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D E C I S I O N
of 15 March 2002

Case Number: T 0334/98 - 3.3.6

Application Number: 89307452.6

Publication Number: 0352135

IPC: C11D 11/00

Language of the proceedings: EN

Title of invention:
Detergent compositions and process for preparing them

Patentee:
UNILEVER PLC, et al

Opponent:
Henkel Kommanditgesellschaft auf Aktien
PROCTER & GAMBLE EUROPEAN TECHNICAL CENTER N.V.
Kao Corporation

Headword:
high bulk density/UNILEVER

Relevant legal provisions:
EPC Art. 56, 123

Keyword:
"Main request and auxiliary request 3 (withdrawn) - no basis for amendment"
"Auxiliary requests 5 and 7 (not allowable) - Article 123"
"Auxiliary requests 1, 2, 4, 6, (novelty-yes); inventive step (no) - solution suggested in the prior art"

Decisions cited:
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Catchword:
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Case Number: T 0334/98 - 3.3.6

D E C I S I O N
of the Technical Board of Appeal 3.3.6
of 15 March 2002

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Decision under appeal: **Decision of the Opposition Division of the European Patent Office posted 29 January 2002 revoking European patent No. 0 352 135 pursuant to Article 102(1) EPC.**

Composition of the Board:

Chairman: P. Krasa
Members: G. N. C. Raths
C. Rennie-Smith

Summary of Facts and Submissions

I. This appeal is from the Opposition's Division decision revoking European patent No. 0 352 135.

Claim 1 of the patent as granted was identical to Claim 1 of the main request before the Opposition Division. Claim 1 read:

"1. A process for the preparation of a granular detergent composition or component having a bulk density of at least 650 g/litre, which process includes the step of neutralising a liquid acid precursor of an anionic surfactant with a solid water-soluble alkaline inorganic material, the process being characterised by the steps of:

(i) fluidising a particulate solid water-soluble alkaline inorganic material in an amount in excess of that required for neutralisation, optionally in admixture with one or more other particulate solids, in a high-speed mixer/granulator having both a stirring action and a cutting action;

(ii) gradually adding the acid precursor to the high-speed mixer/granulator while maintaining a temperature not higher than 55°C, whereby neutralisation of the acid precursor by the water-soluble alkaline inorganic material occurs while the mixture remains in particulate form;

(iii) granulating the mixture in the high-speed mixer/granulator, in the presence of a liquid binder, whereby a granular detergent composition

or component having a bulk density of at least 650 g/litre is formed."

II. Three oppositions were filed, all based on lack of inventive step (Articles 100(a), 56 EPC), the opposition of respondent III (opponent 03) being additionally based on lack of novelty (Articles 100(a), 54 EPC). The notices of opposition cited, inter alia, the following documents:

(1B) GB-A-1 369 269;

(2) A.S.Davidsohn, B. Milwidsky "Synthetic detergents", Longman Scientific & Technical, 7th edition.;

(6) A. Davidsohn, "Spray drying and dry neutralizsation of powdered detergents", Journal American Oil Chemists' Society, January 1978, volume 55, 134 to 40;

(15) G. Jakobi, A. Löhr, "Detergents and textile Washing, Principles and Practice", VCH, Weilheim, 1987.

III. The proprietors filed eight sets of claims as a main request and seven auxiliary requests. In its decision the Opposition Division held that the subject-matter of Claim 1 of the main request lacked novelty over document (1B) and that the subject-matter of the auxiliary requests lacked an inventive step, in particular in view of documents (2),(6) and (15).

IV. The patent proprietors (appellants) filed an appeal against this decision.

V. As a reaction to objections raised by the appellants (letter of 8 June 1998, page 3, paragraphs 1 to 4) against comparative data filed by respondent III, respondent III submitted test protocols with its letters dated 22 December 1998 and 10 December 1999 in order to prove that the compositions of the examples of document (1B) had a bulk density higher than 650 g/l.

VI. With their letter dated 13 February 2002, the appellants replaced the requests then on file by a new main request and seven auxiliary requests (which are only reproduced below to the extent necessary for the purposes of this decision):

VI.1 Main request

Claim 1 differed from Claim 1 as granted in that in step (iii) the passage " a liquid binder, wherein" was replaced by "a liquid binder present in an amount from 3 to 8 wt% of the composition whereby".

