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D E C I S I O N
of 30 March 2004

Case Number: T 0363/00 - 3.3.4

Application Number: 90916579.7

Publication Number: 0447542

IPC: C12H 1/04

Language of the proceedings: EN

Title of invention:
IMPROVED BEER PROCESSING AND COMPOSITION

Patentee:
PQ Corporation

Opponent:
INEOS Silicas Limited

Headword:
Beer adsorbent/PQ

Relevant legal provisions:
EPC Art. 54, 56, 83

Keyword:
"Sufficiency of disclosure (yes)"
"Novelty (yes)"
"Inventive step (yes)"

Decisions cited:
T 0150/82, T 0014/83, T 0205/83, T 0019/90, T 0612/92,
T 0694/92

Catchword:
-



Case Number: T 0363/00 - 3.3.4

D E C I S I O N
of the Technical Board of Appeal 3.3.4
of 30 March 2004

Appellant: INEOS Silicas Limited
(Opponent) Bank Quay
Warrington
Cheshire WA5 1AB (GB)

Representative: Collingwood, Anthony Robert
Lloyd Wise, McNeight & Lawrence
Highbank House
Exchange Street
Stockport
Cheshire SK3 0ET (GB)

Respondent: PQ Corporation
(Proprietor of the patent) Valley Forge Executive Mall
P.O. Box 840
Valley Forge
PA 19482 (US)

Representative: Adam Holger, Dr.
Kraus & Weisert
Thomas-Wimmer-Ring 15
D-80539 München (DE)

Decision under appeal: Decision of the Opposition Division of the
European Patent Office posted 14 February 2000
rejecting the opposition filed against European
patent No. 0447542 pursuant to Article 102(2)
EPC.

Composition of the Board:

Chairwoman: U. M. Kinkeldey
Members: G. L. Alt
G. E. Weiss

Summary of Facts and Submissions

I. The appeal was lodged by the opponent (appellant) against the decision of the opposition division whereby the opposition was rejected and the European patent No. 0 447 542 which had been opposed under Article 100(a) EPC (lack of novelty, lack of inventive step) and Article 100(b)EPC, was maintained unamended on the basis of claims 1 to 17 as granted according to Article 102(2) EPC.

Claims 1 and 3 as granted read:

"1. A composition for treating beer to prevent chill haze, said composition comprising a silica gel reacted with a metal ion and being characterized by a uniform distribution of the metal in the pores and on the surface of the silica, and wherein said multivalent metal is not precipitated in the pores of the silica or around the particles of silica, said composition being prepared by the steps of:

a. combining a solution of sodium silicate or potassium silicate with a solution of an acid to form a silica hydrosol, the composition and proportions of said silicate and acid solution being such that 60 to 85% by weight of the Na_2O or K_2O in the silicate solution is neutralized and the hydrosol contains 8 to 12% by weight SiO_2 ;

b. allowing said hydrosol to set to a hydrogel;

c. granulating said hydrogel into discrete particles;

d. contacting said hydrogel particles with a solution of 3 to 10% by weight of a salt of a multivalent metal and maintaining the pH of the mixture of hydrogel and solution at a value of about 7 to 10.5, whereby the metal reacts or exchanges with the silica but large precipitates of hydrous metal oxide do not form within the pores of the silica or around the silica particles;

e. maintaining contact between the hydrogel particles and metal salt solution until the desired level of metal is reacted or exchanged with the silica;

f. washing the reacted silica;

g. drying and milling the reacted silica; and

h. recovering the product silica."

"3. A composition for treating beer to prevent chill haze, said composition comprising a silica gel reacted with a metal ion and being characterized by a uniform distribution of the metal in the pores and on the surface of the silica, and wherein said multivalent metal is not precipitated in the pores of the silica or around the particles of silica, said composition being prepared by the steps of:

a. combining a solution of sodium silicate or potassium silicate with a solution of an acid to form a silica hydrosol, the composition and proportions of said silicate and acid solution being such that at least all of the Na_2O or K_2O in the silicate solution is neutralized;

- b. allowing said hydrosol to set to a hydrogel;
- c. granulating said hydrogel into discrete particles;
- d. washing said gel;
- e. adjusting the pH of said washed gel to an alkaline value;
- f. contacting said hydrogel particles with a solution of 3 to 10% by weight of a salt of a multivalent metal and maintaining the pH of the mixture of hydrogel and solution at a value of about 7 to 10.5, whereby the metal reacts or exchanges with the silica but large precipitates of hydrous metal oxide do not form within the pores of the silica or around the silica particles;
- g. maintaining contact between the hydrogel particles and metal salt solution until the desired level of metal is reacted or exchanged with the silica;
- h. washing the reacted silica;
- i. drying and milling the reacted silica; and
- j. recovering the product silica.

