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**Datasheet for the decision  
of 24 April 2013**

**Case Number:** T 1337/10 - 3.3.07

**Application Number:** 01954952.6

**Publication Number:** 1313436

**IPC:** A61Q 11/00, A61K 8/25,  
A61K 8/34

**Language of the proceedings:** EN

**Title of invention:**  
Abrasive compositions and methods for making same

**Patent Proprietor:**  
J.M. HUBER CORPORATION

**Opponent:**  
Evonik Degussa GmbH

**Headword:**  
Abrasive compositions/J.M. HUBER CORPORATION

**Relevant legal provisions:**  
EPC Art. 56

**Keyword:**  
"All requests - inventive step (no)"

**Decisions cited:**  
-

**Catchword:**  
-



Case Number: T 1337/10 - 3.3.07

**DECISION**  
of the Technical Board of Appeal 3.3.07  
of 24 April 2013

**Appellant:**  
(Opponent)

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**Decision under appeal:**

Decision of the Opposition Division of the  
European Patent Office posted on 19 April 2010  
rejecting the opposition filed against European  
patent No. 1313436 pursuant to Article 101(2)  
EPC.

**Composition of the Board:**

**Chairman:** J. Riolo  
**Members:** R. Hauss  
M. Tardo-Dino

## Summary of Facts and Submissions

- I. The appeal lies from the decision of the opposition division rejecting the opposition filed against European patent No. 1 313 436.
- II. The patent was granted on the basis of ten claims. Claim 1, which is the only independent claim, reads as follows:

*"1. A method for preparing an abrasive composition, the method comprising the steps of:*

*introducing into a reaction system including a reaction container and a high shear mixing means arranged for treating reaction mixture contents of the reaction container, as the reaction mixture contents, alkali silicate and acid and inter-mixing the reaction mixture thereby to form precipitated silica;*

*withdrawing from the reaction container 5 to 50 volume % per minute of the total reaction mixture, conducting the withdrawn portion through the high shear mixing means and thereafter re-introducing the withdrawn portion into the reaction container;*

*separating the precipitated silica from the reaction mixture with a filter to provide a filter cake;*

*washing the filter cake; and*

*fluidizing the precipitated silica in the filter cake by combining humectant with the precipitated silica, thereby to provide a suspension of abrasive particles containing humectant."*

III. An opposition was filed, opposing the patent in its entirety under Articles 100(a), (b) and (c) EPC on the grounds that the claimed subject-matter lacked inventive step, was not disclosed in a manner sufficiently clear and complete for it to be carried out by a person skilled in the art, and extended beyond the content of the application as filed.

IV. The documents cited in the course of the opposition and appeal proceedings included the following:

D1: WO 97/46485 A1

D4: WO 97/34576 A1.

V. In its decision, the opposition division considered that the wording of claim 1 as granted did not introduce subject-matter extending beyond the content of the application as filed, and that the disclosure of the claimed method was sufficiently clear and complete since the opposed patent described in sufficient detail a way of preparing the desired abrasive composition and also set out a number of alternative process configurations and alternative types of high shear mixing means.

As far as inventive step was concerned, document D4 was regarded as the closest prior art. D4 disclosed a method of preparing precipitated silica which involved employing shearing forces, prior to drying the reaction product. The subject-matter of claim 1 as granted differed from the disclosure of D4 in that the precipitated silica, obtained after filtration as a filter cake, was to be combined with a humectant to yield a suspension of abrasive particles. The data in

example 2/table 3 of the opposed patent showed that rheologically stable liquid abrasive compositions were obtained in that manner, using sorbitol or glycerol as the humectant, without any need for drying or dry milling/comminuting treatments.

The technical problem was defined as the provision of stable liquid abrasive compositions, even in the absence of carboxymethyl cellulose. Although dentifrice compositions including abrasive silica and a polyol humectant were known, the prior art did not disclose or suggest combining a humectant with the filter cake obtained in the preparation of precipitated silica, in order to provide a stable suspension at an intermediate stage of the dentifrice manufacturing process. In consequence, the claimed subject-matter was deemed to involve an inventive step.

