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D E C I S I O N
of 3 May 2002

Case Number: T 0773/98 - 3.3.6

Application Number: 92917958.8

Publication Number: 0598817

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Language of the proceedings: EN

Title of invention:

Process for making granular automatic dishwashing detergent

Patentee:

THE PROCTER & GAMBLE COMPANY

Opponent:

Henkel KGaA

Headword:

ADW detergent composition/PROCTER

Relevant legal provisions:

EPC Art. 56

Keyword:

"Inventive step (yes)"

"Comparative examples in the patent in suit accepted as representing the closest prior art demonstrating the effect attributable to the essential distinguishing feature"

Decisions cited:

T 0181/82, T 0035/85, T 0197/86

Catchword:

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Case Number: T 0773/98 - 3.3.6

D E C I S I O N
of the Technical Board of Appeal 3.3.6
of 3 May 2002

Appellant: Henkel KGaA
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Representative: Semrau, Dr.
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Respondent: THE PROCTER & GAMBLE COMPANY
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Representative: Lawrence, Peter Robin Broughton
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Decision under appeal: Decision of the Opposition Division of the
European Patent Office posted 21 July 1998
rejecting the opposition filed against European
patent No. 0 598 817 pursuant to Article 102(2)
EPC.

Composition of the Board:

Chairman: P. Krasa
Members: G. Dischinger-Höppler
C. Rennie-Smith

Summary of Facts and Submissions

I. This appeal is from the decision of the Opposition Division to reject the opposition and to maintain European patent No. 0 598 817 on the basis of 14 claims as granted, the only independent claim reading:

"1. A process for making a granular automatic dishwashing detergent composition, comprising:

- (a) incorporating alkali metal silicate particles, preferably hydrous silicate, with from 5% to 30%, by weight of the silicate, of low foaming nonionic surfactant with a melting point between 77°F (25°C) and 140°F (60°C), said nonionic surfactant being in a liquid form;
- (b) forming, preferably by agglomerating, spray drying or dry mixing, base granules which are free of alkali metal silicate, said base granules comprising from 5% to 100%, by weight of the base granules of detergency builder; and
- (c) admixing said silicate particles of step (a) with said base granules of step (b) in a weight ratio of between 1:20 and 10:1."

II. A notice of opposition based on lack of novelty and lack of inventive step (Articles 100(a), 54 and 56 EPC), cited the following documents:

- (1) US-A-3 920 568 and
- (2) EP-B-0 010 247.

- III. In its decision, the Opposition Division found that the subject-matter of the claims as granted was novel and inventive in view of the two documents cited by the Opponent. In particular, it was held that neither of these documents was concerned with dishwashing detergent compositions or with the problem underlying the patent in suit which consisted in improving the solubility of silicate contained in the detergent composition. These documents offered no hint to the skilled person of the claimed solution of this problem.
- IV. With a letter dated 23 April 2002, the Respondent (Proprietor) filed, as its first, third and fourth auxiliary requests, fair copies of the three auxiliary requests previously set out in its letter dated 22 May 1998 and, as its second auxiliary request, the text of an additional new request.
- V. Oral proceedings were held before the Appeal Board on 3 May 2002, in the course of which the Board drew attention to document
- (3) US-A-4 379 069,
- referred to in the patent in suit (page 2, lines 15 to 18 and 43 to 46) and indicated that this might represent a closer prior art than the above two citations.
- VI. The Appellant (Opponent), whilst not abandoning its previous submissions on the basis of documents (1) and (2), agreed that document (3) could be taken as a suitable starting point for assessing inventive step. Its arguments can be summarised as follows:

- Document (3) related to the same problem of avoiding formation of insoluble silicate residues on dinnerware due to degradation of silicates in the detergent composition and, to that effect, contained the overall teaching not to use the silicate in an acidic environment possibly created in the presence of aqueous nonionics.
- Document (3) did not, however, contain any reservation against an admixture of nonionics and silicates at a high pH.
- The claimed incorporation of nonionics in molten form into the silicate particles was obvious in the light of document (1) where this was already suggested in order to prevent bleeding of the absorbed nonionic during storage.

For the assessment of inventive step over documents (1) and (2), the Appellant submitted that

- in addition to the residue problem, the claimed process aimed at the solution of a further problem, namely how to incorporate high amounts of nonionic surfactant into the granular concentrate;
- a skilled person trying to solve such problems in a dishwashing detergent composition would consider documents (1) and (2), which belonged to the related technical field of textile detergent compositions, and would thereby arrive at the claimed process since document (2) solved the problem of avoiding silicate residues by incorporating nonionics into the silicate and document (1) solved the problem of incorporating

high amounts of nonionics into the composition, in both cases, in the same manner as the patent in suit.