Further independent claims 10 and 12 related to a method to treat beer to prevent chill haze by means of 200 to 1500 parts per million (ppm) of the compositions as defined in claims 1 and 3.

II. With the statement of grounds of appeal, the appellant submitted documents D26-D30.

- III. In reply to the statement of grounds of appeal the respondent (patentee) filed written submissions together with documents D31-D34.
- IV. The board issued a communication pursuant to Article 11(1) of the rules of procedure of the boards of appeal together with the summons to oral proceedings.
- V. The appellant submitted that he would not attend oral proceedings.
- VI. Oral proceedings were held on 30 March 2004.
- VII. The following documents are referred to in this decision:

D2: US 4,797,294

D8: Vail, J.G., Soluble Silicates, Chemistry, Vol. 1, pages 205-206, Reinhold Publishing Corporation, 1952

D9: US 1,342,102

D17: Characterization of Powder Surfaces, Academic Press, London, 1976, Chapter 8 by D. Barby, pages 353-425

D22: English Translation of JP-48-13834

D26: Declaration of Derek Aldcroft dated 7 June 2000

D27: US 3,940,498

D28: US 2,384,563

D32: US 3,872,217

D35: Sfat, MBAA Technical Quarterly, 1975, vol. 12, no. 4, pages 243-248, cited in the description of the patent in suit on page 2, lines 17-18

VIII. The arguments and evidence submitted by the appellant in writing may be summarized as follows:

Sufficiency of disclosure (Article 83 EPC)

(i) The manufacture of the composition of claims 1 and 3 amounted to an undue burden:

The production of a hydrogel under alkaline conditions reflected by Example 1 and the process features of claim 1 was non-standard. This was, for example, demonstrated by appellant's experimental work in document D26 showing that initial attempts to produce such a hydrogel failed because the conventional mixing and spraying apparatus was blocked by the quickly gelling hydrosol. Only with a modified apparatus and by optimising the mixing-process parameters could the hydrosol be sprayed and discrete hydrogel particles be obtained.

The indication of some important variables in connection with the ion exchange step like reactant temperatures, size of silica gel spheres, concentration of silica gel spheres suspended in water when contacted

with the magnesium sulfate, contact temperature of magnesium sulphate and silica gel spheres was missing.

(ii) Even if all the disclosure gaps were filled and a silica gel was manufactured, it did not have the properties described in the examples of the patent. In chillproof assays the composition did neither - as stated in the patent in suit - perform better than a commercially available hydrogel, nor than Britesorb D300, the commercialized product of the patent or the commercially available xerogel, Stabifix Super. This was an indication that the disclosure of some process steps important for obtaining the subject-matter as claimed must be missing from the description and/or that process parameters were not sufficiently specified and consequently, that the composition of claims 1 and 3 was not sufficiently disclosed.

Novelty (Article 54 EPC)

Document D2 disclosed the preparation of magnesium silicates by ion exchange of magnesium ions onto previously prepared gelled silica. Although process details were not included, the implementation of the process of document D2 by the skilled person would amount to a repetition of the procedural steps of Example 1 of the patent in suit and thus, lead to a composition of claims 1 and 3.

Document D9 disclosed the treatment of chrysotile asbestos with an aqueous inorganic acid to remove cations therefrom and to produce a silica gel-like compound. Despite differences in the manufacturing process, a magnesium silicate was produced that had a

chemical composition that significantly overlapped with that of the claimed composition.

Document D27 disclosed the treatment of magnesium silicates with an acid converting the magnesium contained in the silicate to a soluble salt followed by washing to remove the soluble magnesium salt. The resulting magnesium silicate had a metal content which was within the preferred level of the composition of the patent in suit.

