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Datasheet for the decision of 21 February 2007

Case Number: T 0729/03 - 3.3.05

Application Number: 95307683.3

Publication Number: 0709340

IPC: C01B 33/18

Language of the proceedings: EN

Title of invention:
Method for purifying fine particulate silica

Patentee:
Shin-Etsu Chemical Co., Ltd.

Opponent:
Degussa AG

Headword:
Method for purifying fine particulate silica/SHIN-ETSU CHEMICAL CO.

Relevant legal provisions:
EPC Art. 123(2), (3), 54, 56

Keyword:
"Allowability of the amendments (yes)"
"Novelty (yes)"
"Inventive step (yes)"

Decisions cited:
G 0009/91, G 0010/91, T 0693/98

Catchword:
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Case Number: T 0729/03 - 3.3.05

D E C I S I O N
of the Technical Board of Appeal 3.3.05
of 21 February 2007

Appellant: (Opponent)
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Decision under appeal:
Decision of the Opposition Division of the European Patent Office posted 15 May 2003 rejecting the opposition filed against European patent No. 0709340 pursuant to Article 102(2) EPC.

Composition of the Board:

Chairman: B. Czech
Members: E. Waeckerlin
S. Hoffmann

Summary of Facts and Submissions

I. This appeal lies from the decision of the opposition division posted on 15 May 2003 rejecting the opposition against European patent No. 0 709 340.

II. Claim 1 as granted reads as follows:

"1. A method for purifying halide-bearing fine particulate silica, comprising

continuously feeding the silica with steam, or with a mixture of steam and air at a steam : air volume ratio of at least 0.5 : 1, upwardly through an upright column at a gas linear velocity in the range 1 to 10 cm/s to form a fluidised bed therein, the temperature in the column being in the range 250°C to 350°C whereby the steam eliminates halide from the silica, and removing the purified silica from the top of the column."

III. The opposition was filed against the patent on the grounds of Article 100(a) EPC. In its decision the opposition division held that the claimed subject-matter was novel having regard to the disclosures of the two documents referred to by the parties, namely:

D1: DE-C-1 150 955 (cited by the opponent) and

D2: GB-A-1 197 271 (cited by the proprietor of the patent in suit).

Regarding inventive step, it was stated in the decision that D1 did not provide the teaching that temperatures as low as 250 °C to 350 °C in combination with the

remaining operating parameters of claim 1 would lead to "acceptable results". D2 related to "a higher range of temperature", namely from 400 °C to 600 °C, and it excluded the formation of a fluidised bed. The combination of D1 and D2 did not lead to the method of claim 1 of the patent in suit. Therefore the claimed process was inventive over the teaching of each of the documents D1 and D2, or the combination of D1 and D2.

IV. In its statement of grounds of appeal, the appellant (opponent) argued, *in essence*, that the claimed process did not involve an inventive step in view of D1 in combination with the teaching of document

D3: DE-C-830 786,

which it considered to be "equivalent" to the Japanese patent JP-B-46274/1972 referred to in the patent in suit.

V. With its reply dated 2 February 2004, the respondent (proprietor of the patent) filed four sets of claims as main request and auxiliary requests 1, 2 and 3, respectively. Claim 1 according to said auxiliary request 2 reads as follows:

"1. A method for purifying halide-bearing fine particulate silica, comprising

continuously feeding the silica with steam, or with a mixture of steam and air at a steam : air volume ratio of at least 0.5 : 1, upwardly through an upright column at a gas linear velocity in the range 1 to 10 cm/s to form a fluidised bed therein, the temperature in the column being in the range 250°C to 350°C whereby

the steam eliminates halide from the silica, and

removing the purified silica, ***having a halogen content of 20 ppm or less****, from the top of the column."*

(Amendments with respect to claim 1 as granted emphasised by the board).

The respondent submitted that D3 should not be admitted into the proceedings since it was filed late and lacked relevance. Concerning inventive step, the respondent argued that the claimed subject-matter was not obvious in view of D1, D2 and D3, taken alone or in combination.

VI. In a subsequent letter dated 18 December 2006 the appellant raised objections under Article 84 EPC, pointing out that the description referred to a range of temperatures of from 250 to 600 °C, which differed from the range of from 250 to 350 °C of claim 1.

