Furthermore, D3 (page 4, first full paragraph), like the patent in suit (paragraph [0009]) addresses the sensitivity to heat of cationic surfactants, i.e. the risk of their degradation/decomposition at temperatures above 150°C.

5.2.2 D3 discloses generally (Claim 1) a process for the preparation of a granular detergent composition or component comprising a step of drying an aqueous solution or dispersion, the aqueous solution or dispersion comprising a water-soluble cationic surfactant, wherein the drying step is carried out using a drying gas, and characterised in that the drying gas has a maximum temperature of less than 250 C. According to Claims 5 and 8 of D3, the water-soluble cationic surfactant used in the process is a quaternary ammonium salt having the general formula



wherein R^1 is a C_6 to C_{10} alkyl chain and R^2 , R^3 , and R^4 are C_1 to C_7 alkyl or hydroxy alkyl chains.

The water-soluble quaternary ammonium salt has as counter-(an)ion X^- (page 4, text line 15).

In the examples of D3 (page 8, lines 12 to 13) X^- stands for a chloride anion.

Moreover, D3 (Claims 6 and 9) discloses granular detergent composition or component so-obtained which comprise from 15 to 40%, preferably from 25 to 40%, by weight of water-soluble cationic surfactant. D3 also discloses the incorporation of layered or, preferably, amorphous water-soluble silicates into the surfactant solution (Claim 3) to be dried, as well as (Claims 6 to 9) granular detergent compositions comprising silicates.

5.2.3 More particularly, the Board accepts that the process of Examples 1 and 2 of D3 represents the most appropriate starting point for assessing inventive step, as submitted by the Appellant, in view of the following:

Examples 1 and 2 of D3 (see page 8) disclose a process for preparing granular detergent compositions meeting the compositional criteria of Claim 1 at issue, since comprising more than 20 weight-% of a C_8 - C_{10} dimethyl

hydroxyethylammoniumchloride (i.e. a cationic surfactant falling under "*Formula (I)*") and sodium silicate (i.e. a material qualifying as "*inorganic carrier*").

The process comprises mixing an aqueous solution of quaternary ammonium salt with aqueous solution of sodium silicate to form a "homogeneous single phase mixture", which is then atomized in a co-current spray-drying tower, having an air inlet temperature of about 200°C. The hot granules are collected at the bottom of the spray-drying tower and allowed to cool to ambient temperature. The resulting granules are free-flowing. These examples disclose the production of detergent components, to be included in a finished detergent composition (see the paragraph bridging pages 6 and 7, first sentence). The aqueous slurry sprayed does not contain anionic surfactants.

- 5.2.4 Further, the Board observes that D3 too addresses (see page 1, penultimate and last sentence of the last full paragraph) the problem of preventing the formation of sticky and viscous gels upon contact with water. As apparent from a comparison of Examples 1 to 3 with Comparative Example 4 (comprising also anionic surfactant) of D3 (page 8): Granular compositions obtained in Examples 1 to 3 are free-flowing, i.e. non sticky. It thus appears that the avoidance of undesirable gel formation is inherently achieved in a manner comparable to the one of the patent in suit, namely:
- without using anionic surfactants;
 - by rapidly evaporating the water part of the cationic surfactant solution using a drying gas at a relatively low temperature.
- As regards the argument of the Respondent that Examples 1 and 3 used fillers and did not expressly teach the prevention of gel phase formation, the Board notes that

Example 2 does not use fillers and that it is expressly stated in D3 (page 9, lines 1 to 2) that the granules obtained from Example 2, like those of Examples 1 and 3, are free-flowing, i.e. non sticky.

The technical problem

6. At the oral proceedings before the Board, the Respondent maintained that in the light of the closest prior art (D3/examples 1 and 2) the technical problem consisted in providing an alternative process for the production of granules having a high load of quaternary ammonium salt of Formula (I) without the formation of gel phases during the process.