VI. The appellant (opponent) lodged an appeal against that decision. In the statement setting out the grounds of appeal, the appellant relied on objections under Article 100(c) EPC concerning added subject-matter and under Article 100(a) EPC concerning lack of inventive step.

VII. With the reply to the statement of the grounds of appeal, dated 15 December 2010, the respondent (patent proprietor) requested that the appeal be dismissed and submitted seven auxiliary requests.

Claim 1 of each of the auxiliary requests corresponds to claim 1 as granted, with the following differences:

Claim 1 of the first auxiliary request contains, instead of the feature "... and inter-mixing the

*reaction mixture thereby to form precipitated silica;"*, the modified feature *"... with inter-mixing thereof to form precipitated silica;"*.

Claim 1 of the second auxiliary request contains, instead of the feature *"withdrawing from the reaction container 5 to 50 volume % per minute of the total reaction mixture, ..."*, the modified feature *"withdrawing from the reaction container 8 to 50 volume % portion per minute of the total volume of the reaction mixture contents of the reaction container, ..."*.

Claim 1 of the third auxiliary request contains the modified feature from claim 1 of the first auxiliary request in combination with the modified feature from claim 1 of the second auxiliary request.

Claim 1 of the fourth auxiliary request contains the following modifications with regard to the filter cake (insertions into the wording of granted claim 1 are marked in bold): *"separating the precipitated silica from the reaction mixture with a filter to provide a **wet** filter cake; washing the **wet** filter cake; and fluidizing the precipitated silica in the **wet** filter cake ..."*.

Claim 1 of the fifth auxiliary request contains the modified feature from claim 1 of the first auxiliary request in combination with the insertions from claim 1 of the fourth auxiliary request.

Claim 1 of the sixth auxiliary request contains the modified feature from claim 1 of the second auxiliary

request in combination with the insertions from claim 1 of the fourth auxiliary request.

Claim 1 of the seventh auxiliary request contains the modified feature from claim 1 of the first auxiliary request in combination with the modified feature from claim 1 of the second auxiliary request and the insertions from claim 1 of the fourth auxiliary request.

In all auxiliary requests, "*a high shear mixing means arranged for treating reaction mixture contents*" is replaced by "*a high shear mixing means **arranging** for treating reaction mixture contents*", which is deemed to be a typing error without consequences for the meaning.

- VIII. In a letter dated 28 February 2011, the appellant raised objections under Article 123(3) EPC against auxiliary requests 1 to 3 and under Article 84 EPC against auxiliary requests 4 to 7.
- IX. Oral proceedings were held before the board on 24 April 2013, during which the issue of inventive step was discussed.
- X. The appellant's arguments can be summarised as follows:

*Main request*

With regard to the claimed scope, the abrasive composition to be prepared by the method of claim 1 was not required to be a liquid suspension. Even if the last mandatory method step defined in the claim produced a suspension of fluidised abrasive particles,

further optional processing steps resulting in other formulation types, such as pasty dentifrice compositions, were not excluded by the wording of the claim.

The closest prior art document D4 disclosed a method of preparing precipitated silica, including a step of washing the silica particles (see page 9, step 8), which involved forming an aqueous suspension. The method defined in claim 1 of the patent in suit differed from the disclosure of D4 solely in the mandatory method step of combining the precipitated silica with humectant, to provide a suspension.

This difference had not been shown by the respondent to provide any particular technical effect over the entire claimed scope. In the absence of any results from comparative testing, the alleged effect of suspension stability had not been credibly shown, nor had it been causally linked to the distinguishing method step of adding a humectant. If suspension stability were to be achieved, it was more likely due to the small particle size of the precipitated silica. Particle size was not, however, a distinguishing feature over the prior art D4. Since the wording of claim 1 did not exclude methods which involved drying or dry-milling of the precipitated silica or the addition of thickeners, inventiveness could not be based on the absence of such treatments.

Without evidence of a surprising technical effect, the objective technical problem was the provision of an alternative method for preparing a suspension of abrasive silica particles.



The claimed method was an obvious solution to that problem in the light of the combined teaching of documents D4 and D1.