Furthermore an objection under Article 123(2) EPC was raised, based on the argument that the specific range of from 250 °C to 350 °C set out in claim 1 of all requests was not disclosed in the application as originally filed.

Moreover, the appellant argued that the claimed subject-matter was not inventive in view of each of the documents D1 and D3 taken alone, and of a combination of D1 and D3.

On 19 February 2007 the appellant filed by telefax four additional documents in order to support its argument according to which the use of hydrocarbons as thermal media in fluidised beds was known in the prior art.

VII. Oral proceedings took place on 21 February 2007. During the oral proceedings the respondent submitted a further amended set of claims as fourth auxiliary request. Subsequently the respondent prosecuted the second auxiliary request as the sole (i.e. main) request and submitted a description adapted thereto. The other previous requests were withdrawn.

VIII. As far as the respondent's sole remaining request is concerned, the arguments of the parties can be summarised as follows:

The appellant was of the opinion that due to the fact that the respondent had submitted amended claims, it was entitled to raise objections under Articles 123(2) and 84 EPC. It argued that since the temperature range of present claim 1 was not mentioned in the application as filed, the requirements of Article 123(2) EPC were not fulfilled. During the oral proceedings, it also argued that in the application as filed the additional feature of a "*halogen content of 20 ppm or less*" had only been described in connection with Figure 1. Hence the incorporation of the said feature into claim 1 amounted to a generalisation which was not allowable under Article 123(2) EPC.

The appellant did not raise any objections against the adapted description. During the discussion of one of the auxiliary requests (which was subsequently withdrawn) at the oral proceedings, the appellant however submitted that the features now contained in claim 4, according to which "*the feed rate of the silica is such as to provide from 0.001 to 0.01 g/cm^3 silica*", were unclear.

The appellant argued that D1, in particular example 1 thereof, disclosed a process having all the features of present claim 1 except for the temperature to be used. However, the claimed invention did not solve any problem that was not already solved by D1. From those passages of D1 referring to previously known methods, it could be concluded that de-acidification of silica with steam to a final pH of 4, i.e. to a halogen content of 20 ppm or less, was possible at temperatures as low as 200 °C. Moreover, it was stated in D1 that the required residence time depended on the product purity to be achieved. In order to achieve a product of the desired purity, the skilled person could therefore choose between a fast process at high temperatures and a slower process at lower temperatures without any inventive step being involved. D3 taught that the purification of silica could be carried out in any apparatus with co-current feed of silica and steam at temperatures of from 200 to 500 °C. According to D3, the treatment was performed for a period long enough for removing the impurities entirely or to a large extent. The process of present claim 1 was thus obvious in view of D3. On the other hand, carrying out the process of D1 at the low temperatures indicated in D3 was also obvious.

The respondent held that the appellant's objection under Article 123(2) EPC concerning the temperature range, as well as any objections under Article 84 which did not concern the amendments made after the grant of the patent, should be disregarded. Moreover the late filed document D3 should not be considered.

The respondent also submitted that the amendment consisting of the introduction into claim 1 of the residual halogen content of the silica was supported by the general part of the description and example 1 of the application as filed.

D1 prescribed the use of higher temperatures and did not disclose a range of possible steam : air volume ratios. Only example 1 contained an indication in this respect. The skilled person could not expect from D1, including the passages referring to previously known methods, that an efficient purification leading to a residual halogen content of 20 ppm or less would be possible at much lower temperatures, i.e. between 250 and 350 °C. The prior art did not suggest that this result could be achieved by using a set of specific process conditions, including the particular steam : air volume ratio and the particular temperature as recited in present claim 1. Document D3, which was very old, did not relate to fluidised bed processes and did not mention any steam : air volume ratios. The single example contained in D3 concerned a counter-current process. Document D2 acknowledged processes of the types described in earlier documents D1 and D3, respectively, and suggested the adoption of a counter-current process without fluidisation of the bed.

IX. The appellant requested that the decision under appeal be set aside and that the European patent No. 0 709 340 be revoked.

The respondent requested that the decision under appeal be set aside and that the patent be maintained on the basis of claims 1 to 5 submitted as auxiliary request 2

with letter dated 2 February 2004, the description (pages 2 to 4) submitted during the oral proceedings and figure 1 of the granted patent.

