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D E C I S I O N
of 14 November 2000

Case Number: T 0111/96 - 3.3.6

Application Number: 91903357.1

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Language of the proceedings: EN

Title of invention:
Liquid cleaning products

Patentee:
UNILEVER PLC, et al

Opponent:
PROCTER & GAMBLE EUROPEAN TECHNICAL CENTER N.V.

Headword:
-

Relevant legal provisions:
EPC Art. 56

Keyword:
"Inventive step - no (advantages resulting from a prior art in an obvious manner) "

Decisions cited:
-

Catchword:
-



Case Number: T 0111/96 - 3.3.6

D E C I S I O N
of the Technical Board of Appeal 3.3.6
of 14 November 2000

Appellants:
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Respondent:
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Decision under appeal: Decision of the Opposition Division of the
European Patent Office posted 30 November 1995
revoking European patent No. 0 515 418 pursuant
to Article 102(1) EPC.

Composition of the Board:

Chairman: G. Dischinger-Höppler
Members: G. N. C. Raths
C. Rennie-Smith

Summary of Facts and Submissions

- I. This appeal is from the decision of the Opposition Division to revoke European patent No. 0 515 418. The decision was based on the claims as granted as a main request and on amended claims according to two auxiliary requests.

The only independent claim of the main request reads:

"1. A non-aqueous liquid cleaning composition comprising a non-aqueous organic solvent, particles of solid material dispersed in the solvent, from 0.01-15% by weight of a deflocculant material and a dispersant, wherein the dispersant is a hydrophobically modified silica."

Claim 1 of the first auxiliary request differs therefrom in that the passage "selected from alkanolic acids, alkyl sulfonic acids, aralkyl sulfonic acids and their salts, organic acids, peracids and persulfonic acids, Lewis acids, anionic surfactants and zwitterionic surfactants" is inserted between "deflocculant material" and "and a dispersant".

Claim 1 of the second auxiliary request differs from that of the first auxiliary request by the addition at the very end of the claim of the feature "wherein the composition has a viscosity of less than 2.500 mPas at 21 s⁻¹".

- II. The notice of opposition was based on lack of inventive step. During the opposition proceedings, the Opponent (Respondent) filed inter alia, the following documents:

- (2) GB-A-1 205 711;
- (4) Degussa, Technical Bulletin Pigments No. 18, Special Effects when Using Aerosil R 972 in Coating Systems, 2nd edition, October 1986; and
- (5) GB-A-2 173 224.

III. In its decision, the Opposition Division held that a person skilled in the art would consider hydrophobically modified Aerosil an obvious alternative to hydrophilic, non-modified Aerosil as silica carrier in the compositions of document (2). No unexpected effect due to the use of the hydrophobic silica over hydrophilic silica had been shown.

IV. With their statement of grounds of appeal, the Appellants (Proprietors) filed experimental data in the form of documents (8) to (10) to show the superior flowing properties of compositions according to document (2) containing hydrophobic silica sold under the trademark Sipernat D17 instead of hydrophilic silica sold under the trademark Aerosil 200.

V. Oral proceedings were held before the Appeal Board on 14 November 2000.

VI. The Appellants submitted in essence that

- by the incorporation of a hydrophobically modified silica dispersant, the compositions claimed in the patent in suit solved the problem of setting in non-aqueous liquid detergent products;
- document (2) disclosed non-aqueous built liquid detergents containing hydrophilic silica as inorganic carrier material, but did not hint at any possible use of hydrophobic silica;

- the problem of setting, in particular in connection with the use of silica, was not addressed in any of documents (2), (4) and (5);
- the viscosity values given in document (2) were not comparable with those of the patent in suit and not such as to suggest a solution to the setting problem; and
- document (4) would not have been considered by a skilled person since it related to an entirely different technical field, and document (5) taught away from using silica material.

VII. The Respondent rejected the Appellants' experimental data as irrelevant and presented in essence the following arguments:

- by referring to Aerosil in general as a particularly suitable inorganic carrier, document (2) covered both hydrophilic and hydrophobic silica;
- the problem of setting was mentioned in documents (2) and (4), the latter recommending hydrophobic silica as a solution to this problem;
- document (4) would further have been considered by a person skilled in the art via the disclosure of document (5) that the rheological behaviour of non-aqueous detergents and paints was comparable.

VIII. The Appellants requested that the decision under appeal be set aside and that the patent be maintained as granted or alternatively in accordance with the first or second auxiliary request filed on 14 November 1995.

The Respondent requested that the appeal be dismissed.

Reasons for the Decision

Main Request

The only point at issue is whether or not the claimed subject-matter is based on an inventive step.

1. *Technical background*

The patent in suit relates to liquid non-aqueous cleaning products containing particulate solid materials, such as detergency builders, which require a stabilising system to keep the solids in suspension (page 2, lines 3 to 9).