VII. The Respondent's arguments can be summarised as follows:

- The claimed process aimed at the production of a detergent composition for automatic dishwashing which did not produce insoluble silicate residues on the dishes. It was intended to incorporate into the composition not high amounts of nonionics but only such amounts as were usually sufficient for the purpose of dishwashing.
- In dishwashing processes even very low levels of insolubles were unacceptable, whereas in the washing of laundry, as e.g. in document (2), large amounts of insolubles such as zeolites were deliberately added.
- Further, document (1) did not address the problem underlying the patent in suit but instead related to a high surfactant textile detergent system.
- Therefore, documents (1) and (2) were wholly unrelated to the problem underlying the patent in suit and no one concerned with that problem would refer to those documents.
- Document (3) was concerned with dishwashing compositions but indicated that the order of addition of the ingredients was critical and that the silicate should be added last. Therefore, this document taught away from the claimed subject-

matter.

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

The Respondent requested as its main request that the appeal be dismissed or alternatively that the patent be maintained on the basis of one of the four auxiliary requests filed with its letter of 23 April 2002.

Reasons for the Decision

Main Request

Lack of novelty being no longer in dispute, the only issue to be decided is whether or not the claimed process is based on an inventive step.

1. *Technical background*

1.1 The patent in suit relates to a process for making a granular automatic dishwashing (ADW) detergent composition exhibiting improved solubility (page 2, lines 5 to 6).

According to the patent in suit it was known that alkali metal silicate polymerises into insoluble matter when exposed to less alkaline environments and that residues from ADW detergents that remain on the dishware after washing are predominantly those silicates (page 2, lines 20 to 23). With reference to document (3) it is stated that such a less alkaline environment was thought to be created by the addition of nonionic surfactants. A high level thereof was,

however, desirable both for its cleaning function and for the "water sheeting" effect (page 2, lines 34 to 46).

- 1.2 Hence, the patent in suit seeks to provide a process by which an ADW detergent composition with improved solubility and sufficient nonionic surfactant can be made (page 2, lines 47 to 50).

2. *Closest prior art*

- 2.1 In contrast to documents (1) and (2), which both relate to laundry detergent compositions, document (3) is concerned with ADW detergent compositions (document (1), column 1, lines 10 to 12; document (2), page 2, lines 1 to 3; document (3), column 1, lines 21 to 33).

In the Board's opinion, document (3) is more suitable as a starting point for the assessment of inventive step than document (1) or (2), not only for the uncontested reason that compositions for textile washing differ from those used for dish washing, but also for the reason invoked by the Respondent that insolubles are much less tolerable in dishwashing detergent solutions than in laundry detergent solutions. The latter is based on the Respondent's convincing argument that laundry detergent compositions often contain large amounts of deliberately added insoluble compounds such as aluminosilicate builder material (see for instance document (2), page 4, lines 15 to 28) which credibly suggests that textiles are much less susceptible to the deposition of silicate residues than is dinnerware.

- 2.2 Document (3) not only relates to ADW detergent

compositions but also mentions the problem of insoluble residues which, if due to the degradation of silicates, adversely affects the aesthetic appearance as well as the stability of the surfaces of dishes (see column 1, lines 21 to 33). In order to reduce these disadvantages, document (3) suggests preparing a silicate free alkaline blend containing the builder, the surfactant, the alkaline agent and filler, if any, and mixing that blend with solid alkali metal silicate and a chlorine donor (see column 1, lines 53 to 63 and Examples I and II in combination with Tables 7 and 8). The preferred surfactant in document (3) is a nonionic type from the "Pluronic" series of ethylene oxide-propylene oxide block polymers (column 2, lines 57 to 59 and Table 3) of which series, in the patent in suit, certain compounds are said to be also suitable for the purpose of the claimed invention (page 4, lines 29 to 31). It is emphasized that the order of raw material addition is critical (column 1, lines 64 to 66) in the sense that the solid silicate should be added only in the presence of an alkaline agent such as soda ash in order to minimize direct contact between the nonionics/H₂O premix and the solid silicates (column 1, line 68 to column 2, line 7; column 6, lines 36 to 45). Three different sequences (A, B and C) of addition of the various components in amounts as covered by the claimed subject-matter are compared in Table 3. In sequence A a dry charge of builder, sodium silicate and sodium sulfate is mixed with nonionic surfactant (together with water in a liquid premix if the builder is anhydrous sodium tripolyphosphate). This product is thoroughly blended with soda ash and a chlorine donor. Sequence B differs therefrom in that the sodium silicate and the chlorine donor are only mixed in at the end of the sequence, whilst in sequence C the dry

charge to be mixed with the nonionic already comprises the soda ash (see also column 5, lines 36 to 42, Table 3 and column 6, lines 54 to 59). In Table 4 it is shown that the solubility rating for sequence A, where silicate and nonionic/H₂O are contacted in the absence of soda ash, is much worse than for sequences B and C which both add the nonionic/H₂O to a premix comprising soda ash.