Reasons for the Decision

1. *Allowability of the amendments under Article 123(2) EPC*

1.1 The board notes that the temperature range of from 250 °C to 350 °C set out in claim 1 results from an amendment made during the examination of the application and consisting in the replacement of the value of 600 °C for the upper limit of the range by the value of 350 °C.

1.1.1 The appellant's corresponding objection thus relates to an amendment made before the grant of the patent, but it has not been raised as a ground of opposition under Article 100(c) EPC within the framework of the opposition proceedings. The objection was raised only during the appeal proceedings after the respondent had submitted amended claims.
At the oral proceedings, the respondent did not agree to the taking into consideration of this objection. Hence, in accordance with the principles for appeal review laid down by the Enlarged Board of Appeal in its decisions G 9/91 (OJ EPO 1993, 408, reasons points 18 and 19) and G 10/91 (OJ EPO 1993, 420, headnote 3 and opinion point 3), the appellant's objection under Article 123(2) EPC concerning the amended temperature range is not admissible and has therefore to be disregarded despite the fact that other post grant amendments have been carried out in claim 1 (see also

decision T 693/98 of 25 April 2002, not published, point 2 of the reasons). The examination of the compliance of the present amended claims with the requirements of Article 123(2) EPC is thus restricted to those amendments which were effected to the patent in its granted form.

1.1.2 In this context, the board observes, however, that the specific temperature value of 350 °C is disclosed in the application as originally filed (see page 5, lines 10 to 12), and that the temperature given in example 1, i.e. 300 °C, lies within the amended range of temperatures.

1.2 The only post-grant amendment objected to by the appellant under Article 123(2) EPC is the insertion into claim 1 of the features *"having a halogen content of 20 ppm or less"* concerning the purity of the silica obtained.

1.2.1 A basis for this amendment can be found in the statement on page 5, lines 24-25 of the application as originally filed. Moreover, example 1 of the application as filed mentions a chlorine content of 14 ppm achieved under operating conditions in full accordance with present claim 1, at a treatment temperature of 300 °C.

1.2.2 The quoted feature was not extracted from Figure 1 as contended by the appellant, but from a text passage of the "detailed description" referring to Figure 1 (see page 3, line 18 to page 6, line 3 of the application as originally filed). Figure 1 on its part is a schematic representation of the apparatus used when carrying out

the process described in the "detailed description", showing also the flow of the various material streams. Nothing in the "detailed description" or in Figure 1 itself suggests that some particular feature shown in the Figure but not comprised in present claim 1 would be required to achieve a "*halogen content of 20 ppm or less*". Moreover, the appellant has not indicated which particular subject-matter extending beyond the content of the application as originally filed would be contained in the patent as a result of the amendment. now contained particular subject-matter.

1.2.3 Therefore the board concludes that the said restricting amendment of claim 1 complies with the requirements of Article 123(2) and (3) EPC, since it neither adds subject-matter extending beyond the content of the application as originally filed, nor extends the protection conferred.

1.3 The board is also satisfied that the post-grant amendments effected to the description meet the requirements of Article 123(2) EPC. This was not disputed by the appellant.

2. *Clarity and support by the description (Article 84 EPC)*

2.1 Non-compliance with the requirements laid down in Article 84 EPC is not a ground for opposition according to Article 100 EPC. The board notes that the appellant's only remaining objection under Article 84 EPC concerns the alleged lack of clarity of the features of present claim 4, which is identical to claim 4 of the patent as granted. Since the objection does not arise from the amendments made to the claims

after grant, it does not need to be examined in the present appeal proceedings (see e.g. the book "Case Law of the Boards of Appeal of the EPO", 4th edition 2001, section VII.C.10.1.2, last paragraph on page 485).

2.2 The amended description does no longer refer to temperatures lying outside of the claimed range. Thus the appellant's earlier objection against inconsistencies contained in the patent as granted does not have a basis anymore.

2.3 The appellant has not raised any objections under Article 84 against the post-grant amendments of claim 1 and of the description. The board is also satisfied that these amendments meet the requirements of Article 84 EPC.