Inventive step (Article 56 EPC)

Document D2 was the closest prior art document.

The subject-matter of claims 1 and 3 was obvious with regard to either document D2 alone or with regard to a combination of document D2 with document D8 or document D28:

(i) D2 disclosed a mixture of magnesium silicate and silica gel which had the same empirical formula as the composition of the patent in suit. Therefore a skilled person would expect that magnesium silicates having a composition similar to the mixtures of document D2 would be effective chillproofing agents. Moreover, document D2 disclosed that such products could be made by ion exchange of magnesium ions onto gelled silica.

(ii) Document D8 disclosed magnesium silicates. The teaching of document D8 to partially neutralize the sodium silicate before adding the magnesium salts would be used by the skilled person to implement the process of document D2.

(iii) Document D2 did not disclose any process details. However, these could be taken from document D28 dealing with the preparation of magnesium silicates.

IX. The arguments and evidence submitted by the respondent in writing and during the oral proceedings may be summarized as follows:

Sufficiency of disclosure (Article 83 EPC)

(i) The preparation of a silica hydrogel under alkaline conditions with subsequent manufacturing of particles was a standard procedure as shown by documents D17, D22 or D32.

(ii) It was not necessary to include more details about the ion exchange step or any other steps of the process because they were all well-known.

(iii) The results of appellant's chillproofing effectiveness tests in document D26 were unexpected. It could be seen from the submission dated 2 December 1999 and filed during opposition proceedings that when the results were plotted into a graph the curves did not show the expected concave shape, but had a convex or even zig-zag-like course. In contrast, if the results of the chillproof assay of the patent were plotted, the curves had the expected shape. A person skilled in the art would conclude from this discrepancy that the appellant's assays were not carried out properly.

Novelty (Article 54 EPC)

The essential feature of the process of the patent in suit was the granulation of the silica hydrogel into discrete particles before carrying out the ion exchange. None of documents D2, D9 or D27 disclosed this granulation step. Moreover, the documents were also silent on various further process variables of the patent in suit, like pH or concentration of metal during ion exchange. Consequently, none of the documents disclosed clearly and unambiguously a composition having the morphology of the composition of the patent in suit.

Inventive step (Article 56 EPC)

Before the priority date of the patent in suit difficult-to-chillproof beers had been treated with a mixture of silica gel and differing amounts of polyvinylpolypyrrolidone (PVPP). This was the closest prior art represented for example by D35. In view of this disclosure, the problem to be solved was therefore - as already stated in the patent - to provide a chillproof agent avoiding the use of PVPP.

None of documents D2, D8 or D28 suggested in any way the specific combination of steps of the patent, especially not that granulation had to be performed before the ions are exchanged. Consequently, the prior art could not be considered as suggesting to solve the underlying problem with a silica gel composition having the specific morphology disclosed in the patent.

Requests

- X. The appellant requested in writing that the decision under appeal be set aside and that the patent be revoked.

- XI. The respondent (patentee) requested that the appeal be dismissed and that the patent be maintained.

Reasons for the Decision

Introduction into the proceedings of late-filed documents D26 to D34 (Article 114 EPC)

- 1. None of the parties objected to allowing documents D26 to D34 to be introduced into the proceedings. Since furthermore none of these documents increases the complexity of the case so as to interfere with the smooth and efficient conduct of it, they are admitted into the proceedings pursuant to Article 114(1) EPC.

Sufficiency of disclosure (Article 83 EPC)

- 2. In the assessment as to whether a European patent application fulfils the requirement of Article 83 EPC, it is an established principle in the case law of the boards of appeal that, for the disclosure of an invention to be sufficiently clear and complete, the skilled person, on the basis of the information provided in the application itself and by using the common general knowledge at the application date (or priority date, as the case may be), has to be able to achieve the desired result without undue burden and

without exercising inventive skill (see eg decisions T 694/92 OJ EPO 1997, 408 and T 612/92 of 28 February 1996).

