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DECISION
of 26 October 2000

Case Number: T 0119/97 - 3.3.5

Application Number: 88302743.5

Publication Number: 0287232

IPC: C01B 33/193

Language of the proceedings: EN

Title of invention:
Amorphous silicas

Patentee:
Crosfield Limited

Opponent:
Akzo Nobel N.V.
RHONE-POULENC CHIMIE

Headword:
-

Relevant legal provisions:
EPC Art. 56, 114(1)

Keyword:
"Inventive step - no, obvious alternative (main and first auxiliary request) - yes (second auxiliary request)"
"Remittal for adaptation of the description"

Decisions cited:
-

Catchword:
-

Case Number: T 0119/97 - 3.3.5

DECISION
of the Technical Board of Appeal 3.3.5
of 26 October 2000

Appellant/other party:
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Decision under appeal: **Interlocutory decision of the Opposition Division of the European Patent Office posted 28 November 1996 concerning maintenance of the European patent No. 0 287 232 in amended form.**

Composition of the Board:

Chairman: R. K. Spangenberg
Members: A.-T. Liu
J. H. Van Moer

Summary of Facts and Submissions

I. The appeal is from the interlocutory decision of the Opposition Division concerning the maintenance of European patent No. 0 287 232 following two notices of opposition.

II. The decision was based on an amended set of claims filed at the oral proceedings of 2 October 1996. This set consisted of 8 claims, of which claims 1, 6 and 8 were independent claims, with claims 2 to 5 dependent on claim 1, either directly or indirectly and claim 7 dependent on claim 6.

The independent claims read as follows:

"1. An amorphous silica having
i) a pore volume in the range from 1.6 to 2.5 cc g^{-1},
ii) a mean pore diameter in the range from 8.0 nm to 20.0 nm,
iii) a surface area from at least 550 m^2g^{-1} up to 1100 m^2g^{-1}
iv) an oil absorption in the range from 250 to 350 cc/100g,
v) an average particle size in the range 15 to 25 micrometer (sic).

6. A method of preparing an amorphous silica, suitable for use in beer clarification, and having
i) a pore volume in the range from 1.6 to 2.5 cc g^{-1},
ii) a mean pore diameter in the range from 8.0 nm to 20.0 nm,
iii) a surface area at least 550 m^2g^{-1},
iv) an oil absorption in the range from 250 to 350 cc/100g,

v) an average particle size in the range from 15 to 25 micrometers, which is produced by the reaction of sodium silicate, having a $SiO_2:Na_2O$ ratio in the range from 2.8 to 3.5:1, with mineral acid, the concentration and volume of the reactants being controlled to give a reaction in the pH range from 10 to 10.5, in the presence of a water soluble electrolyte comprising a cation selected from the group comprising sodium and potassium with an associated anion selected from the group comprising bromide, chloride and nitrate, wherein the electrolyte:silica weight ratio is from 0.10 to 1 to 0.25 to 1, the precipitation reaction being performed in the temperature range for (sic) 20°C to 50°C and the silicas (sic) separated from the reaction medium.

8. A process for the treatment of a fermented alcoholic beverage which comprises contacting the beverage with a silica having i) a pore volume in the range from 1.6 to 2.5 cc g^{-1}, ii) a mean pore diameter in the range from 8.0 nm to 20.0 nm, iii) a surface area from at least 450 m^2g^{-1} up to 1100 m^2g^{-1}, iv) an oil absorption in the range from 250 to 350 cc/100g, v) an average particle size in the range from 15 to 25 micrometers, and separating the silica from the beverage."

III. Among twenty-one documents which had been cited in the course of the opposition proceedings, reference shall be made specifically to the following in the present decision:

A3: US-A-3 959 174

R6: Lucilite data, Technical bulletin, ABM Brewing & Food group

IV. The opposition division held that the subject-matter of claim 1 was distinguished from the silica according to A3 by the stipulated particle size. Furthermore, the performance of the silica as claimed was accepted to be superior to the known compounds. An inventive step could be acknowledged since this effect was not expected in view of the prior art teachings.

V. With the Statement of grounds of appeal, the appellant Akzo Nobel N.V. (opponent 01) filed new documents, inter alia:

A18: MBAA Technical Quaterly, Vol. 16, No. 2, 1979, pp. 90-100.

A19: The Chemistry of Silica, R.K.Iler, Ed. John Wiley & Sons (1979), pp 174-177, 230-233, 554-561, 612-613.