D1 disclosed in examples 7 to 9 the preparation of an abrasive composition in the form of a dentifrice, containing precipitated silica and sorbitol in its usual function as a humectant. As acknowledged in the patent in suit, it was conventional practice to introduce the abrasive polishing material to dentifrice compositions either in dry powder form or after re-dispersion. Dispersing the precipitated silica with humectant before introducing it into a dentifrice formulation such as described in example 9 of D1 was therefore a mere routine measure which the skilled person would envisage as a way to solve the technical problem.

*Auxiliary requests 1 to 7*

With regard to the auxiliary requests, the appellant relied on the same arguments as with regard to the main request.

As far as the feature "wet filter cake" appearing in auxiliary requests 4 to 7 was concerned, the appellant held that the expression did not introduce any relevant difference in meaning. Without a defined lower limit for the moisture content, it also encompassed dried filter cakes with some residual moisture; hence, a drying step was not excluded.

XI. The respondent's arguments can be summarised as follows:

*Main request*

The claimed method differed from the disclosure of the closest prior art D4 in the process step of combining precipitated silica obtained as a filter cake with humectant, to provide a liquid suspension.

Since that process step was the final step of the claimed method, claim 1 was to be construed as defining a method which provided, as its end product, a liquid suspension containing silica and humectant. Said suspension was a storable multi-component additive which could be further employed in the preparation of a dentifrice composition. The claimed method did not however encompass the manufacture of a finished dentifrice composition, such as a toothpaste, but only the preparation of the storable liquid suspension pre-mix. It was evident in the light of the description that no other meaning could be intended.

Due to the addition of the humectant, the claimed method provided storage-stable silica suspensions. Evidence of the suspensions' stability against settling had been provided in the form of the test results presented in table 3 of the patent specification. The appellant had failed to present counter-experiments in support of its allegation that no advantageous technical effect was obtained by the distinguishing method step.

As a further advantage, drying and dry-milling of the precipitated silica were avoided, since the suspensions

prepared by the claimed method contained appropriately small particle sizes of silica and were stable in liquid form.

The objective technical problem was the provision of a suspension of precipitated silica which was stable against sedimentation.

The proposed method of preparation was not obvious having regard to the state of the art. None of the cited documents dealt with the problem of providing storage-stable suspensions. Still less did they suggest that stable suspensions could be obtained by adding humectant to a filter cake of precipitated silica.

*Auxiliary requests 1 to 7*

Auxiliary requests 1 to 3 re-introduced wording used in the application as filed, with a view to countering objections under Article 100(c) EPC. The modifications made in claim 1 of each request did not change the respondent's argumentation concerning inventive step, which was the same as for the main request.

Auxiliary requests 4 to 7 corresponded to the main request and auxiliary requests 1 to 3, wherein the absence of a drying step had been further emphasised by indicating in claim 1 of each request that the wet, and therefore non-dried, filter cake was subjected to the humectant fluidisation step.

XII. The appellant requested that the decision under appeal be set aside and that European patent No. 1 313 436 be revoked.

XIII. The respondent requested that the appeal be dismissed or, alternatively, that the patent be maintained on the basis of one of auxiliary requests 1-7 as submitted with the letter dated 15 December 2010.

### **Reasons for the Decision**

1. The appeal is admissible.
2. Inventive step - main request
  - 2.1 Patent in suit
    - 2.1.1 The patent in suit seeks to provide a method for preparing an abrasive composition of precipitated silica, suitable for dentifrice use.
    - 2.1.2 The method proposed in claim 1 of the main request involves forming precipitated silica by acidulation of an alkali silicate and subjecting the reaction mixture to high shear forces. Shearing is achieved by withdrawing a defined percentage per minute of the volume of the reaction mixture from the reaction container, conducting it through a high shear mixing means and re-introducing the withdrawn mixture into the reaction container. According to the patent in suit, the high shear treatment serves to reduce particle size. In this manner, appropriately sized abrasive particles can be obtained (i.e. particles susceptible of providing high cleaning efficacy without undue abrasion), without any need for drying and dry-milling. Such post-treatment is to be avoided because it is

costly and may affect the brightness of the silica abrasive by introducing impurities (see the patent specification, paragraphs [0002], [0004] to [0006], [0010]). Wet milling or screening of the particles is envisaged if very small particle size is desired or if the high shear treatment is not sufficient (see the patent specification, paragraph [0037] and figure 1).