Claim 8 differed from Claim 1 as granted in that, the passage "comprising either from 5 to 35 wt% of non-soap detergent active material consisting at least partially of anionic surfactant together with from 15 to 45 wt% (anhydrous basis) of crystalline or amorphous alkali metal aluminosilicate, or from 12 to 70 t% of non-soap detergent active material consisting at least partially of anionic surfactant together with 20 to 50 wt% of sodium tripolyphosphate, and" was inserted between "A process for the preparation of a granular detergent composition or component" and "having a bulk density".

VI.2 First auxiliary request

The set of Claims of the first auxiliary request differed from the set of claims of the main request in that the passage "or from 12 to 70 wt% of non-soap detergent active material consisting at least partially of nonionic surfactant together with 20 to 50 wt% of sodium tripolyphosphate," was deleted in claim 8 of the first auxiliary request.

VI.3 Second auxiliary request

The set of claims of the second auxiliary request differed from the set of claims of the first auxiliary request in that claim 8 read as follows:

"8. A process as claimed in any preceding claim, characterised in that the solids present in step (i) comprise sodium carbonate in admixture with one or more detergency builders selected from crystalline and amorphous metal aluminosilicates, alkali metal phosphate, and mixtures thereof."

VI.4 Third auxiliary request

Claim 1 of the third auxiliary request was identical to Claim 8 of the main request.

VI.5 Fourth auxiliary request

Claim 1 of the fourth auxiliary request differed from Claim 1 of the main request in that the passage "comprising from 5 to 35 wt% of non-soap detergent active material consisting at least partially of anionic surfactant together with from 15 to 45 wt% (anhydrous basis) of crystalline or amorphous alkali metal aluminosilicate, and" was inserted between

"A process for the preparation of a granular detergent composition or component" and "having a bulk density".

VI.6 Fifth auxiliary request

Claim 1 of the fifth auxiliary request differed from Claim 1 of the fourth auxiliary request in that "5" was replaced by "10".

VI.7 Sixth auxiliary request

Claim 1 of the sixth auxiliary request differed from Claim 1 of the main request in that

- the passage "containing (a) from 5 to 35 wt% of non-soap detergent-active material consisting at least partially of anionic surfactant, (b) from 15 to 45 wt% (anhydrous basis) of crystalline or amorphous alkali metal aluminosilicate and" was inserted between "A process for the preparation of a granular detergent composition or component" and "having a bulk density";

- the passage "comprising crystalline or amorphous alkali metal aluminosilicate detergency builder" was inserted between "solids" and "in a high-speed mixer/granulator".

VI.8 Seventh auxiliary request

Claim 1 of the seventh auxiliary request differed from Claim 1 of the sixth auxiliary request in that "5" was replaced by "10" under (a).

VII. Oral proceedings took place on 15 March 2002.

The appellants' arguments, in writing and at the oral proceedings, were in summary as follows:

- The goal of the patent in suit was to obtain a non-sticky, free-flowing powder detergent, being of granular form and having a certain particle size limitation. The skilled person considering document (1B) would not replace tripolyphosphate with zeolite because that document taught to keep the water content low in order to avoid, for example, tackiness and lumping (page 4, lines 84 to 90); zeolite comprising bound water would release water when the temperature is increased. The temperature reached by the process according to document (1B) was between 50 to 110°C or even higher (page 1, lines 76 to 80), whereas the patent in suit required a temperature below 55°C. An amount of 3 to 8 wt% of binder, in particular of water, was a specific amount allowing the goal set in the patent in suit to be rached (letter of 8 June 1998, page 2 and paragraph bridging pages 5 and 6).

- Processes disclosed by document (2) may either lead to a product in the form of a dough, but not in granular form, or to a product having a bulk density lower than 650 g/l, ie lower than the value required by claim 1 (letter of 8 June 1998, paragraph bridging pages 3 and 4, and 3rd complete paragraph on page 4).

- The skilled person considering document (2) had no incentive to replace tripolyphosphate by aluminosilicate; document (2) did not disclose a Lödige type mixer.

- Processes disclosed by document (6) may lead to a product having a bulk density of 600 to 900 g/l, but this product would be mixed with lightweight powder from a spray-drying process, the final product being sticky (letter of 8 June 1998, page 4, 5th complete paragraph).

For these reasons, so the appellants argued, the conclusions reached by the Opposition Division were wrong.