According to the patent in suit, several attempts have been made to provide solid-suspending properties in non-aqueous liquids, inter alia by using silica dispersants. However, use of these dispersants is said to lead to the "**setting**" of the composition, often resulting in an unacceptably high viscosity. On the other hand, it has been found that non-aqueous liquid detergent compositions which are free from dispersants, in particular from silica-containing dispersants, sometimes suffer from physical instability, i.e. clear layer or phase separation (page 2, lines 10 to 21, Examples I and II), hereinafter referred to as "**settling**".

Hence, the patent in suit seeks to provide a non-aqueous liquid cleaning product wherein the problems of setting and settling are overcome (page 2, lines 22 to 24).

2. *Closest prior art*

Document (2) shows a typical prior art use of silica dispersants in non-aqueous liquid detergents. It pertains to built detergent compositions which can be made by a simple admixture of the components. The essential components of the compositions consist in a liquid detergent active material, an anhydrous inorganic builder salt and an inorganic carrier material (page 1, lines 68 to 79).

As to the settling property, the compositions of document (2) are said to have good stability in that they show no significant phase separation after storage for long periods of time (page 2, lines 82 to 86), e.g. for 2 months (see Examples VIII, IX, XII, XIII and XV).

Concerning setting, the Appellants argued that document (2) taught product viscosities of 10000 mPas and higher (page 3, lines 9 to 11) which indicated that setting was not addressed in document (2) as a problem to be solved, because compositions exhibiting such high viscosities had set.

This argument is not accepted by the Board since document (2) mentions in particular that the compositions must be **pourable**, which means **non-set**, and that for this purpose it is preferred that the viscosity of the compositions should be in the range of 300 to 3000 mPas (300 to 3000 centipoise) (page 3, lines 4 to 9). In contrast, viscosities of 10000 mPas and higher are merely said, without any explanation, to be "often satisfactory".

Consequently, document (2) addresses the same technical problem as in the patent in suit (see under 1. above) and the Board accepts it as a suitable starting point for assessing inventive step and, like the parties, takes this document as the closest prior art.

3. *Technical problem plausibly solved*

According to Claim 1 it is suggested to solve this problem by incorporating 0.01 to 15% by weight of a deflocculant material and a dispersant, the latter being a hydrophobically modified silica, into a non-aqueous organic solvent containing dispersed solid material.

3.1 From the description of the patent in suit it follows that the liquid phase, i.e. the non-aqueous organic solvent, is preferably made up of nonionic surfactants (page 4, lines 15 to 21 and Examples) and the particulate solids are predominantly detergency builders (see under 1. above), in particular alkali metal carbonates and tripolyphosphates (page 5, lines 17 to 42 and Examples). Various inorganic and organic acids and their salts are said to be suitable deflocculant materials, including anionic surfactants, in amounts ranging most preferably from 4 to 10% by weight, based on the final composition (page 5, line 46 to page 6, line 24). An anionic surfactant, namely ABS-acid (i.e. alkylbenzene sulfonic acid) is used in the examples in amounts of 8%, 6% and 3.4% by weight of the total composition respectively.

3.2 Alkali metal carbonate and tripolyphosphate are not only mentioned in the patent in suit but also in document (2) as the preferred inorganic builder salts, here in the sodium form, (page 2, lines 53 to 61 and Examples).

Moreover, document (2) also discloses compositions containing a nonionic surfactant in combination with an anionic surfactant (page 2, lines 33 to 52). According to Examples XII and XIIIa, anionic alkylaryl sulfonic acid or dodecylbenzene sulfonic acid is used in addition to the nonionic surfactant Tergitol[®] 15-S-9 (see Example II) in amounts of 2% and 7% by weight. The compositions of Examples XII and XIIIa, therefore, contain not only the same kind of non-aqueous organic solvent and solid particles, but also the same deflocculant in the same amount as the compositions of the patent in suit.

In document (2), a carrier material is required as a further essential compound of the composition. Suitable carrier materials are light, highly voluminous metal oxides, such as silica, alumina, magnesia and ferric oxide, having an average surface area of from 50 to 500 m²/g, a bulk density of from 10 to 180 g/l and a particle size of from 1 to 100 nm (1 to 100 m μ). Particular emphasis is laid on silicas such as those commercially available under the trademark Aerosil (page 1, line 68 to page 2, line 4).

As agreed by the parties, two kinds of silicas under the trademark Aerosil were on the market in 1968, the year of publication of document (2), namely hydrophilic and hydrophobic silicas. However, hydrophobically modified silica as used according to the patent in suit is not mentioned verbatim in document (2).