The solution

7. The patent in suit as in the amended version at issue proposes to solve this problem by a process for the preparation of a granular detergent component comprising at least 20 wt% of a cationic surfactant, which is a water-soluble quaternary ammonium salt of the general Formula I as set out in Claim 1 at issue, which is characterised in particular in that it is "*carried out in a fluidised bed*" and comprises "*(i) fluidising granules of ... inorganic carrier material ... using a drying gas having a temperature within the range of from 100 to 150°C,*
(ii) spraying an aqueous solution of the surfactant ... onto the mass of fluidised moving granules of the inorganic carrier material in the presence of the drying gas,
(iii) cooling the resulting granular detergent component by mixing in the presence of a gas having a temperature not exceeding 50°C".

8. The success of the solution
 - 8.1 Examples 1 to 11 of the patent in suit illustrate the claimed process for the preparation of detergent components and indicate some properties of the products so-obtained.
 - 8.2 According to paragraphs [0023] and [0031] of the patent in suit, the formation of gel phases of intermediate concentration upon drying was avoided without including special ingredients such as anionic surfactants for that purpose as suggested by e.g. WO 96/17042 acknowledged in paragraph [0005] of the patent in suit. The claimed method is stated to prevent the formation of gel phases because the large amount of water associated with the cationic surfactant is rapidly driven off upon contact with the carrier granules, without thermally affecting the cationic surfactant.
 - 8.3 The Board is thus satisfied that the technical problem posed is effectively solved, i.e. that the claimed process leads to granular detergent components comprising a high load of cationic surfactant without gel-phase formation, the so-obtained granules thus being free-flowing and suitable for being incorporated into particulate laundry compositions. This was not in dispute.

Obviousness

9. It remains to be decided whether the claimed solution was obvious for the skilled person starting from the closest prior art D3 and aiming to solve the problem posed, having regard to common general knowledge and the teachings of the prior art relied upon by the Appellant.

9.1 D3 taken alone

9.1.1 The specific processes according to the closest prior art (D3/examples 1 and 2) do not comprise spraying an aqueous solution of the surfactant onto inorganic carrier granules fluidised under the operating conditions defined in Claim 1 at issue. Instead, the examples of D3 essentially disclose co-current spray-drying of a solution of the quaternary ammonium salt surfactant and sodium silicate.

9.1.2 As pointed out by the Appellant, D3 (page 4, first full paragraph, line 7) also discloses the possibility of using "spray-granulation" as an alternative way of drying the aqueous solution of the cationic surfactant. This alternative method is stated to be advantageous since the maximum temperature can be kept below 200°, preferably below 150°C, thereby reducing the risk of surfactant degradation (page 1, third full paragraph).

However, D3 contains no further details concerning the "spray-granulation" method generally mentioned. In particular, it is not apparent from D3 that a non-spray-drying process such as a fluidised bed is meant. The Appellant alleged, without providing supporting evidence, that spray-granulation implied the use of a fluidised bed. It did not however provide evidence potentially supplementing the teaching of D3 in terms of operating conditions to be used in such fluidised bed or regarding the use of granules of inorganic carrier.

9.1.3 Hence, for the Board, D3 taken alone does not provide a hint towards a process with all the combined features of Claim 1 at issue. The Board comes to this conclusion irrespective of whether examples 1 and 2 (co-current spray drying) or the "spray-granulation" process

disclosed more generally in D3 (page 4) are taken as the starting point (alternative approach of the Appellant presented during oral proceedings).

9.2 Document D5

9.2.1 D5 is also concerned with the drying of aqueous solutions of heat labile surfactants (D5, page 1, first paragraph). Although this document also mentions cationic surfactants (page 8, second full paragraph) such as cetyl-trimethyl-ammonium-bromide (page 9, third last line), drying of solutions of cationic surfactants is neither claimed (D5, claim 2) nor exemplified therein. The prevention of gel phases is not addressed either. For the Board, D5 is thus not the most appropriate starting point for assessing inventive step.

9.2.2 D5 (Claim 1) teaches mixing an aqueous solution of the surfactant with a particulate solid sorbent material and removing moisture from the sorbent particles at a temperature which does not degrade the surfactant. The mixing preferably takes place in a closed mixer such as a Lödige mixer in which the mixture is continuously in turbulent motion. The aqueous solution is sprayed onto the sorbent particles and the drying is achieved by blowing hot gas through the mixer during its operation (see Claim 6, Figure 2 and the examples of D5).

