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Datasheet for the decision of 1 March 2012

Case Number: T 0071/10 - 3.3.03

Application Number: 01964275.0

Publication Number: 1313781

IPC: C08F 257/00

Language of the proceedings: EN

Title of invention:
Functionalized polymeric media for separation of analytes

Patent Proprietor:
Avantor Performance Materials, Inc.

Opponent:
Metrohm AG

Headword:
-

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

Relevant legal provisions (EPC 1973):
-

Keyword:
"Amendments - added subject-matter: yes (main request); no (auxiliary request 1)"
"Novelty: yes (auxiliary request 1)"
"Inventive step: yes (auxiliary request 1)"

Decisions cited:
T 0142/05

Catchword:
-

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Beschwerdekammern | Boards of Appeal | Chambres de recours

Case Number: T 0071/10 **-** 3.3.03

D E C I S I O N
of the Technical Board of Appeal 3.3.03
of 1 March 2012

Appellant: (Opponent)
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Decision under appeal:
Interlocutory decision of the Opposition Division of the European Patent Office posted 19 November 2009 concerning maintenance of European patent No. 1313781 in amended form.

Composition of the Board:

Chairman: B. ter Laan
Members: O. Dury
C. Vallet

Summary of Facts and Submissions

I. The appeal by the opponent lies against the interlocutory decision of the opposition division announced on 21 October 2009 and posted on 19 November 2009 to maintain European patent No. EP 1 313 781 B1, based on application No. 01 964 275.0, corresponding to the international application published as WO 02/18464 A2, in amended form.

II. The application as filed contained 48 claims of which claims 1-3, 7, 8, 10, 14, 32 and 39 read as follows:

"1. Functionalized polymer beads prepared by
a) polymerizing a polyvinylidene monomer to form a self-crosslinked homopolymer; and then
b) polymerizing a functional monomer to covalently bond to said homopolymer."

"2. The functionalized polymer beads of claim 1 wherein said polyvinylidene monomer is selected from the group consisting of divinylbenzene, trivinylbenzene, divinylpyridine, divinyltoluene, divinylnaphthalene, ethyleneglycol dimethylacrylate and N, N-methylenediacrylamide."

"3. The functionalized polymer beads of claim 2 wherein said polyvinylidene monomer is divinylbenzene."

"7. The functionalized polymer beads of claim 1 wherein said functional monomer is selected from the group consisting of N-methyl-N-vinylacetamide, aminostyrene, methylacrylate, ethylacrylate, hydroxymethyl-

ethylacrylate, hydroxyethylacrylate and hydroxyethylacrylate."

"8. The functionalized polymer beads of claim 7 wherein said functional monomer is N-methyl-N-vinylacetamide."

"10. The functionalized polymer beads of claim 1 wherein said functional monomer is selected from the group consisting of N-(4-vinylbenzyl)-N, N-dimethylamine, vinylbenzyltrimethylammonium chloride, 4-vinylbenzoic acid, styrene sulfonic acid and methacrylic acid."

"14. A method for preparing a functionalized polymer bead, said method comprising the steps of:
a) polymerizing a polyvinylidene monomer to form a homopolymer; and then
b) polymerizing a functional monomer which covalently binds to said homopolymer."

"32. A method of separating an analyte from a solution comprising said analyte and a first solvent, said method comprising contacting said solution with functionalized polymer beads of claim 1 whereby said analyte is adsorbed onto said functionalized polymer bead."

"39. A method of separating analytes in a mixture by passing said mixture through a column of functionalized polymer beads of claim 1."

Claims 4, 5 and 11 were dependent claims directed to embodiments of claim 1. Claims 6, 9 and 12-13 were dependent on claims 5, 8 and 11, respectively.

Claims 15 to 20, 22, 23, 25, 26 and 29 were dependent on claim 14, claim 21 on claim 20, claim 24 on claim 23, claims 27 and 28 on claim 26, claim 30 on claim 29, and claim 31 on claim 30.

Claims 33, 34, 37 and 38 were dependent on claim 32, claim 35 on claim 33 and claim 36 on claim 34.

Claims 40 to 45 and 47 were dependent on claim 39, claim 46 on claim 45 and claim 48 on claim 47.

III. The granted patent was based on claims 1-47 corresponding to claims 1-17 and 19-48 as originally filed, respectively, and wherein each of claims 7 and 17 further comprised an amendment which is, however, not relevant for the current decision.

IV. A notice of opposition against the patent was filed on 4 April 2007, in which the revocation of the patent in its entirety was requested on the grounds of Art. 100(a) EPC (lack of novelty as well as lack of an inventive step) and Art. 100(c) EPC.

With the decision under appeal the patent was maintained on the basis of the auxiliary request filed during the oral proceedings before the opposition division. Said auxiliary request comprised 24 claims, claim 1 reading as follows (amendments as compared to claim 1 as filed are shown in **bold**, deletion in ~~strikethrough~~):

"1. Functionalized polymer beads prepared by
a) polymerizing **di**~~poly~~vinylbenzene monomer to form a self-crosslinked homopolymer; and then
b) polymerizing a functional monomer to covalently bond to said homopolymer **wherein the functional monomer is selected from the group consisting of N-methyl-N-vinylacetamide and styrene sulfonic acid**."

