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DECISION
of 20 November 2003

Case Number: T 0094/00 - 3.3.7

Application Number: 90102469.5

Publication Number: 0384226

IPC: C09D 7/12

Language of the proceedings: EN

Title of invention:
Inorganic hydrogel flatting agents

Patentee:
W. R. Grace & Co.-Conn.

Opponent:
UNILEVER PLC / UNILEVER NV

Headword:
-

Relevant legal provisions:
EPC Art. 83

Keyword:
"Sufficiency of disclosure - the sole method disclosed and discussed for the determination of the claimed parameter (pore volume of a hydrogel) provides a parameter for a product (xerogel) different from the claimed one - no"

Decisions cited:
-

Catchword:
-



Case Number: T 0094/00 - 3.3.7

DECISION
of the Technical Board of Appeal 3.3.7
of 20 November 2003

Appellant:
(Proprietor of the patent) W.R. Grace & Co.-Conn.
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Decision under appeal: Decision of the Opposition Division of the
European Patent Office posted 16 November 1999
revoking European patent No. 0384226 pursuant
to Article 102(1) EPC.

Composition of the Board:

Chairman: R. E. Teschemacher
Members: B. J. M. Struif
P. A. Gryczka

Summary of Facts and Submissions

I. The mention of the grant of European patent No. 0 384 226 with respect to European patent application No. 90 102 469.5 was published on 27 July 1994. Claim 1 read as follows.

"A flattening agent comprising an inorganic hydrogel having a pore volume of at least 1.0 ml/g, a volatiles content of at least 40 wt.%, an average particle size in the range of 1 to 10 μm and a particle size distribution such that when the inorganic hydrogel flattening agent is dispersed in a coating vehicle, the fineness of grind is at least 4.75 on a Hegman scale."

II. A notice of opposition was filed against the granted patent, requesting the revocation of the patent in its entirety on the grounds of lack of novelty, lack of inventive step and insufficient disclosure under Article 100(a) and (b) EPC, respectively.

III. In a decision posted on 16 November 1999, the opposition division revoked the patent under Article 102(1) EPC. That decision was based on a main request and three auxiliary requests.

Claim 1 of the main request corresponded to claim 1 as granted. In claim 1 of the first auxiliary request the range of the average particle size was restricted to 2 to 7 μm . Claim 1 of the second auxiliary request differed from claim 1 of the first auxiliary request in that at the end thereof the following feature was supplemented:

- ",the hydrogel before being milled to said particle size being washed at a pH of 5 to 10."

Claim 1 of the third auxiliary request corresponded to claim 7 of the main request and read as follows:

"A process for the production of a flatting agent comprising (a) forming an inorganic hydrogel, (b) washing the hydrogel at a pH of 5 to 10, and c) milling the resultant hydrogel while maintaining a volatiles content of at least 40 wt.%, to produce an inorganic hydrogel flatting agent having a pore volume of at least 1.0 ml/g, an average particle size in the range of 1 to 10 μm and a particle size distribution such that when the inorganic hydrogel flatting agent is dispersed in a coating, the fineness of grind is at least 4.75 on a Hegman scale."

The decision can be summarized as follows:

- (a) The amendments in claim 7 of the main request were in compliance with the requirements of Article 123(2) and (3) EPC.
- (b) With respect to the requirements under Article 83 EPC, the particle size was sufficiently disclosed, whilst the pore volume and the particle size distribution of the claimed flatting agent were not. The pore volume was calculated from the fraction of the pore volume which was occupied by the solvent as determined by the total volatiles test, plus the fraction of the pore volume unoccupied by the solvent. The later pore volume was measured according to the patent specification by mercury porosimetry according to the method of

Ritter and Drake, mentioned in D19A, which required a high vacuum to cause evaporation of water. Thus, the pore volume of water was counted twice and the method disclosed in the opposed patent did not lead to the true pore volume. There were no instructions in the patent in suit how the porosimetry method should be modified to obtain reliable results. The patentee had in particular not shown how water could remain in the pores when heat or vacuum was applied.

Although the particle size distribution was measured by a standard method, its measurement depended strongly on the method for producing the dispersion, in particular which coating vehicle, shear rate and vessel dimension were used. Since two different coating vehicles were exemplified, two different Hegman values could be obtained.

Consequently, the subject-matter of the main request did not meet the requirement under Article 83 EPC.

- (c) The above arguments also applied to the first and second auxiliary requests. The results of the process of claim 1 of the third auxiliary request could only be verified by the product obtained by said process. Since this product was defined by the same features as in claim 1 of the main request, the process as a whole was also not adequately disclosed.