The parties further agreed that hydrophobically modified silica is chemically and physically different from hydrophilic or non-modified silica in that it is the product of the reaction of hydrophilic silica with an organic hydrophobing reactant. In this product, the free hydrophilic hydroxy groups (here silanol groups) at the outer surface of silica have, at least

partially, been substituted by hydrophobic groups, such as alkyl groups, which render the surface less hydrophilic (see also patent in suit, page 2, line 50 to page 3, line 5).

In view of these facts, the Board considers that - being aware of the differences between hydrophilic and hydrophobic silica - the authors of document (2) would have explicitly mentioned hydrophobic silica if this was meant to be a possible embodiment of the inorganic carrier material.

As a consequence, the Board concludes that the silica used in Examples XII and XIIIa of document (2) as inorganic carrier material is not hydrophobically modified.

- 3.3 The Appellants submitted experimental data obtained by reworking Examples XII and XIIIa of document (2), once with Aerosil 200 as a commercially available hydrophilic silica and once with the hydrophobically modified silica (Sipernat D17) used according to the patent in suit. The resulting viscosities were measured, after storage for one week at 50°C and after storage for 7 weeks at 25°C, with a "Bolin VOR" viscosimeter. The Appellants argued that - in contrast to the Brookfield method used in document (2) (page 3, lines 32 to 36) - this viscosimeter allowed well-defined shear rate conditions and a direct comparison of the viscosities of non-Newtonian liquids which typically include non-aqueous built liquid detergent compositions.

The experimental results are represented in documents (8) and (9) as a graph showing the measured viscosities at different shear rates. It is evident from these graphs that the compositions obtained with hydrophobic silica have substantially lower viscosities than those with hydrophilic silica.

In addition, in document (10) the Appellants provided photographs of three products in glass bottles - hydrophilic silica, hydrophobic silica and a product containing no silica at all - showing the physical stability (phase separation) of each after three weeks. As can be seen from the photographs, phase separation in the product with hydrophobic silica is increased over that obtained with hydrophilic silica, but clearly lower than in a product without any silica at all. According to the Appellants, phase separation when expressed as a percentage of clear layer formation was 4% for hydrophobic silica, 2% for hydrophilic silica and 16 % for no silica.

The Respondent expressed some doubts concerning the results obtained and the method of measuring the viscosity. In particular, it doubted that Aerosil 200 was representative of the silica used in Examples XII and XIIIa of document (2). However, Aerosil 200 is at least comparable with the silica of said examples in respect of its BET surface area. According to the physico-chemical data of Aerosil 200 given in document (4) (see page 21), the BET surface area is $200 \pm 25 \text{ m}^2/\text{g}$. In comparison, the average BET surface area of the silica used in Examples XII and XIIIa of document (2) is given as $175 \text{ m}^2/\text{g}$ (see page 3, lines 37 to 38 and Example I). Since the Respondent did not

provide any evidence to cast doubt on the accuracy of the Appellants' tests, the Board accepts these tests as comparative experiments and indicative of the effects obtained with the claimed subject-matter in view of the compositions disclosed in document (2).

3.4 In comparison with the compositions according to document (2), documents (8) and (9) show that, by using hydrophobically modified silica, a particular rheological property of the composition is improved, namely its pourability, i.e. the non-setting property after storage for a particular period of time at particular temperature conditions (see above under 3.3). With regard to the settling problem, i.e. the undesired phase separation, no improvement over the prior art is observed, as demonstrated by document (10).

4. It remains, therefore, to be decided whether, in view of the available prior art documents, it was obvious for someone skilled in the art to solve by the means claimed the problem of setting after storage under particular conditions without unduly worsening the settling, i.e. stability of the composition.

4.1 Document (2) does not give any hint as to how the setting (high viscosity) of the non-aqueous liquid detergent composition after storage for some time can be reduced. It does, however, suggest that silicas sold under the trademark Aerosil are suitable for use as inorganic carrier materials (see above under 3.2).

4.2 Information about Aerosil is given in document (4) which is a product information paper, distributed by Degussa AG, concerning the basic characteristics and applications of Aerosil in general and, in particular, the characteristics of hydrophobic Aerosil R 972 in comparison to hydrophilic Aerosil 200 (see page 1). It

discloses on page 2 (middle column) that Aerosil 200 has about 1000 silanol groups per particle, whereas in Aerosil R 972 about 70% of the silanol groups originally present enter into a chemical reaction with dimethyl dichloro silane, thereby reducing the silanol group density so greatly that the original hydrophilic starting material becomes hydrophobic. On page 7, under the heading "3.1 Use as Suspending Agent", reference is made to the well-known anti-settling effect of silicas for pigments and fillers in the coating industry (see middle column). It is further stated that, due to the lower silanol group density, hydrophobic Aerosil is not so strongly agglomerated as Aerosil 200. Therefore, the hydrophobic variant can be dispersed more easily (right-hand column, last 8 lines). On page 12, suggestions are given to explain the suspension behaviour and the thixotropic effect of Aerosil. It is assumed that interparticle hydrogen bonding via the silanol groups takes place, resulting in the formation of a three-dimensional structure (left-hand column). A schematic representation is given in Figure 10 to show that hydrogen bonding between silanol groups of different silica particles is responsible for the formation of aggregates as well as for the formation of a three-dimensional "chickenwire" structure.