According to document (3), up to 6% by weight of nonionic surfactant is incorporated in the final detergent composition (Claim 1 and Table 8).

3. *Technical problem*

3.1 It follows from the above that a suggestion of how to make an ADW detergent composition with improved solubility was already disclosed in document (3).

Further, the added amount of up to 6% by weight of nonionic surfactant according to document (3) fulfills the "high level" requirement within the meaning of the patent in suit where about 4.5% by weight of the final composition is sufficient (see Tables 5, 7 and 9 in combination with Claim 1).

3.2 As a consequence, the technical problem to be solved as against document (3) is how to provide an alternative process for preparing a soluble granular ADW detergent containing a sufficiently high level of nonionic surfactant level.

4. *Solution of the problem*

4.1 The solution to this problem, as suggested in Claim 1

of the patent in suit, is that all the alkali metal silicate is, in particulate form, to be premixed with a nonionic surfactant having a melting point between 25°C and 60°C whilst being in a liquid form to incorporate it into the particles (see also page 7, lines 50 to 53). To this effect, the nonionic is melted by heating it preferably to temperatures of between 60°C and 93.3°C, followed by applying the thus liquefied surfactant onto and into the silicate particles via several mixers and finally cooling the mixture (page 7, line 53 to page 8, line 11). This product is admixed with builder containing base granules in the suitable weight ratio.

- 4.2 Although no direct comparison between the products of the claimed process and the products of the examples of document (3) is on file, it is shown in Examples III to V of the patent in suit that the products of the claimed process exhibit improved solubility over products obtained in a manner similar to that exemplified in document (3) (in particular Example II) by first admixing the heated nonionic surfactant with a premix of sodium carbonate and sodium sulfate and adding sodium silicate last.

Whilst document (3) mentions neither heating of the nonionic surfactant nor that the nonionic surfactant as such must be added in liquid form, it is nevertheless allowable, if not necessary, to modify in the present case prior art embodiments for comparison in order to provide a variant wherein the effect attributable to the essential distinguishing features of the invention is more clearly demonstrated (see T 197/86, OJ EPO 1989, 371; T 35/85 of 16 December 1986, not published in the OJ EPO; T 181/82, OJ EPO 1984, 401). Thus, the

comparative examples given in the patent in suit, which are a modification of the examples of document (3) but fall within the general teaching of the latter, are accepted by the Board as showing the influence of the order of addition of raw material in the process in issue. In view of the examples and comparative examples of the patent in suit, it is credible that the above mentioned technical problem as against document(3) has been solved by the claimed process.

5. *Inventive step*

It remains to be decided whether, in view of the available prior art documents, it would have been obvious to someone skilled in the art to solve this problem by the means claimed.

5.1 The Appellant argued that it was clear from Table 4 of document (3) that the best overall solubility rating was achieved by applying sequence C of Table 3 in which a sodium silicate containing dry charge was premixed with the nonionic detergent in the presence of soda ash in order to avoid formation of insoluble silica under otherwise acidic conditions created by the aqueous nonionic detergent. Thus, the essential teaching as set out in column 6, line 46 to column 7, line 7 and Table 5 of document (3) was not to use silicate in an acidic environment.

Thus, the argument continued, it was evident from document (3) that there existed no prejudice against a direct contacting of alkali silicate with nonionic detergent as long as the environment was not acidic.

5.2 In fact, according to the teaching of document (3) it

is essential to provide alkaline protection of the alkali metal silicate as a physical barrier in order to minimize direct contact between the acidic nonionic/H₂O premix and the solid silicate (column 6, lines 41 to 45 and 54 to 59). In contrast, the claimed process does not require such protection. Instead, the nonionic is directly mixed with the silicate in molten form without any further additives (see 4.1 above). This implies, in the Board's opinion, the absence of aqueous conditions in the sense of deliberately added water. Water being present in step (a) of the claimed process only as water of hydration (see page 3, lines 30 to 32), one might conclude that pH conditions would not pose any problem at all.

- 5.3 However, document (3) in Example II (see in particular Table 8) also contains an embodiment where no water is added with the nonionic detergent. Nevertheless, even in this case, the sodium silicate is added last after the addition of sodium citrate builder to an admixture of the nonionic detergent with an initial premix of filler (sodium sulfate) and sodium carbonate.