IV. On 13 January 2000 the patentee (appellant) filed a notice of appeal against the above decision, the prescribed fee being paid on the same day. In the statement setting out the grounds of appeal filed on 16 March 2000, the appellant submitted three auxiliary requests. During the appeal proceedings *inter alia* the following further documents were cited:

D19: H. L. Ritter and L. C. Drake: Pore-Size Distribution in Porous Materials; Pressure Porosimeter and Determination of Complete Macropore-Size Distributions, Industrial and Engineering Chemistry, Vol. 17, No. 12, (1945), 782 to 786

D19A: L. C. Drake and H. L. Ritter: Macropore-Size Distribution in Some Typical Porous Substances, Industrial and Engineering Chemistry, Vol. 17, No. 12, (1945), 787 to 791

D21: ISO/CD 15901-1, Pore Size Distribution and Porosity of solid materials by Mercury Porosimetry and Gas Adsorption- Part 1: Mercury Porosimetry, 2002

V. In response to a communication of the board, the appellant submitted by letter dated 27 October 2003 an amended main request and three auxiliary requests which replaced the former requests on file. Claim 1 of the main request remained in the version as granted.

Claim 1 of the first auxiliary request read as follows:

"A flatting agent comprising an inorganic hydrogel having a pore volume of at least 1.0 ml/g, which pore volume is determined by adding the pore volume obtained by mercury porosimetry to the solvent occupied pore volume, a volatiles content of at least 40 wt.%, an average particle size in the range of 1 to 10 μ m and a particle size distribution such that when the inorganic hydrogel flatting agent is dispersed in a coating vehicle, the fineness of grind is at least 4.75 on a Hegman scale."

Claim 1 of the second auxiliary request read as follows:

"A flatting agent comprising an inorganic hydrogel having a pore volume of at least 1.0 ml/g, a volatiles content of at least 40 wt.%, an average particle size in the range of 1 to 10 μ m and a particle size distribution such that when the inorganic hydrogel flatting agent is dispersed in a coating vehicle, the fineness of grind is at least 4.75 on a Hegman scale, determined in White Baking Enamel Formula #P-1734-217B of Cargill Co."

Claim 1 of the third auxiliary request read as follows:

"A flatting agent comprising an inorganic hydrogel having a pore volume of at least 1.0 ml/g, which pore volume is determined by adding the pore volume obtained by mercury porosimetry to the solvent occupied pore volume, a volatiles content of at least 40 wt.%, an average particle size in the range of 1 to 10 μ m and a particle size distribution such that when the inorganic hydrogel flatting agent is dispersed in a coating vehicle, the fineness of grind is at least 4.75 on a

Hegman scale, determined in White Baking Enamel Formula #P-1734-217B of Cargill Co."

(Emphasis added to the differences from claim 1 as granted)

VI. Oral proceedings were held on 20 November 2003.

The appellant's arguments can be summarized as follows:

- (a) The total pore volume was the sum of the solvent occupied pore volume and the mercury pore volume as measured on the final, small particle size hydrogel flattening agent. The solvent occupied pore volume was determined by heating the hydrogel sample to a temperature sufficient to remove any volatiles. The pore volume unoccupied by the solvent was determined by standard mercury porosimetry as described in D19A mentioned in the patent in suit. According to that article, before measuring the samples were heated and treated under vacuum so that no significant amount of water remained in the samples. The method was adequately and sufficiently described to provide reliable and repeatable results. It was irrelevant whether or not these values came close to the theoretic or true pore values of the hydrogel and whether the water was counted twice.
- (b) The same arguments applied to the auxiliary requests wherein also the methods of measurements were specified in order to remove any uncertainty in this respect.

VII. The respondent's arguments can be summarized as follows:

- (a) Whilst the pore volume of the hydrogel occupied by solvent could be measured as specified in the patent in suit, the pore volume unoccupied by solvent could not be measured by a mercury porosimetry as described in D19A. The subject-matter of the opposed patent was directed to a hydrogel which according to D19A must be heated and/or evacuated to remove the water in order to determine the pore volume by mercury porosimetry. According to the patent in suit, by removing the water of the hydrogel the pore structure collapsed and the result was a xerogel. Since the pore volume of the hydrogel but not that of a xerogel should be determined, the patent in suit did not give any guidance to the skilled person how to adapt the mercury porosimetry method to hydrogels so that the portion of the pore volume unoccupied by solvent could be measured. Furthermore, according to D19A the pressure which was necessary to cause the mercury penetrating the pore structure was limited to 10 000 psi whilst an intrusion pressure up to 60 000 psi was required to provide reliable results. Thus, without additional information the porosimetry method of D19A could not be applied. Furthermore, no accurate pore volume could be measured since the pore volume occupied by water was counted twice by that method.

- (b) The same arguments applied for the auxiliary requests.

VIII. The appellant requested that the decision under appeal be set aside, that the case be remitted to the first instance and that the patent be maintained on the basis of the main request, alternatively on the basis of one of the three auxiliary requests filed by letter dated 27 October 2003, replacing the previous requests on file.

IX. The respondent requested that the appeal be dismissed.

Reasons for the Decision

1. The appeal is admissible

Amendments

2. The question whether the amendments effected in the main and the three auxiliary requests are in compliance with Article 123(2) and (3) EPC can be left open, since all requests fail to meet the requirements pursuant to Article 83 EPC.