Contrary to the Appellants' allegation that document (4) did not mention the setting problem, it is stated explicitly in said Figure 10 that this structure develops "on setting". Therefore, a person skilled in the art realizes from the teaching of document (4) that the presence of silanol groups and the silanol group density is not only responsible for the extent of agglomeration but also for the aggregation of the silica particles into a set structure.

- 4.3 The Appellants argued that a skilled person concerned with the technical field of detergent compositions would not consider document (4), because this document related to the completely different technical field of paints and coatings.
- 4.4 However, the expertise of a person skilled in the art of detergent compositions will, in the Board's opinion, extend to detergent materials in general. Such a person must be assumed to know everything about detergent compositions and the components used therefor, including the physical and chemical properties of such components. In particular when looking to improve a prior art composition, he or she is supposed to consider all available information referring to such compounds, irrelevant of whether the information is directly related to the particular technical field of detergents or not. Given the particular reference in document (2) to Aerosil as a suitable carrier material, a skilled person would, therefore, take account of the information provided in document (4) and learn from it that the rheological behaviour of silica in liquids is dependent on the silanol groups.
- 4.5 For these reasons, the Board is of the opinion that the skilled person would not have needed any further information in order to arrive at the claimed subject-matter. Thus document (5), while it discloses that the rheological behaviour of non-aqueous built liquid detergent compositions can be compared with the rheology of paints (see page 1, lines 29 to 30), need not be considered in this context.

Concerning this document, the Appellants submitted that it warned the skilled person not to use any silica at all and, therefore, taught away from consideration of document (4). In fact, the presence of silica such as Aerosil is not preferred in document (5). However, the

Appellants have overlooked that this warning is based on the particular reason that silica decomposes peroxyacid bleaching agents contained in the compositions as essential ingredients (page 11, lines 33 to 39). Therefore, where peroxyacid stability is not the object to take into account, such a warning would not deter the skilled person from considering a document such as document (4) referring to the rheological behaviour of silica in paints.

5. The Board therefore comes to the conclusion that a person skilled in the art would, for the purpose of improving the setting behaviour, have tried hydrophobically modified silica instead of the hydrophilic silica in the compositions of document (2) because, given the information in document (4), he or she would have expected advantageous rheological effects with respect to setting and/or settling.

In the present case, therefore, the principle applies that no inventive step can be based solely on the presence of a particular improved property of the claimed composition if such an improvement, in this case a particular rheological behaviour, would be derived by a skilled person in an obvious manner from the prior art.

For these reasons, the main request must fail.

Auxiliary requests I and II

- 6.1 As to the amendments made to the claims of the auxiliary requests, the Respondent did not raise any objections under Article 84 or 123 EPC. Since these requests fail for the reasons set out below, there is no need to go into details here.

- 6.2 The amendment made to Claim 1 of auxiliary request I consists in restricting the deflocculant material to particular groups of acids, including the group of anionic surfactants. Considering that an anionic surfactant is already present in the compositions disclosed in the closest prior art (see point 3.2), Claim 1 of auxiliary request I does not add any inventive feature to Claim 1 as granted.
- 6.3 Claim 1 of auxiliary request II adds to Claim 1 of auxiliary request I a restriction with respect to the viscosity of the composition. As is shown in the Appellants' experimental data represented in documents (8) and (9), this last feature merely represents an inherent property resulting from the claimed composition in view of the prior art compositions (see 3.3 above). The viscosity is not, therefore, an additional technical feature further defining the claimed composition and cannot make any difference to the assessment of inventive step.
- 6.4 The same conclusion as drawn for Claim 1 of the main request (see 5 above) therefore applies mutatis mutandis to Claim 1 as worded in each of the two auxiliary requests.

Order

For these reasons it is decided that:

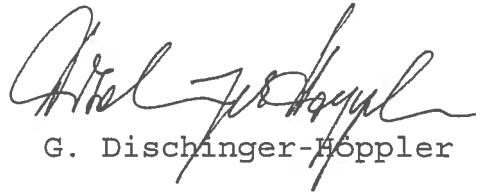
The appeal is dismissed.

The Registrar:



G. Rauh

The Chairman:



G. Dischinger-Hoppler