VI. At the oral proceedings which were held on 26 October 2000 in the absence of the appellant Rhone-Poulenc Chimie (opponent 02), the respondent filed two new sets of claims as basis for a first and a second auxiliary request.

VII. Claims 1 and 7 according to the first auxiliary request were based on claims 1 and 8 of the impugned decision and further included the additional feature taken from claim 2 of the same decision. They thus incorporated the stipulation of a silica:

"(vi) containing less than 20 ppm of beer soluble iron wherein this represents less than 30 % of the total iron present."

The remaining claims 2 to 6 remained essentially unchanged with respect to claims 3 to 7 of the impugned decision.

VIII. Claim 1 of the second auxiliary request read as follows:

"A method of preparing an amorphous silica, suitable for use in beer clarification, and having

i) a pore volume in the range from 1.6 to 2.5 cc g^{-1},
ii) a mean pore diameter in the range from 8.0 nm to 20.0 nm,
iii) a surface area at least 550 m^2g^{-1},
iv) an oil absorption in the range from 250 to 350 cc/100g and,
v) an average particle size in the range 15 to 25 μm

and which is produced by the reaction of sodium silicate, having a $SiO_2:Na_2O$ ratio in the range 2.8 to 3.5:1 with mineral acid, the concentration and volume of the reactants being controlled to give a reaction in the pH range from 10 to 10.5, in the presence of a water soluble electrolyte comprising a cation selected from the group comprising sodium and potassium with an associated anion selected from the group comprising bromide, chloride and nitrate, wherein the electrolyte : silica weight ratio is from 0.10 to 1 to 0.25 to 1, the precipitation reaction being performed in the temperature range of 20°C to 50°C and the silicas (sic) separated from the reaction medium."

The dependent claims 2 to 6 were substantially based on claims 2 to 5 and 7 of the decision under appeal.

IX. The appellants' arguments may be summarised as follows:

- The utilisation of silica in the treatment of beer and the optimal combination of properties of a silica for beer stabilisation were discussed in A18.

- A skilled person desirous to develop a silica for beer treatment would consider document A3 and combine this teaching with A18.

- The additional stipulation of an upper limit for beer soluble iron was obvious in view of R6.

- The claimed methods of preparing an amorphous silica lacked an inventive step with regard to A3 in combination with A19 which disclosed various methods for precipitating silica.

- Claim 1 according to the first auxiliary request was not submitted in due time and should therefore not be admitted into the proceedings.

X. The respondent also submitted new documents with his reply to the appeals. His arguments may be summarised as follows:

- A18 indicated the optimal parameters for a silica hydrogel. The skilled person would not have any reason to suspect that high surface area and high porosity were relevant factors for a silica xerogel.

- There was no reason to combine the teachings of the unrelated prior art documents A3 and A18.

- Moreover, A3 did not disclose a xerogel with the optimum properties as indicated in A18. Even a combination of these two documents would not lead to the claimed silica.

- There was no incentive for the skilled person to further restrict the silica to an even lower limit of beer soluble iron content than required in R6.

- The claimed process was inventive since there was no suggestion in the cited prior art for combining the process parameters as stipulated.

XI. At the end of the oral proceedings, the appellant 01 requested that the decision under appeal be set aside and that the European patent No. 0287232 be revoked. He further requested, auxiliarily, that the first auxiliary request of the respondent be declared inadmissible. If said request was held admissible and had some chance of success, he requested remittal to the first instance for further prosecution.

With the letter dated 23 October 2000, appellant 02 requested that the decision under appeal be set aside and the patent be revoked.

The respondent (patentee) requested that the appeal be dismissed or, in the alternative, that the patent be maintained on the basis of the claims filed at the oral proceedings as first or second auxiliary request.

Reasons for the Decision

Main request

1. *Inventive step*

1.1 The patent in suit relates to synthetic amorphous silicas suitable for use in the treatment of beer and other alcoholic fermented beverages. The aim of the treatment is to stabilise the beverage properties during storage (see page 2, lines 5 to 6).

1.2 As background to the invention, the patent in suit explicitly cites document A18 which discusses in detail the various parameters of silica used to prevent permanent haze in beer (see patent in suit, page 3, lines 19 to 21). The Board therefore concurs with the appellant 01 that this article comprises the prior art teaching closest to the claimed subject-matter. A18 discloses in particular a silica hydrogel with optimum properties for the treatment of beer, namely a pore volume of 2,0 cc/g, a surface area of about 1000 m^2/g, a mean pore diameter of 8 nm and a mean particle size of 15 μm (page 95, paragraph bridging left hand and right hand columns and page 96, left hand column, last paragraph, Figure 14).