VIII. The respondents disputed these submissions in writing and at the oral proceedings.

They argued that in all the requests Claim 1 did not mention a specific particle size distribution. The different detergent components and their amounts as well as the process steps were either known, or would be obvious, from the disclosures of documents (1B), (2) and (15).

IX. At the beginning of the oral proceedings held on 15 March 2002, the appellants, could not, in response to a request from the Board, indicate a basis for the passage "from 12 to 70 t% of non-soap detergent active material consisting at least partially of anionic surfactant together with 20 to 50 wt% of sodium tripolyphosphate" in Claim 8 of the main request and in Claim 1 of the third auxiliary request.

The appellants thereupon withdrew the main and third auxiliary requests.

X. The appellants requested that the decision under appeal be set aside and that the patent be maintained

according to the first, second, fourth fifth, sixth or seventh auxiliary request (the main and third auxiliary requests having been withdrawn during the oral proceedings under IX above).

The respondents requested that the appeal be dismissed.

XI. At the end of the oral proceedings the Chairman announced the decision of the Board.

Reasons for the Decision

1. *Article 123 EPC*

Claim 1 of the fifth auxiliary request differed, inter alia, from Claim 1 as originally filed in that the passage "comprising from 10 to 35 wt% of non-soap detergent active material consisting at least partially of anionic surfactant together with from 15 to 45 wt% (anhydrous basis) of crystalline or amorphous alkali metal aluminosilicate, and" was inserted between "A process for the preparation of a granular detergent composition or component" and "having a bulk density".

The application as originally filed read "from 5 to 35 wt% of non-soap detergent active material consisting at least partially of anionic surfactant together with from 15 to 45 wt% (anhydrous basis) of crystalline or amorphous alkali metal aluminosilicate".

As the change of the lower limit of the range from "5" to "10" had no basis in the application as originally disclosed, Claim 1 of the fifth auxiliary request contravened Article 123(2) EPC, as did Claim 1 of the

seventh auxiliary request.

The sets of claims in the fifth and seventh auxiliary requests were therefore not allowable.

The Board is satisfied that the sets of claims in the first, second, fourth and sixth auxiliary requests meet the requirements of Article 123 EPC.

2. *Articles 54, 83 and 84 EPC*

The Board is also satisfied that the requirements of these Articles of the EPC are met by the first, second, fourth and sixth requests. No objections to these requests under Article 54 EPC were raised by the respondents.

Since these requests fail for other reasons, no further arguments need be given.

3. *Article 56 EPC*

3.1 According to the patent in suit, the technical problem to be solved was to produce detergent powders and detergent powder components of high bulk density and small particle size (patent in suit, page 3, lines 52 to 55). The process included the step of neutralizing a liquid acid precursor "at relatively low temperatures".

3.2 A method for neutralization of detergent acid was disclosed by document (1B). The process temperature was from 50°C to 110°C, 50°C being below 55°C as specified in the patent in suit. A neutralized linear dodecyl benzene sulphonate, sodium salt, was prepared by dry neutralization in a high shear mixer of the Lödige type

and the product obtained had a particle size distribution satisfying the granular form requirement of the patent in suit. The test reports of respondent III enclosed with its letters of 22 December 1998 and 10 December 1999 reproduced the examples of document (1B); it was shown that bulk densities higher than 650 g/l were obtained.

However, the Board cannot agree that document (1B) should be seen as the closest prior art for the evaluation of inventive step since it did not explicitly disclose the bulk density.

3.3 Document (2) concerned synthetic detergents. It referred to a method of neutralization and absorption. The process depended on the utilization of a non neutralized alkylbenzene sulphonic acid which was neutralised with soda ash. There was no limit to the amount of active material that could be incorporated into the powder. This document is of interest because dry neutralization was addressed but it did not disclose high bulk densities for the products obtained.

3.4 Detergent powders having a high bulk density, ie from 600 to 900 g/l, were disclosed by document (6). "The process may be carried out in such a manner that powders either with a granular or a finer structure are produced. The process is flexible enough to produce powders in a wide range of particle size." (page 140, left hand column, lines 1 to 4 from the bottom). The process addressed in document (6) was a dry neutralization process consisting of a "dispersion system which distributes the active matter onto the detergent builders. This very homogeneous dispersion of the surfactants onto the builders is accomplished in a

mixer which has especially designed plough type horizontal mixing blades rotating at high speed and passing very close to the mixer walls. This design guarantees very efficient blending of all the components. In addition, ultra high speed rotating disintegrators are inserted to prevent any lump formation." (page 139, right hand column, paragraph 3, lines 4 to 13). Fixation of the active matter components is accomplished by mechanical disintegration of the solid builder material onto which the active matter components are added by means of a rather simple dispersion system.