Sufficiency of disclosure

3. According to Article 83 EPC, the European patent application must disclose the invention in a manner sufficiently clear and complete for it to be carried out by the skilled person. The essence of the appellant's arguments regarding sufficiency of disclosure was that the flattening agent could be reproduced because the patent specification disclosed sufficiently how the pore volume of the claimed

hydrogel materials could be measured by using the mercury porosimetry described in D19A.

- 3.1 There is a detailed description in the patent in suit how hydrogel flattening agents can be produced by forming an inorganic hydrogel, washing the hydrogel and milling the resulting hydrogel while maintaining a volatile content of at least 40 wt.% (page 3, lines 27 to page 4, line 5). In example 1 the production of a silica hydrogel is specifically described.

- 3.2 The claimed hydrogel is however defined by specific parameters, in particular by a specific pore volume of at least 1 ml/g so that the question arises, whether the skilled person is able to determine the claimed parameters on the produced hydrogel and thereby to check whether the claimed products have been produced.
 - 3.2.1 According to the patent in suit, the total pore volume is measured on the final, small particle size hydrogel flattening agent and is the sum of the solvent occupied pore volume and the mercury pore volume (page 4, lines 21 and 22). The mercury pore volume corresponds to that fraction of the pore volume that is unoccupied by the solvent. To obtain the total pore volume, the solvent occupied pore volume, as determined by the total volatiles test is added to the mercury pore volume (page 4, lines 18 to 20).

 - 3.2.2 It is not disputed that the pore volume occupied by solvent can be reliably determined by heating the hydrogel at 955 °C for one hour so that any volatiles will be removed (page 4, lines 33 to 36). Thus, the remaining question is, whether the pore volume

unoccupied by solvent can be determined by taking into account the methods mentioned in the patent in suit.

3.2.3 According to the patent in suit, the mercury pore volume is measured by following the instructions given in D19A (page 4, lines 6 to 9). According to D19A the method can be used for porous silica-alumina gels (page 787, left column, first paragraph). Before measuring the pore volume the porous samples were heated for 16 h at 540 °C and evacuated for 0.5 h at 0.001 mm Hg (page 788, left column, last paragraph). According to the appellant, that specific treatment was also carried out when measuring the pore volume unoccupied by solvent as specified in the examples of the patent in suit (compare bridging paragraph, pages 5 and 6). As confirmed by both parties, by such a sample pre-treatment water is completely removed from the hydrogel.

3.2.4 Furthermore, also according to D21 which makes reference to D19A and D19 (D21, Bibliography), the determination of porosity of solid materials by standard mercury porosimetry requires the removal of absorbed water and other organic molecules which may obscure the porosity (page 7, point 8.2.1, Sample pre-treatment).

3.2.5 However, according to the patent in suit the final gel properties can be controlled by the rate and method of drying. If the hydrogel is dried slowly, the pore structure collapses and results in a xerogel (page 2, lines 28 and 29). Furthermore, the patent in suit mentions that a minimum volatiles content of at least 40 weight percent is necessary to preserve the pore

volume of the hydrogel (page 4, lines 31 to 33). Thus, if the volatiles, in particular water, are completely removed from the hydrogel, as required by the pore volume measuring method of D19A, the pore structure of the claimed hydrogel cannot be preserved. Consequently, when following the teaching of D19A a pore volume of a product (xerogel), which is not identical to the claimed hydrogel, will be determined.

Since according to claim 1 a hydrogel is defined by its specific pore volume and volatiles content, the skilled person must be able to measure the pore volume of that hydrogel and it is insufficient to determine the pore volume of a completely different product (xerogel), even if that separate determination provides, as argued by the appellant, accurate and reliable results.

3.2.6 Therefore, the pore volume measured according to D19A cannot represent the pore volume of the claimed hydrogel. The skilled person looking for a solution to that conflicting situation will not find any guidance in the patent specification as to how the specified mercury porosimetry can be modified so that the pore volume as defined in the claims, in particular the part of the volume unoccupied by the solvent, can be determined.

3.3 From the above it follows that the patent in suit as a whole, also when taking into account common general technical knowledge, does not provide sufficient technical information as to how the claimed hydrogel having the required pore volume can reliably be reproduced. Since the determination of the pore volume is already deficient, the question can be left open,

whether or not the other claimed parameters can reliably be determined. Consequently, the requirements pursuant to Article 83 EPC are not met.

3.4 The same considerations as specified under points 3.2 and 3.3 for the main request apply to the auxiliary requests as well. Since the teaching in the patent in suit as a whole is insufficient under Article 83 EPC, the amendments to the claims cannot remedy these objections.

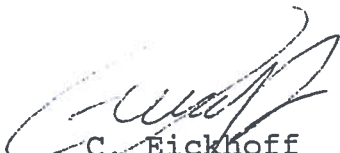
Order

For these reasons it is decided that:

The appeal is dismissed

The Registrar:

The Chairman:


C. Eickhoff




R. Teschemacher

17.02.04 Str
10.07.04 R.