3. *Prior art cited*

3.1 D1 discloses a method for purifying highly dispersed halide-bearing oxides, particularly silica, comprising continuously feeding the oxide co-currently with steam, optionally diluted with an inert gas such as air upwardly such as to form a fluidised bed in an upright column, the temperature in the fluidised bed being in the range from about 450 °C to about 800 °C, whereby the steam eliminates halide from the oxide, and removing the purified oxide from the top of the column. The gas velocity through the column is at least about 2,0 cm/s. According to example 1 of D1 highly dispersed silica is treated with a mixture of steam and air at a steam : air volume ratio of 4.5 m^3 : 5 m^3 (i.e. of 0.9 : 1), the residence time being about 90 seconds. The flow rate of the fluidised suspension of particles

is about 10 to 12 cm/s. Reference is made in particular to the following passages of D1: Claims 1, 2, 5; col. 3, lines 13-40; col. 4, lines 29-38; col. 5, lines 4-14; col. 5, lines 23-29; col. 5, lines 45-53; col. 5, line 66 to col. 6, line 12, example 1.

3.1.1 The residual halogen content of the product obtained according to example 1 of D1 expressed in terms of the value of the pH of the treated silica is 4.0 (see col. 6, lines 11-12). According to the appellant this pH value corresponds to a halogen content of less than 20 ppm. The lack of corresponding evidence was only pointed out by the respondent at the oral proceedings, during which it expressly neither admitted nor contested the validity of the appellant's assertion. Under these circumstances the board has no reason to doubt the validity of the appellant's assertion. The board thus concludes that example 1 of D1 discloses the purification of silica to a residual halogen content of 20 ppm or less.

3.1.2 D1 also contains a reference to a temperature range of from 200 °C to 500 °C (see col. 1, line 49 to col. 2, line 24) but these temperatures are only mentioned in connection with different de-acidifying methods according to the prior art and not in connection with the method suggested in D1. There is no teaching in D1 to use temperatures below about 450 °C in the process described therein, let alone a temperature in the range of from 250 °C to 350 °C as required by claim 1 of the patent in suit.

3.2 D2 relates *inter alia* to a method for purifying chloride-bearing fine particulate silica, wherein the silica is continuously fed downwardly through a vertical column, in counter-current to a mixture of steam and air, the temperature in the column being in the range of from 400 °C to 600 °C, and removing the purified silica from the bottom of the column (see D2, claim 1; page 2, line 56 to page 3, line 7; page 3, examples 1 to 2). In the process of D2 the flow rate of the gas mixture is expressly kept at a level low enough to prevent the formation of a fluidised bed.

3.3 Document D3 has been cited in the grounds of appeal and has been discussed in detail in the respondent's reply. It relates to the purification of halide-bearing fine particulate silica by a heat treatment at temperatures in the range from 500 °C to as low as 200 °C in a gas or vapour flow (see page 3, lines 95-102). Moreover, it refers in some detail to methods of the kind briefly mentioned as earlier prior art in D1 (see col. 1, line 49 - col. 2, line 24) and in the patent in suit (see col. 1, lines 19-27). In the decision under appeal the use of such low temperatures is considered as a distinctive feature of some importance in the assessment of inventive step. Under these circumstances the board considers it appropriate to take D3 into consideration despite its late filing.

3.3.1 More particularly, D3 discloses a continuous method for purifying highly dispersed halide-bearing oxides, e.g. silica, by means of a heat treatment at temperatures of from about 200 °C to about 500 °C in the presence of a flow of gaseous or vaporous media (see claim 9 referring back to claims 1 and 2; page 3, lines 69-102),

in which case a co-current or, alternatively, a counter-current flow is preferred (see page 3, lines 113-117). The vaporous or gaseous media may be air, steam, nitrogen, producer gas etc. (see page 3, lines 106-109). The heat treatment may be carried out on an endless steel belt or in a separate apparatus, for example in rotary tubes ("Drehrohre") or rotating cylinders equipped with trickling installations ("rotierende Zylinder mit Rieselvorrichtungen") (see page 3, line 123 to page 4, line 2; page 4, lines 9-15).

3.3.2 D3 does not disclose the use of an upright column with a co-current gas flow to generate a fluidised bed. Nor is there any indication of the gas linear velocity or the use of a mixture of steam and air. As far as the residual halogen content of the purified product is concerned, it is stated in D3 that the heat treatment is continued until the impurities are entirely or to a large extent removed (see claim 9; page 3, lines 99-102). No numerical value is defined, however, for the maximum residual halogen content to be achieved.