3. One question at issue here is therefore whether the disclosure of the patent in combination with the common general knowledge enables the preparation of a silica gel composition having the physical nature according to claims 1 and 3 without undue burden and without exercising inventive skill.
4. In order to support the argument that the manufacture of the composition of the patent in suit involves an undue burden the appellant submitted experiments demonstrating initial failure to prepare silica hydrogel spheres when following the alkaline gel-making process described in Example 1 of the patent in suit. Moreover, he submitted that important process details must be missing from the disclosure of the ion exchange procedure.
5. In the interpretation of the notion "undue burden" the boards of appeal consider that a certain amount of routine experimentation is acceptable to transform failure into success provided that it does not require inventive activity (see eg decision T 14/83, OJ EPO 1984, 105).
6. What has to be considered as routine in the here relevant technical field of production of silica gels, especially in the manufacture of alkaline-set gels, are be reflected in the following documents:

Document D17 states on page 367, last paragraph, first sentence that "the literature refers at length to gels made at $\text{pH} > 4$, in the region where the gel time is short", cites several related articles and explains some general principles of this procedure.

Document D22 discloses a method and apparatus to manufacture silica hydrogels under alkaline conditions by introducing an aqueous solution of an alkali silica and an acid simultaneously through separate channels into a vessel with an outlet so that the silica sol gels in less than 1.2 seconds after having been discharged into the air through the outlet.

Document D32 discloses an apparatus and process which is especially suited to prepare substantially spherical, silica containing hydrogels "by spraying droplets of silica hydrosols obtained by reacting alkaline silica containing raw material with acidic solutions into a gaseous medium and allowing the droplets to solidify while falling freely" (see the abstract). The droplets contain about 10% by weight of silica. The hydrosol is converted to a hydrogel within one second.

7. In the board's view these documents refer to process conditions that are similar to those contemplated by Example 1 of the patent in suit. Therefore, should there have been any difficulties during the gel preparation process under alkaline conditions they could have been resolved without inventive skill on the basis of common general knowledge as reflected by the documents discussed above.

8. Moreover, the board would like to draw the attention to two further points:

Firstly, the appellant submits himself in point 5.1.3 of the notice of opposition in the context of the discussion of lack of novelty that "the preparation of acid sols and alkaline sols is well-known".

Secondly, when arguing lack of novelty (see section 8 above) the appellant seems to accept that the disclosure of document D2 which provides apparently even less detailed information of process steps is sufficient to destroy novelty of the claimed composition.

9. Therefore, it is concluded that the adaptations of the process that were necessary due to failure in producing the composition of the patent do not exceed the routine level and that, consequently, the preparation of silica hydrogel particles of the patent in suit does not involve an undue burden.
10. With regard to the argument that disclosure of important process parameters concerning the ion exchange step was missing from the patent in suit, the board observes that the appellant did not submit tangible evidence demonstrating difficulties in carrying out this part of the process. Thus, his submissions are mere allegations. However, an objection for lack of sufficiency may only be successful if there are serious doubts substantiated by verifiable facts (see eg decision T 19/90, OJ EPO 1990, 476). These are missing here.

11. In a second line of argumentation the appellant submitted that even if he was able to fill the gaps in the disclosure and was able to prepare a silica gel composition, it did however not have the chillproof properties to be expected in view of the patent in suit, namely the improved efficiency when compared to a silica hydrogel. This is shown by chillproof assays filed with the notice of opposition and comparing the obtained product with a commercially available hydrogel. At the same time comparative tests were performed with a commercially available xerogel, Stabifix Super and Britesorb D300, the commercialized product of the patent in suit.

12. With his submission dated 2 December 1999 the respondent has filed a graph in which the results of these assays are plotted. The respondent submits that in view of the type of assay a person skilled in the art would expect a concave shape of the curves resulting from an increase in efficiency as the dose of chillproofing agent is increased with the haze levelling off at high doses. The board notes that this shape is indeed obtained if the results of the patent, for example of Table 1, are plotted. The plot of the appellant's results shows however a zig-zag shaped curve for the commercial hydrogel and more or less convex curves for Stabifix Super and Britesorb D300.

13. The board is convinced by the respondent's argument that the unexpected shape of these curves is the indication of a methodical error during the chillproofing assays with the consequence that they cannot be taken into account as evidence. Hence it must be concluded that the appellant has not demonstrated

that the process disclosed in the patent in suit is deficient and therefore leads to "wrong" products.

14. In summary, the arguments and evidence put forward by the appellant could not convince the board of the insufficiency of the disclosure. Hence, the patent is considered to fulfil the requirements of Article 83 EPC.