The claimed method further involves separating the precipitated silica from the reaction mixture by filtering, washing the filter-cake and fluidising the precipitated silica in the filter-cake by combining it with a humectant to provide a suspension. According to the patent in suit, the suspensions thus obtained are rheologically stable and resistant to settling and re-agglomeration during transport and storage. They can be employed as pre-mixed components in the preparation of oral cleaning compositions (see the patent specification, paragraphs [0008] to [0010]).

## 2.2 Closest prior art

2.2.1 Document D4 has been regarded as the closest prior art both in the decision under appeal and in the parties' submissions. The board does not see any reason to select a different starting point.

2.2.2 D4 relates to precipitated silicas with improved dentifrice performance characteristics and discloses a method (see claim 14) for preparing precipitated silica from sodium silicate and a mineral acid by an acidulation reaction. Said method involves subjecting the reaction product to shearing forces prior to drying. While high shear treatment is not mandatory, it is

explicitly envisaged in D4 (see page 7, lines 12 to 16). The shear treatment may take place concomitantly with the acidulation reaction, as illustrated in example 1. The reaction mixture is filtered and washed with water to remove salt from the filter cake (see page 9, step 8). The filter cake may be dried to 3 to 10% moisture content (see page 9, step 9). The aqueous suspension and the wet or dried filter cake obtained according to the method of D4 are abrasive compositions of precipitated silica.

Document D4 teaches that the proposed method results in reducing the average particle size of the precipitated silica, which helps avoid undesirable milling treatment (see page 4, lines 15 to 23). Example 1 of D4 describes a method for preparing precipitated silica which involves pumping the reaction medium through a line blender to impart shear forces, said method closely corresponding to the method described in examples 1 and 2 of the patent in suit.

2.3 Difference between the claimed method and the method disclosed in D4

2.3.1 The subject-matter of claim 1 of the patent in suit differs from the disclosure of D4 in the mandatory method step of providing a suspension by combining a humectant with the precipitated silica of the filter cake (feature A), such a step not being disclosed in D4.

2.3.2 Nor does D4 explicitly disclose the withdrawal of 5 to 50 volume % per minute of the reaction mixture from the reaction container, conducting this withdrawn portion through the high-shear mixing means and thereafter

re-introducing it into the reaction chamber (feature B). However, it has not been established that this feature is a distinguishing feature of the claimed method over the disclosure of D4, taking into account that the most preferred shear rate of 4.5 employed in D4 (see page 9, lines 1 to 2) is above the minimum shear rate of 3.0 indicated in the patent in suit (see paragraph [0023] of the patent specification). The shear rate in this case is defined as the number of times the entire reactor contents are recirculated during the reaction time.

2.3.3 The respondent has not contested the above assessment regarding feature B and has based its line of argument in support of inventive step exclusively on feature A.

#### 2.4 Technical problem and solution

2.4.1 According to the respondent's interpretation of claim 1, the distinguishing method step of combining a humectant with the precipitated silica of the filter cake is the final step of the claimed method. It has the technical effect of providing, as the end product of the claimed method, a suspension of silica abrasive which is resistant to settling and which can therefore be stored in liquid form until it is used, e.g. in the preparation of dentifrice formulations. Drying and dry-milling of the silica can be avoided.

2.4.2 In the framework of the problem-and-solution approach employed by the boards for assessing inventive step, an alleged advantage in the form of a technical effect can only be taken into account in the definition of the objective technical problem if said effect is reflected

in the technical features of the claim and is based on a distinguishing feature over the disclosure of the prior art, and if the effect is obtained over the entire scope of the claim.

Hence, in order to determine the objective technical problem, it must be ascertained whether all of these criteria are met by the alleged technical effects.