3.4 None of the four documents submitted by the appellant on 19 February 2007 relates to the purification of particulate silica. The contents of these documents would only be relevant if the method for heating the fluidised bed were specified in the claims. Since this is not the case, the board concludes that the four documents are *prima facie* irrelevant. Therefore their contents are disregarded in accordance with Article 114(2) EPC.

4. *Novelty - Claim 1*

It follows from the above analysis that none of the documents D1 to D3 discloses a method having all the features of present claim 1. The subject-matter of claim 1 is therefore novel.

5. *Inventive step - Claim 1*

5.1 Considering the similarity of the method for purifying highly dispersed halide-bearing silica disclosed in D1, and of the method of claim 1, the board can accept the parties' view that D1 represents the closest prior art.

5.2 At the oral proceedings the appellant expressed doubts as to whether the use of lower temperatures than those disclosed in D1 made the claimed process necessarily more economical than the process according to D1. Using lower temperatures would require longer residence times and/or larger or more apparatuses. However, starting from the method disclosed in D1, example 1, the technical problem to be solved can in any case be seen, in accordance with what is stated in the patent in suit (see section [0005]), in the provision of a further method for purifying halide-bearing fine particulate silica whereby a comparably low residual content of halogen is achieved.

5.3 According to the remaining example of the patent, fumed silica resulting from pyrogenic hydrolysis was treated in accordance with the method of claim 1 at a temperature of 300 °C, a gas linear velocity of 3.4 cm/s and a steam : air volume ratio of 1 for a period of 7 minutes. The residual chlorine content of

the product was found to be 14 ppm, i.e. well below the maximum value of 20 ppm according to claim 1. It is thus plausible, and it has not been disputed by the appellant, that the technical problem set out above is solved by the claimed method.

5.4 Hence, it remains to be investigated whether, starting from D1, the provision of the method of claim 1 is an obvious solution of the stated technical problem.

5.4.1 D1 is concerned with the provision of a method for purifying highly dispersed oxides, oxide mixtures and mixed oxides obtained by thermal decomposition of volatile components of metals and/or metalloids, using steam and optionally an inert gas, at elevated temperatures, whereby the oxides are kept in a fluidised suspension, the aim being that an optimal effect be achieved at a considerably shortened treatment time, while avoiding the drawbacks of the previously known methods (see col. 3, lines 13-27).

5.4.2 According to D1 (see col. 1, line 49 to col. 2, line 35) the previously known methods consisted in a heat treatment carried out at temperatures of from 200 °C to 500 °C, optionally in a flow of gas and/or steam. More specifically it is mentioned in D1 that steam had been used for the purpose in methods carried out in a rotary tube, on an endless steel band, or in a worm-type conveyor, but that in order to reach a pH of 4, the de-acidifying treatment had to be carried out for 8 to 10 minutes. Referring to these particular methods, D1 then states that they were prone to operating problems because they had to be carried out in devices with moving parts at the required temperatures of about

500 °C. According to D1 (see column 5, lines 23 to 34), the method described therein may be carried out using shorter treatment times than in the prior art, while not requiring an apparatus with moving parts and still leading to a product with an acidity reduced to a pH value of 4.0 to 4.3. A thorough purification could thus be achieved with low energy requirements.

5.4.3 First of all, the board notes that the quoted passages of D1 relating to previously known methods do not expressly refer to a specific method operated at a temperature lower than about 500 °C in a gas flow containing steam. More particularly, D1 does not say in the quoted passage that the purity expressed as a pH value of 4 could be achieved within 8 - 10 minutes at temperatures much lower than 500 °C, let alone at temperatures in the specific range of present claim 1. On the other hand, although the authors of D1 acknowledge that the duration of the treatment will depend on the purity to be achieved (see column 3, lines 63 to 66), and although they were aware of the possibility to de-acidify oxides by subjecting them to a heat treatment at a temperature as low as 200 °C, they considered it essential to observe a minimum temperature of about 450 °C in the fluidised bed, preferably of about 500 °C (see claim 1 and column 3, lines 28 to 40) in order to achieve a rapid and far reaching purification.