1.3 The Board accepts the respondent's submission that the problem to be solved by the claimed invention is the provision of a further silica for beer stabilisation, as an alternative to the silica hydrogel of A18. For the stated purpose, claim 1 proposes an amorphous silica with the following properties:

i) a pore volume in the range from 1.6 to 2.5 cc g^{-1},

ii) a mean pore diameter in the range from 8.0 nm to 20.0 nm,

iii) a surface area from at least 550 m^2g^{-1} up to 1100 m^2g^{-1}

iv) an oil absorption in the range from 250 to 350 cc/100g, and

v) an average particle size in the range 15 to 25 μm.

In agreement with the parties represented at the oral proceedings, the Board is satisfied that features i) to iii) and v) of claim 1 correspond to the optimal properties of a hydrogel as disclosed in A18 (see point 1.2 above). It is, however, also undisputed that A18 is silent about the oil absorption of the silica (see feature iv) of claim 1). Whilst this characteristic is positively a measure of the porosity of the silica, the Board is convinced by the respondent's submission that it also depends on the surface loading of the silica. It is plausible that, since the pores of a silica hydrogel are filled with water, its oil absorption capacity will inevitably be low. In effect, the Board holds that the silica proposed in claim 1 is distinguished from the silica hydrogel of A18 by the stipulated (higher) oil absorption capacity.

1.4 The experimental data shown in Table II of the patent in suit prove that the silica modification according to claim 1 is indeed also effective for beer stabilisation. The problem as stated by the patentee is thus solved by the silica as claimed. This is not in dispute.

1.5 The Board, however, considers the modification proposed by the invention obvious with regard to A18 with the knowledge of A3 for the following reasons.

1.5.1 The teaching in A18 is generally about the effect of different parameters on the suitability of silica for beer stabilisation, a priori without any restriction as to the type of silica to be considered. Whilst it is stated that the ideal combination of properties is achieved with the S-type hydrogel, the situation faced in A18 is that the xerogels investigated therein are unsuitable for having either a pore diameter which is too small or a surface area too low for the intended purpose (see in particular page 95, left hand column, last paragraph).

When reading A18, the skilled person is thus directly encouraged to look for a xerogel with the same properties that are found to be desirable for beer stabilisation, as an alternative to the S-type hydrogel.

1.5.2 Xerogels having in combination high pore volume and high surface area are, on the other hand, disclosed in A3 (see for example Tab. III, column 9). Furthermore, this document outlines the conditions for tailoring the silica to specifications, including the obtention of a product with an average particle size up to 20 microns (column 1, lines 23 to 59). Thus, the Board holds that a xerogel with the parameters determined in A18 as being highly desirable for beer stabilisation can be obtained following the general teaching of A3.

The only remaining question is therefore whether it is obvious for the skilled person to select one which has at the same time an oil absorption as stipulated in claim 1.

1.5.3 Interpretation of feature (iv) of claim 1.

The Board accepts the respondent's submission that the oil absorption capacity of a silica is not directly related to its surface area and pore volume. As already indicated in point 1.3 above, this value also depends on the surface loading of the silica. Since A3 in contrast to A18 explicitly concerns xerogels, the surface loading of the silica in A3 will be comparable to that of the claimed silica. The respondent has not submitted any arguments to the contrary.

The Board also concurs with the respondent in that the oil absorption is an approximate measure of pore size distribution in that, due to the molecular dimensions of the oil to be absorbed, pores with a diameter less than 3 nm (micropores) are too small to be penetrated by the oil. The absorption value thus does not reflect the volume of these micropores.

It is, however, undisputed that the oil absorption data are not obtained by an exact method. The Board therefore holds that not the exact value of oil absorption is of utter relevance for the assessment of inventive step but rather the implication of this parameter. In the Board's judgment, the stipulation of feature (iv) in combination with the remaining features (i) to (iii) in claim 1 means that the silica with the indicated high surface area and pore volume (and consequently with the indicated mean pore diameter):

(i) should be a xerogel and

(ii) must have most or all of the pores above the micropore range, thus above 3 nm.

Other properties which may be directly or indirectly implicated by the stipulation of oil absorption have not been indicated by the respondent.