#### 2.4.3 Stability of the suspension against settling

Claim 1 is directed to a method for preparing an abrasive composition. The method is defined as "comprising", i.e. including, several mandatory method steps or features. The last mandatory step mentioned in the claim requires fluidising the precipitated silica in the filter cake by combining it with a humectant to provide a suspension of abrasive particles containing humectant. However, the open definition of the method does not exclude the possibility that it may comprise subsequent method steps. While the product to be prepared by the claimed method is required to be an abrasive composition (see line 1 of claim 1), it is neither explicitly nor implicitly required to be a liquid suspension as obtained by the humectant fluidisation step. Since the wording of claim 1 is clear and can be understood by the skilled person, there is no reason to consult the description to read further limitations into the claim.

Claim 1 therefore also encompasses embodiments in which the suspension is obtained at an intermediate stage of the method and is further processed.



Furthermore, claim 1 does not contain any feature requiring that the suspension obtained by humectant fluidisation of the precipitated silica in the filter cake should be stored for any length of time.

As a consequence, the alleged benefit of stability of the suspension against settling is not relevant to part of the claimed scope, viz. those embodiments of the claimed method in which the suspension obtained by the humectant fluidisation step is further processed without intermediate storage.

Nor indeed does claim 1 contain any explicit or implicit requirement that the suspension obtained by the humectant fluidisation step be resistant to settling. In particular, no parametric criteria for resistance to settling or to re-agglomeration are defined in the claim. As far as the technical features which may have an impact on settling behaviour are concerned, the claim does not indicate the particle size of the precipitated silica, the nature of the humectant or the ratio of humectant to silica.

Since the alleged technical effect of suspension stability is not reflected in the features of claim 1 and is furthermore not pertinent to the entire claimed scope, it cannot be taken into account in the definition of the objective technical problem.

#### 2.4.4 Avoidance of drying and dry-milling

The wording of claim 1 does not exclude drying or dry-milling of the precipitated silica from the claimed

method. Hence, inventive step cannot be based on the absence of such treatments.

2.4.5 The only result of the distinguishing method step which is obtained over the claimed scope is thus the provision of an abrasive composition of precipitated silica by a method which involves a step of forming a suspension of precipitated silica containing humectant, either at an intermediate stage or as the end product of the claimed method.

2.4.6 The objective technical problem can accordingly be defined as the provision of a further method for preparing an abrasive composition containing precipitated silica.

2.4.7 In the light of the examples described in the patent specification, the board is convinced that the technical problem is solved by the method as defined in claim 1 of the patent in suit.

2.5 Obviousness of the solution

2.5.1 Document D4 seeks to provide precipitated silica for dentifrice use. Its teaching is focused on the manufacturing process of precipitated silica of suitable particle size, by acidulation combined with shear treatment, resulting in a washed filter cake of precipitated silica. The silica may then be dried and milled (see D4: pages 7 to 9). As already mentioned (see paragraph 2.2.2 *supra*), the aqueous suspension and the wet or dried filter cake obtained according to the method of D4 are abrasive compositions of precipitated silica.

2.5.2 As far as the preparation of further abrasive compositions by further methods involving humectant is concerned, the teaching of D4 may be combined with that of document D1, which discloses the preparation of dentifrice compositions comprising precipitated silica and humectant (see D1: claims 17 to 18 and page 6, lines 7 to 10; examples 7 to 9).

According to example 7 of D1, precipitated silica is prepared by an acidulation reaction. After filtration and washing, a suspension of precipitated silica is obtained (example 7) which is then spray dried (example 8). According to example 9 of D1, the spray-dried silica of example 8 is used for preparing a dentifrice composition containing sorbitol as a conventional humectant. The dentifrice composition according to example 9 of D1 is an abrasive composition in the sense of claim 1 of the patent in suit, since it is a composition which contains abrasive silica particles.

While it has not been established whether precipitated silica particles prepared according to D4 can be distinguished from particles prepared according to D1, both documents D1 and D4 envisage dentifrice use of the respective silica particles, which must therefore be considered as equally suitable for use in dentifrice formulations. With the knowledge of the teaching of both D1 and D4, it would be obvious to the skilled person that the composition of example 9 of D1 could be prepared with the equivalent particles obtained according to D4.

2.5.3 Document D1 does not disclose any details of the manufacturing process of the dentifrice composition according to example 9, but merely indicates that the (spray-dried) silica obtained according to example 8 was employed.