Novelty (Article 54 EPC)

15. The silica gel composition of claims 1 and 3 of the patent in suit is characterized by "a uniform distribution of metal in the pores and on the surface of the silica and wherein said multivalent metal is not precipitated in the pores of the silica or around the particles of silica". These structural features are the direct consequence of the manufacturing process. The features of this process are used as further means of characterization in claims 1 and 3. Neither the respondent nor the appellant have attempted to determine the distribution of metal directly, nor are there data about this feature in the prior art documents at the disposition of the board. Consequently, claims 1 and 3 have to be considered as product-by-process claims.
16. If products are defined by their production process, they must fulfil the requirements of patentability themselves (eg decision T 150/82, OJ EPO 1984, 309). This principle is based on the reasoning that different processes may well lead to the same product.

17. The respondent submitted during oral proceedings that, apart from proper mixing of the starting compounds, the most important process step which ensures the occurrence of the above-mentioned structural feature is the granulation of the hydrosol into discrete particles before ion exchange takes place. He said that, if large, non-granulated agglomerates of hydrogel were subjected to ion exchange, ions are hindered to diffuse into all the pores due to the irregular surface. Moreover, the longer the diffusion took the higher was the chance that precipitation of magnesium hydroxide or magnesium silicate occurred as a competition reaction to the proper ion exchange.

18. The question to be answered is therefore whether any of documents D2, D9 or D27 discloses silica gel particles as claimed.

19. The appellant argues that document D2 discloses the same essential process steps as the patent. Consequently, the produced compound must be identical to that of the patent. With regard to document D9 the appellant submitted that the preferred magnesia, silica and water contents of the compound of D9 and that of the patent greatly overlap indicating that the same compound is disclosed. The silica gel of document D27 is considered novelty-destroying by the appellant because it contains the same final magnesium oxide content as the composition of the patent.

20. Since there is no explicit disclosure in any of these documents of a compound having a uniform distribution of metal in the pores on the surface of silica, the next question is whether there is an implicit disclosure of such a compound, i.e. whether by carrying out a process disclosed in documents D2, D9 or D27 a silica gel having the characteristics of that of the patent in suit is inevitably obtained. Decision T 205/83 (see OJ EPO 1985, 363) states that if a chemical product cannot be defined by structural characteristics, but only by its method of manufacture, novelty could only be established if evidence was provided that modification of the process parameters resulted in other products.
21. In document D2 the process assumed to be leading to a silica gel identical to that of the patent is disclosed in a short paragraph in the introduction (column 2, last paragraph): "Amorphous and porous magnesium silicate is required to provide superior chillproofing performance with silica gel. Such silicates are articles of commerce and can be prepared by a number of ways such as ... ; and ion exchange of magnesium ions onto previously prepared precipitated gelled silica followed by washing, dewatering and drying steps to provide the appropriate silicate". The document does not mention any of the specific process parameters recited in claims 1 and 3, like, for example, the percent weight of the salt of the metal to be added or the pH at which the solution is to be maintained during ion exchange. Most importantly however, document D2 is silent about the granulation of the hydrogel before the ions are exchanged. Since this step has to be considered as having a decisive influence on the

morphology of the product (see point 17 above), the board is convinced that the different manufacture process disclosed in document D2 will give rise to a product which is different from the claimed one.

22. Regarding document D9 it is noted that the identity of the ratio of magnesium, silica and water is - at least in the case of silica gels - not necessarily an indication that the overall structure of such gels is identical. The authors of document D9 do not only use a different starting material - chrysotile asbestos with a fibrous structure - but also leach out magnesium ions from the product instead of adding it by ion exchange. Here again, the board is of the opinion that these differences in the manufacturing process have an influence on the final structure of the silica gel so that the process of document D9 would not result in a product identical to that of the patent, even if it was granulated at the end of the process.
23. Similar considerations apply to the compound disclosed in document D27: The removal of magnesium from a conventional magnesium silicate gel will result in a loss of magnesium ions from the surface and not in its uniform distribution over the surface. Consequently, document D27 does not disclose the product claimed in the patent in suit.
24. Hence, none of documents D2, D9 or D27 discloses explicitly or implicitly the composition of claims 1 and 3. Therefore, the board comes to the conclusion that their subject-matter is novel.