The Board therefore concludes that document (3) contains no hint that, as an alternative, the nonionic tenside should simply be mixed with the alkali metal silicate, i.e. in the absence of sodium carbonate.

- 5.4 The only prior art cited by the Appellant against the patent in suit were documents (1) and (2) which both relate to laundry detergents. Whilst accepting that those skilled in the art would consider this particular technical field as related to the technical field of dishwashing detergents, the Board holds for the following reasons that these documents do not teach any

solution to the present technical problem (see 3.2 above).

- 5.5 The Board concurs with the Appellant's opinion that it is not crucial to the assessment of inventive step of the claimed subject-matter that document (1) is silent about any content of builder material. Indeed, it is considered to be essential for that purpose that document (1) does not address the problem of preventing residues caused by alkali metal silicates. In fact, document (1) seeks to overcome the problem of bleeding of surfactant absorbed by silicates during storage of a laundry detergent composition (see column 1, lines 62 to 66). It proposes to solve this problem by using a very particular type of sodium silicate for absorbing liquid nonionic surfactants in a weight ratio of nonionic surfactant to sodium silicate carrier of 0.4:1 to 1.2:1 and admixing, based on the weight of the total detergent composition, 30 to 80% of this product with 20 to 70% of spray dried granules containing a conventional anionic surfactant (column 1, lines 44 to 48, column 2, lines 21 to 29 and 32 to 49, column 3, lines 3 to 14, column 8, lines 3 to 7 and column 11, lines 9 to 56).

Thus, whilst suggesting a similar process for making a non-bleeding laundry detergent composition, document (1) does not contain any hint that this process could be applied - by adapting the respective ingredients (see 2.1 above) - for making ADW detergent compositions with the required solubility.

- 5.6 Concerning document (2), the Board agrees with the Appellant insofar as this document is concerned with the problem of avoiding the formation of residues of

alkali metal silicates. However, unlike the patent in suit, document (2) deals with the cleaning of laundry (page 3, lines 51 to 62) and not of dishware. In view of the differences of the respective substrates and detergent compositions (see 2.1 above), it is immediately questionable whether or not a person skilled in the art would have considered citation (2) when aiming at the solution of the current technical problem (see 3.2 above).

Moreover this document, in solving its technical problem, teaches to the use of a particular kind of water soluble alkali metal silicate (page 4, lines 10 to 14) which is mixed, in an unspecified weight ratio, with a powder obtained by spray drying and containing the aluminosilicate builder and surfactants. It is suggested, but only if a part of the surfactant is a heat-sensitive nonionic tenside, that this may be sprayed onto the sodium silicate instead of being mixed with the builder material (page 6, lines 10 to 42 and page 19, lines 2 to 5). Thus, there is no connection in document (2) between the solubility problem on the one hand and the addition of nonionic surfactant to the alkali metal silicate on the other hand, and therefore no hint to the solution of the technical problem of avoiding formation of silicate residues from ADW detergent compositions on dinnerware as claimed in the patent in suit.

6. The Board therefore concludes that, while the various steps of the claimed process were in principle known, either from document (3) for the same purpose but in a different sequence safeguarding minimization of direct contact between nonionics and solid silicates, or from documents (1) or (2) for a different purpose in a

process for making a different composition, their particular combination according to the process of Claim 1 of the patent in suit in order to obtain an alternative to the process of document (3) was not obvious in view of the prior art documents whether considered individually or in combination.

7. No other result is obtained if one starts from document (1) or (2) as the closest prior art as suggested by the Appellant.

Being uncontested that compositions for dishwashing differ from those used for laundry and in view of the fact that problems with insolubles are of a different order of magnitude (see 2.1 above), the problem solved as against these documents consists in providing a process for making a granular ADW detergent composition which contains nonionic surfactant in an amount sufficient for this purpose and which none the less exhibits the solubility necessary in this field of application. As already indicated, documents (1) and (2) do not give any hint as to the solution of this problem (see 5.5 to 5.6 above). The only document on file which proposes a solution is document (3). This document, however, by its teaching that alkaline material must be present if a nonionic tenside is to be mixed with sodium silicate, teaches away from the claimed process.

8. Accordingly, the Board holds that the process of Claim 1 is based on an inventive step within the meaning of Article 56 EPC.

Dependent Claims 2 to 14, which refer to preferred embodiments of Claim 1, are based on the same inventive

concept and derive their patentability from that of Claim 1.

Auxiliary requests

9. Since the above findings mean that the Respondent's main request is allowable, the auxiliary requests need not be considered.

Order

For these reasons it is decided that:

The appeal is dismissed.

The Registrar:

The Chairman:

G. Rauh

P. Krasa