1.5.4 Obviousness of the oil absorption requirement

The Board notes that the mechanism for beer stabilisation is well understood in the art and explicitly explained in A18. It is indicated therein that particular protein fractions are most active in haze formation. Rough calculations would suggest that a pore size of least 2.5 nm is necessary for accommodating such a fraction and therefore the effect of silica pore size on beer stabilisation has been investigated. The conclusion drawn in A18 is that optimum results are obtained with mean pore diameters in the range between 3 and 12 nm (see page 95, left hand column, first paragraph). Thus, A18 not only indicates the ideal combination of properties for the silica but also expressly discourages from selecting those with a pore diameter less than 3 nm since these micropores do not contribute to beer stabilisation. The stipulation of oil absorption is thus indirectly derivable from A18 through the suggested restriction.

1.5.5 The respondent has submitted that the ability to measure the pore size distribution is a relatively recent technique. It is thus not clear whether it was known, at the priority date of the patent in suit, that large pores are responsible for oil absorption. In the Board's judgment, this question does not have any bearing on the assessment of inventive step.

It suffices that the significance of pore size is discussed and the restriction to large pores is explicitly taught in A18. Clearly, methods for determining the volume of large pores, such as mercury intrusion, already existed at the priority date. The skilled person is thus not only directed to making the selection but also has the means to carry out such restriction. Even if the reasons for the oil absorption were not known, the restriction imposed by A18 would automatically lead the skilled person towards a silica with a high oil absorption value (see also point 1.5.3 above). It is thus irrelevant whether this restriction is expressed in terms of oil absorption or with other parameters.

1.6 The Board is also convinced that the combination of A3 with A18 is not based on hindsight.

1.6.1 The respondent has advanced the argument that chronology must be taken into consideration in evaluating the prior art. Document A3, published in 1976, was already public knowledge when A18 was being written. In his opinion, the authors of A18 must have then be aware of the existence of A3. Nevertheless, they suggested a silica hydrogel and dissuaded from xerogels for use in beer stabilisation. Furthermore, A3 was exclusively concerned with silica for use as flatting or thickening agents and not for beer stabilisation. The skilled person therefore did not have any incentive for combining the disclosures of A3 and A18.

1.6.2 The Board observes that, although A18 was published in 1979, this paper or at least its essential content was presented at a convention in October 1978 (see A18, footnote page 90). Thus, there was only a short time lag between the publication date of document A3 and the time period when the authors of A18 were working on their paper. The Board is, therefore not convinced that the time factor works in favour of an inventive step in the present case.

Furthermore, even if the authors of A18 were aware of A3, they may well have had their own reason for focussing on a hydrogel. As is clear from the text of A18, they were unable to obtain a xerogel silica with the properties found to be desirable for beer stabilisation (see point 1.5.1 above). As pointed out by the appellant, one of the authors of A18 (Dr. Lovell) is with Unilever Research Commpany and J. Crosfield and Sons, Ltd. (see A18, footnote at page 90). The article A18 being written for presentation at a congress, the authors' main interest may well have been the promotion of a new company product rather than the desire for drawing attention to products of a competitor such as W.R. Grace & Co., the proprietor of A3. Therefore, the Board cannot derive from the lack of reference to A3 in A18 that a skilled person, unconnected to either company, would be hampered in considering the content of these documents in combination.

1.6.3 When looking for a xerogel as suggested in A18, the skilled person would necessarily be intrigued by A3 whose title proclaims a method for selectively producing high pore volume silica gel. In the introductory part of A3, it is further stated that silica gels other than those intended for use as thickeners, flatting or reinforcing agents can be produced by the disclosed method of synthesis (column 1, lines 30 to 33). The Board therefore finds that, without requiring the benefit of hindsight, it is obvious for the skilled person to apply the teaching of A3 with the aim of obtaining the silica as indicated in A18.

1.7 The Board also has difficulty following the respondent's allegation that the teaching of A3 is not enabling for obtaining the desired silica.

1.7.1 The respondent has not submitted any technical data in support of the above allegation. He has relied on the argument that A3 only discloses either a silica with a high pore volume and a particle size which is much smaller than desired or large particle silica but in combination with a low pore volume. Thus, A3 would not relate to processes for obtaining a large particle silica with a high pore volume.

1.7.2 The above interpretation of A3 has been refuted by the appellant who has correctly pointed out that the terms "high" and "low" are applied in A3 only in a sense relative to each other. Thus, when the silica is for instance to be used as flatting agent, it should have an average particle size in the range from 1 to 20 microns and a "lower" pore volume from about 1.2 to 2.5 cc/g (column 1, lines 28 to 30 and 42 to 44). Interpreted in context, the expression "low pore volume" in A3 thus encompasses the "high" pore volume of 2.0 cc/g as suggested in A18 as well as the "high" pore volume range of 1.6 to 2.5 cc/g as stipulated in claim 1.