Inventive step (Article 56 EPC)

25. In the course of the beer production process haze, the so-called chill haze, develops during storage at low temperatures. It is caused by coagulation of organic materials in the beer. The higher the malt content of the beer is, the more impurities occur and the more difficult is it to remove them.
26. The respondent conceded during oral proceedings that, in view of the results presented in the patent in suit, document D35 relating to chillproofing of **high** malt beers was a more appropriate starting point for the assessment of inventive step than document D2 relating to chillproofing agents for beer in general without putting emphasis on the specific type of high malt beer.
27. Under the problem/solution approach generally adopted by the boards of appeal the closest prior art document to be used as a starting point for objectively assessing inventive step is generally one having the same underlying objective or purpose of the patent. Since document D2 does not relate to the removal of haze from **high** malt beers, the board agrees with the respondent that the document D35 is the closest prior art document. It discloses that difficult-to-stabilize beers are treated with a combination of silica gel and polyvinylpolypyrrolidone (PVPP).
28. In view of this document the problem to be solved by the patent is the provision a chillproofing agent that is effective in beers with a high malt content and that does not involve PVPP, thus rendering the chillproof process easier to handle.

29. As a solution to this problem the composition of claims 1 and 3 is provided. That the intended effect is actually achieved by this composition is apparent from Tables 1-3 describing positive results of chillproof tests with difficult-to-treat 70% or 80% malt beer.
30. In order to evaluate inventive step, the question has to be answered whether it was obvious in the light of the prior art to provide the compositions of claims 1 and 3 as a solution to the above formulated problem.
31. The closest prior art document D2 teaches that a mixture of magnesium silicate and silicate gel having, as calculated by the appellant, the same empirical formula as the composition of the invention, is a better chillproof agent than silica gel alone. The introductory part of document D2 discloses how each of the two constituents - which are both known as such - can be prepared. The whole point of document D2, and this is also reflected by its examples, is that a blend of magnesium silicate and silica gel is better for removing chill haze than a silica gel alone. Thus, in the light of the whole disclosure content of this document alone a skilled person would not be led to prepare a magnesium silicate gel as a single-composition chillproof agent.
32. The appellant further relies on the combination of D2 with either of documents D8 or D28 to argue lack of inventive step.
33. In the board's view document D2 read in combination with document D8 cannot render the claimed composition

obvious: Document D8 is a text book and discloses in one of its chapters that "magnesium silicates may be prepared by exchanging magnesium into an alkaline silica gel, the partial neutralization of the sodium silicate solution before adding magnesium salts, and the addition of other salts to the sodium silicate before precipitation." If it was assumed that a skilled person took from document D2 the information of an advantageous ratio of magnesium to silicate and from document D8 the information of a possible manufacture process of magnesium silicates, the skilled person would however not find an indication in these documents about the need of a specific surface distribution of the magnesium and its relationship to chillproof properties.

34. Similar considerations apply to a combination of document D2 with document D28. Document D28 deals with the preparation of magnesium silicates by interaction of a magnesium salt with an alkali metal silicate. Thus, this process relies on a different manner to prepare magnesium silicates which leads to morphologically different products.
35. Finally, the board observes that the patent in suit on page 2, lines 37-38 states that "the metal must be introduced into our products as described because other ways of providing metal do not provide the desired chillproofing activity". Thus, the patent in suit confirms that granulation of the gelled silica with subsequent ion exchange are the decisive process steps with regard to the final morphology of the silica gel.

36. Hence, it is concluded that document D2 alone or this document in combination with document D8 or document D28 suggests preparing the composition of claims 1 and 3 as a solution to the problem stated above. Consequently an inventive step is present for the subject-matter of claims 1 and 3.

37. Independent claims 10 and 12 relate to a method of treating beer to prevent chill haze. The essential feature of this method is to contact the beer with 200 to 1500 parts per million of a metal reacted silica gel as defined in claims 1 and 3. Since this composition was found patentable, the method of claims 10 and 12 derives its inventive step from that of the product.

Order

For these reasons it is decided that:

The appeal is dismissed.

The Registrar:

The Chairwoman:

P. Cremona

U. Kinkeldey