5.4.4 The board can accept that in view of the information contained in D1 concerning earlier methods, the skilled person could expect that running the process of example 1 of D1 (i.e. 520 °C to 570 °C) at substantially lower temperatures of from 250 °C to

350 °C, as required by present claim 1, would also lead to some purification. However, nothing in D1 suggests that a residual halogen content as low as 20 ppm or less (corresponding to a pH value of 4) could be achieved under these process conditions, let alone within acceptable treating times. Hence the skilled person, confronted with the stated technical problem, would not, in the absence of *ex-post facto* considerations, have envisaged such a substantial lowering of the treating temperature.

5.4.5 Document D3 teaches in a general manner that de-acidification can be done at relatively low temperatures of from about 200 °C to about 500 °C. However, the only example of D3 where the temperatures used in the process are given concerns a de-acidification method wherein the oxides are carried by an endless steel belt in counter-current to a heated stream of e.g. air or steam, and wherein the temperature in the treatment chamber is e.g. 300 °C or 400 °C (see D3, claim 9; page 3, lines 95-97; page 3, line 123 to page 4, line 8).

5.4.6 In D3 steam and air are considered as equally suitable. Mixtures of steam and air, let alone any specific steam : air volume ratios, are not mentioned. D3 is also silent on operational conditions such as the velocity of the gas stream, the use of an upright column or the formation of a fluidised bed. Moreover, although it is generally stated in D3 that the heat treatment is upheld until the impurities are "entirely or to a large extent removed" (see page 3, lines 99-102), no specific information is given regarding the degree of purity which can actually be achieved or the

required duration of the treatment, particularly at temperatures as low as those required by present claim 1.

5.4.7 Hence, even assuming that the skilled person, starting from the process of example 1 of D1, would take D3 into consideration, despite the fact that on the one hand D3 has been published earlier (i.e. in 1952) than D1 (having the application date of 20 April 1961), and that on the other hand D3 does not relate to a co-current fluidised bed method, it would also not, without applying *ex post facto* considerations, be induced by D3 to modify the method of example 1 of D1 by reducing the temperature to the range of 250 °C to 350 °C, in order to solve the technical problem posed.

5.5 In view of the fact that it cannot be gathered from either D1 or D3 that it was possible to achieve a high purity silica (having a halogen content of 20 ppm or less) in acceptable treatment times in a fluidised bed method under the specific operating conditions indicated in present claim 1, taking, purely for the sake of argument, D3 as the starting point in the assessment of inventive step also must lead to the conclusion that the method of present claim 1 is non-obvious.

5.6 Document D2 (based on a priority application filed on 21 January 1967) is a more recent document than both D1 and D3. It is concerned *inter alia* with overcoming the problem of small quantities of acid accompanying the outflow of purified material withdrawn form the co-current fluidised bed in a process of the type described in D1 (see D2, page 1, line 70 to page 2,

line 26). The solution suggested by D2 is to replace co-current operation in a fluidised bed by a mode of operation where the oxide particles descend under gravity in counter-current to an ascending stream of a mixture of steam and a gas. The flow rate of the gas phase is kept at a level low enough to prevent the finely divided oxide from forming a fluidised bed, and the treatment zone is heated to 400 °C to 600 °C (see D2, claim 1; page 2, lines 56-72 and lines 110-128). Considering that D2, despite being more recent, suggests to avoid fluidised bed formation and to use temperatures far higher than required by present claim 1, it leads away from the invention as claimed.

5.7 The board concludes, therefore, that the method of present claim 1 involves an inventive step.

6. *Novelty and inventive step - Dependent claims 2 to 5*

Claims 2 to 5 of the patent in suit are dependent on claim 1. Novelty and inventive step of their subject-matter follow from the dependencies.

Order

For these reasons it is decided that:

1. The decision under appeal is set aside.

2. The case is remitted to the first instance with the order to maintain the patent on the basis of:
   - claims 1 to 5 submitted as auxiliary request 2 with letter dated 2 February 2004;
   - the description (pages 2 to 4) submitted during the oral proceedings; and
   - figure 1 of the granted patent.

The Registrar: The Chairman:

Ch. Vodz B. Czech