1.7.3 The Board holds that the onus is on the respondent to prove his allegation. In the absence of any concrete evidence to the contrary, the Board cannot dismiss the general teaching of A3 as unenabling for obtaining a silica with the parameters as found desirable in A18.

1.8 As a consequence, the main request must fail.

First auxiliary request

2. *Admissibility*

The subject-matter of claim 1 as amended is strictly based on claim 2 of the impugned decision. Thus, it is to be expected that such claim may come under discussion, should the respondent's main request fail.

Furthermore, both appellants have already commented upon the subject-matter of this claim (see Statement of the grounds of appeal dated 8 April 1997, page 7 last paragraph and Statement of the grounds of opposition dated 7 October 1993, item 4.2). The Board therefore does not see any reason for not admitting the present request into the proceedings.

3. *Inventive step*

The subject-matter of claim 1 of this request differs from that of the main request essentially in the stipulation that the silica is to contain less than 20 ppm of beer soluble iron.

It is undisputed that an upper limit of soluble iron is a standard requirement in the art, as indicated for example in R6 (see "Composition / Typical analysis"). The Board concedes that it is not clear whether the limit of 20 ppm specified in R6 is based on the ignited product or on the hydrogel as such. In the latter case, the limit of beer soluble iron based on dry silica would be formally higher than that stipulated in claim 1. It is however common knowledge that, if the silica contains any iron, its dissolution upon use for treating beer should be as low as possible. The lowering of the tolerable limit for beer soluble iron is, as such, self evident.

The respondent has also failed to indicate any significance in the combination of the stipulated limit with the other parameters. The Board therefore finds that the additional restriction of beer soluble iron in silica to said limit does not involve an inventive step.

Second auxiliary request

4. *Novelty*

It is undisputed that the combination of process parameters of claim 1 is not disclosed in any of the citations.

5. *Inventive step*

5.1 The Board considers A3 to represent the closest prior art since it relates to a process for obtaining a silica having properties found to be suitable for use in beer stabilisation (see point 1.5.2 above).

5.2 With respect to A3, the problem to be solved can therefore be seen in the provision of a further process as an alternative to that of A3.

5.3 There is no doubt that the process as proposed in claim 1 indeed leads to a silica with the properties as stipulated in the claim. The only question is therefore whether the claimed process is obvious in view of the available prior art.

5.4 The process of claim 1 is at least distinguished from A3 in that:

(i) the concentration and volume of the reactants, sodium silicate and mineral acid, are controlled to give a reaction in the pH range from 10 to 10.5, and

(ii) the reaction is carried out in the presence of a water soluble electrolyte which is a cation selected from the group comprising sodium and potassium with an associated anion selected from the group comprising bromide, chloride and nitrate.

The process of A3 comprises an alkaline gelation technique in which the reactants are not added simultaneously. In contrast, the sodium silicate solution is titrated with a mineral acid to a pH above 10.5 for gelation. Furthermore, the electrolyte which is added to the sodium silicate is either ammonia or sodium sulfate and not one of the species as indicated above (compare A3, column 3, lines 19 to 20 and claim 1).

5.5 The Board can agree with the appellant in so far as a general theory of silica polymerisation is outlined in A19. In particular, a schematic rendering of possible pathways for producing silica is illustrated in Figure 3.1 (page 174). The Board, however, cannot derive either from that document or from A3 any pointer towards the present modification.

5.6 If the Board had followed the appellant's approach in considering A19 to represent the closest prior art, it would have had as much difficulty selecting among the various possibilities offered in A19 a combination of process steps which would lead to a silica suitable for use as beer stabiliser, and having the properties as stipulated in claim 1.

5.7 The other documents cited during the opposition appeal proceedings do not contain any more relevant information. This is not in dispute.

5.8 As a consequence, the Board has come to the conclusion that the subject-matter of claim 1 of the second auxiliary request involves an inventive step. Claims 2 to 6 are dependent claims relating to specific embodiments of that subject-matter. The patent can therefore be maintained with these claims, after the necessary adaptation of the description.

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 according to the following documents:

   - Claims 1 to 6 (second auxiliary request)

   - A description to be adapted.

The Registrar:

S. Hue

The Chairman:

R. Spangenberg

R. Spangenberg