3.5 The Board considers document (6) to be the closest prior art for evaluating inventive step, since it deals with the liquid bulk densities of detergent powders as does the patent in suit and the skilled person looking for a process leading to a high bulk density would consult this document.

3.6 Thus, in the light of document (6), the problem underlying the patent in suit was to provide an alternative process for producing detergent powders of high bulk density and of granular form.

3.7 In view of examples 1 to 29 of the patent in suit, the Board accepts that this technical problem was credibly solved by neutralizing a liquid acid precursor of an anionic surfactant (a linear alkylbenzene sulphonate) with solid water-soluble alkaline inorganic material (ie the builder) in a Fukae or a Sapphire mixer. According to the description a Lödige mixer may also be used (page 5, lines 51 to 52). The process temperature should not be higher than 55°C (page 6, lines 36 to 41).

3.8 The question remains to be decided whether a process run at a temperature not higher than 55°C and comprising the use of a mixer having stirring and cutting means was obvious or not.

3.9 A mixer having stirring and cutting means was disclosed by document (2). This document was concerned with the production of finished detergents made of alkyl benzene sulphonic acid neutralized with soda ash. It taught: "Recently special disintegration high-speed mixers, fitted with special rotating knives have been successfully introduced by the authors to produce these dry-neutralized powders in one single step, dispensing with ageing and grinding. This type of mixer is produced by the German firm Lödige (Paderborn) and their licenses in the UK and the USA." (page 208, last but one line to page 209, line 3). And further: "If a relatively high amount of alkyl benzene sulphonic acid is being used, the mixer should be fitted with a water-cooling jacket to dissipate the heat of reaction."(page 209, paragraph 2).

The skilled person was thus aware that it was feasible to run the dry neutralization process of document (6) in a Lödige mixer having a stirring action and a cutting action. Further, there was a clear hint that the temperature should be controlled. Since there was no evidence on file showing that the temperature of 55°C, beyond which the process should not be run, was crucial, the limitation to that temperature in Claim 1 was arbitrary.

3.10 It follows that the subject-matter of Claim 1 of the first auxiliary request lacks an inventive step, as does Claim 1 of the second auxiliary request which is

identical.

3.11 The subject-matter of Claim 1 of the fourth auxiliary request differed from Claim 1 as originally filed in that the passage "from 5 to 35 wt% of non-soap detergent active material consisting at least partially of anionic surfactant together with from 15 to 45 wt% (anhydrous basis) of crystalline or amorphous alkali metal aluminosilicate, and" was inserted between "A process for the preparation of a granular detergent composition or component" and "and, having a bulk density".

The incorporation of components such as aluminosilicate, and the indication of their amounts represent further embodiments usual in the field of detergents (see document (15), table 25); as these components did not produce any effects, they cannot serve to produce an inventive step.

Hence the subject-matter of Claim 1 of the fourth auxiliary request does not meet the requirements of Article 56 EPC.

3.12 The subject-matter of Claim 1 of the sixth auxiliary request differed from Claim 1 of the fourth auxiliary request in that the passage "present in an amount from 3 to 8 wt% of the composition" was inserted between "binder" and "whereby" under (iii).

The test results submitted by respondent III (see Appendix 17, to its letter dated 22 December 1988) proved that the compositions having binder concentrations such as 6.43 wt% or 7.90 wt%, falling within the claimed range of 3 to 8 wt%, did not lead to

unexpected results compared with compositions having concentrations outside the claimed range, such as 2.75 wt%, and 8.80 wt%. Thus the range of binder of 3 to 8 wt% was an arbitrary range.

It follows that the subject-matter of Claim 1 of the sixth auxiliary request did not involve an inventive step.

3.13 Since the independent Claim 1 in any of the first, second, fourth and sixth auxiliary requests does not meet the requirements of Article 56 EPC, none of the sets of claims in those requests is allowable.

Order

For these reasons it is decided that:

The appeal is dismissed.

The Registrar: The Chairman:

G.Rauh P.Krasa