As acknowledged in the patent in suit, in a summary of the background art (see paragraph [0002] of the patent specification), the abrasive polishing material has conventionally been introduced to dentifrice compositions in flowable dry powder form, or via re-dispersions of flowable dry powder forms of the polishing agent prepared before or at the time of formulating the dentifrice.

When reading example 9 of D1, it would therefore have been evident to the person skilled in the art that the spray-dried silica of example 8 could be re-dispersed before being introduced into the dentifrice composition. Since the composition according to example 9 of D1 contains as liquid components 47% of a 30% sorbitol solution and additionally about 22% deionised water, the liquid available for such a re-dispersion step could only be water or a sorbitol (i.e. humectant) solution.

It would, moreover, have been evident to the skilled person choosing to use a silica dispersion (suspension) as a pre-mix that preliminary drying of the precipitated silica was not required and could be dispensed with, since a dispersion could readily be obtained by adding the liquid suspension medium, e.g. a sorbitol solution, to the wet filter cake.

In this context, the board considers that the feature of claim 1 of the patent in suit: "combining humectant with the precipitated silica" is met by combining a humectant solution with the precipitated silica, as further confirmed by paragraph [0030] of the patent specification, which mentions that the humectant used for fluidising the filter cake of precipitated silica can be introduced as an aqueous solution of sorbitol.

2.5.4 As a consequence, adding to the method according to D4 a step of preparing a suspension of precipitated silica in a humectant-containing liquid, in order to provide a further method for preparing an abrasive composition, was obvious in the light of document D1 combined with the general knowledge of the skilled person. The subject-matter of claim 1 of the main request does not involve an inventive step within the meaning of Article 56 EPC.

3. Inventive step - auxiliary requests 1 to 3

3.1 According to the respondent, the amendments in auxiliary requests 1 to 3 were introduced in order to meet objections under Article 100(c) EPC. With regard to inventive step, the parties have relied on the same line of argument as discussed in the context of the main request.

3.2 The board sees no reason to differ. The modified features in the auxiliary requests concern the acidulation reaction and shear treatment and have not been shown to establish a new distinguishing feature over the disclosure of document D4. Since the method step of humectant fluidisation of the precipitated

silica remains the only established distinguishing feature, the board's reasoning with regard to inventive step is not affected by the amendments.

3.3 As a consequence, the subject-matter of claim 1 of each of auxiliary requests 1 to 3 does not involve an inventive step within the meaning of Article 56 EPC, for the same reasons as explained in the context of the main request.

4. Inventive step - auxiliary request 4

4.1 Claim 1 of auxiliary request 4 differs from claim 1 of the main request solely in the use of the expression "wet filter cake" instead of "filter cake".

4.2 The expression "wet filter cake" can only mean that the filter cake contains liquid. It is assumed in favour of the respondent that the wording of claim 1 according to auxiliary request 4 excludes drying of the precipitated silica of the filter cake before it is combined with humectant to form a suspension.

4.3 As explained above (see point 2.5.3 *supra*, paragraphs 3 to 4), such an additional requirement would not, however, render the claimed method inventive, as it constitutes an obvious routine modification in the manufacturing phase of forming pre-mixes.

4.4 As a consequence, the subject-matter of claim 1 of auxiliary request 4 does not involve an inventive step within the meaning of Article 56 EPC.

5. Inventive step - auxiliary requests 5 to 7
- 5.1 Auxiliary requests 5 to 7 combine the modifications of auxiliary requests 1 to 3 with the feature "wet filter cake" of auxiliary request 4.
- 5.2 As explained above, none of the amendments introduced into auxiliary requests 1 to 4 affect the board's reasoning with regard to inventive step. Since the amendments are independent of one another, the permutations proposed in auxiliary requests 5 to 7 do not create a new situation in this respect.
- 5.3 As a consequence, the subject-matter of claim 1 of each of auxiliary requests 5 to 7 does not involve an inventive step within the meaning of Article 56 EPC.
6. In view of these findings, it is not necessary to consider the objections under Articles 100(c), 123(3) and 84 EPC.

**Order**

**For these reasons it is decided that:**

1. The decision under appeal is set aside.
2. The patent is revoked.

The Registrar:

The Chairman:

S. Fabiani

J. Riolo