The drying gas is injected into the apparatus during operation of the mixer, but the movement of the particles is assured by mechanical parts (Claim 6; Page 6, last full sentence; Figure 2). Although some passages of D5 invoked by the Appellant refer to a bed (page 15, last sentence) and to the use of gas to dry and move the sorbent particles (page 17, last paragraph: "zur Bewegung des Sorbens und der Tensidlösung im

Mischgerät"), D5 does not disclose a bed fluidised by the drying gas, as required by Claim 1 at issue.

9.2.3 It is apparent from the foregoing analysis that even taking into account the disclosure D5 to supplement the teaching of D3 (in respect of "spray-granulation"), and to modify the process of to the closest prior art D3/ examples 1 and 2 accordingly, would not lead the skilled person to a process falling within the ambit of Claim 1 at issue in an obvious manner.

9.3 D3 taken in combination with D2.

9.3.1 D2 (Claim 1) discloses a process for the production of washing- and cleaning-active surfactant granules with densities above 500 g/l. The latter are prepared by granulation of a composition comprising a surfactant component and a liquid non-surface-active component. The composition, having a liquid to paste-like form under normal pressure at temperatures of 20 to 40°C is simultaneously granulated and dried, optionally with admixture of an organic or inorganic solid component.

9.3.2 According to D2, said process may also be carried out using cationic surfactants (page 3, line 11; Claim 2), but this document focuses (see examples and page 3, lines 12 to 13) essentially on the use of surfactants or surfactant mixtures selected from the group consisting of anionic surfactants (page 3, line 42 *et seq.*) and nonionic surfactants (page 4, line 7 *et seq.*), i.e. with surfactants not necessarily prone to form gel phases upon drying.

9.3.3 The process of D2 (Claim 8), in which the surfactant-containing composition (see 9.3.1, *supra*) is dried and, at the same time, totally or partially freed from the

non-surface-active liquid component, is carried out in a fluidised bed, batchwise or continuously. According to the examples the fluidisation medium used is air. The solid component (Claim 14) may *inter alia* be a carrier (Claim 14), i.e sodium carbonate (Examples). The surfactant-containing composition can be introduced into the fluidised bed through a single nozzle or through several nozzles (Claim 9). The fluidising air has a temperature between 80 and 400°C at the base plate, a temperature between 60 and 120°C at a distance of about 5 cm from the base plate, and an exit temperature between 60 and 120°C. Lower temperatures are only mentioned in connection with the air fed to the classifying apparatus ("Sichter"), which is not described in more detail (Claim 12; Page 5, lines 6 to 12; Examples).

- 9.3.4 More generally, D2 (paragraph bridging pages 4 and 5) also discloses that the process may be carried out using any apparatus suitable for providing simultaneous granulation and drying.
- 9.3.5 For the Board, it emanates from the foregoing analysis that the skilled person starting from D3 and opting for the "spray granulation" alternative is not induced by D2 to implement the latter in the specific manner defined in features i) to iii) of Claim 1 at issue, if only because D2 does not address the temperature sensitivity of cationic surfactants.
- 9.3.6 A process according to Claim 1 would thus not be obvious to the skilled person starting from D3 and being aware of D2.

9.4 Documents D1a and D6

9.4.1 For the Board, these documents do not disclose any more relevant information pointing towards the claimed solution of the technical problem. Since at the oral proceedings, the Appellant expressly no longer relied upon any of D1a and D6 in support of its inventive step attacks, more detailed reasons need not be given in this respect.

10. In the board's judgement, the subject-matter of Claim 1 at issue thus involves an inventive step (Articles 52(1) and 56 EPC).

Conclusion

11. None of the objections raised by the Appellant prejudices the maintenance of the patent in the amended form held allowable by the Opposition Division.

Order

For these reasons it is decided that:

The appeal is dismissed.

The Registrar:

The Chairman:



D. Magliano

B. Czech

Decision electronically authenticated