V. The decision under appeal was based, ***inter alia***, on the following documents:

D2: Römpp Chemie Lexikon, 9. Edition, 1991, Georg Thieme Verlag, Stuttgart, pages 3327-3329, "Pfropfcopolymere"
D3: WO 99/39823 A1
D9: WO 94/24236 A1

VI. In its decision the opposition division held that the main request did not meet the requirements of Art. 123(2) EPC and Rule 139 EPC. The auxiliary request was found to meet the requirements of Art. 123(2) EPC, Rule 139 EPC and Art. 84 EPC. In this regard, it was in particular decided that the amendment ":m" to "µm" (twice) on page 4, line 33 of the patent in suit was a correction allowable under Rule 139 EPC which did not infringe Art. 123(2) EPC. Novelty was acknowledged considering that none of documents D2, D3 and D9 cited against the novelty of the claimed subject-matter disclosed the combination of a divinylbenzene homopolymer backbone and side chains derived from either N-methyl-N-vinylacetamide or styrene sulfonic acid. Regarding inventive step, D3 was considered as closest prior art. The opposition division held that D3 taught away from preparing polymeric beads using

divinylbenzene homopolymer as polymeric backbone and that using either N-methyl-N-vinylacetamide or styrene sulfonic acid as side chains was not obvious in the light of the teaching of the prior art, in particular D2, D3 and D9.

VII. On 15 January 2010, the opponent (appellant) lodged an appeal against the above decision. The prescribed fee was paid on the same day. With the statement setting out the grounds for the appeal, received on 29 March 2010, the appellant requested that the patent be revoked. The following documents were also submitted:

D10: Printout of a datasheet of product 414565, taken from the internet site of Sigma-Aldrich on 22 February 2010,

D11: Material Safety Data Sheet of Sigma-Aldrich, product Divinylbenzene, 414565

The following document was further filed together with two other documents by letter dated 30 December 2011:

D12: Fischer et al., "Kinetik der radikalischen Polymerisation und Copolymerisation von N-vinyl-Nmethylacetamid", Makromol. Chem. 184, 1247-1254 (1983)

VIII. By letter of 28 June 2010, the respondent (patent proprietor) filed comments on the statement of grounds of appeal and requested the dismissal of the appeal (main request). By letter of 16 December 2011, the respondent requested, alternatively, the maintenance of the patent in amended form according to any of two auxiliary requests filed therewith and further

announced that he would not attend the oral proceedings before the Board. Additional experimental data given as Examples A-C, which were said to correspond to examples 14-16 of the application as filed, were filed simultaneously.

Auxiliary request 1 comprised a new page 4 of the patent specification and a set of 20 claims of which claims 1 and 3 read as follows (amendments as compared to claims 1 and 14 of the application as filed, respectively, shown in **bold**, deletion in ~~strikethrough~~):

"1. Functionalized polymer beads prepared by
a) polymerizing **di**~~poly~~vinylidene monomer to form a self-crosslinked homopolymer; and then
b) polymerizing a functional monomer to covalently bond to said homopolymer **wherein the functional monomer is N-methyl-N-vinylacetamide**."

"3. A method for preparing a functionalized polymer bead, said method comprising the steps of:
a) polymerizing **di**~~poly~~vinylidene monomer to form a self-crosslinked homopolymer; and then
b) polymerizing a functional monomer to covalently bond to said homopolymer **wherein the functional monomer is N-methyl-N-vinylacetamide**."

Claims 7 and 13 were, respectively, identical to claims 32 and 39 as originally filed.

Claims 2, 4-6 and 8-10 were dependent claims directed to embodiments of claims 1, 3 and 7, claim 11 depended on claim 9, claim 12 on claim 13 (sic). Claims 14-19 depended on claim 13 and claim 20 on claim 19.

On page 4, line 33 of the patent specification the term "µm" according to the granted patent was replaced, twice, by ":m".

IX. Oral proceedings were held on 1 March 2012 in the absence of the respondent, as announced.

X. The appellant's arguments may be summarised as follows:

Main request

Amendments

(a) The combination of divinylbenzene in step a) and either N-methyl-N-vinylacetamide or styrene sulfonic acid in step b) according to claim 1 was not specifically disclosed in the application as filed, neither in the claims nor in the description. The examples of the application as filed concerned specific porous polymeric beads and could not be considered as a valid basis for the subject-matter of claim 1 which encompassed both porous and non-porous polymeric beads in general. As a consequence, the main request did not meet the requirements of Art. 123(2) EPC.

Auxiliary request 1

Amendments

(b) The combination of divinylbenzene in step a) and N-methyl-N-vinylacetamide in step b) according to claim 1 did not meet the requirements of

Art. 123(2) EPC for the same reason as for the main request.

(c) The term "beads" according to claim 1 had an ambiguous meaning and could only be clarified by taking into account the patent specification. Replacing "µm" by ":m" in the patent specification led to an infringement of Art. 123(3) EPC. This objection was in line with decision T 142/05 (not published in OJ EPO), according to which the deletion of a feature in the description could lead to a broadening of the scope of protection of the claims.

Novelty

(d) The term "homopolymer" according to claim 1 encompassed copolymers. This conclusion was confirmed during the opposition proceedings by the comparative tests filed by the patent proprietor according to the teaching of D3, using a mixture of divinylbenzene monomers (letter of 19 November 2007). Consequently, the subject-matter of claim 1 was not novel over example 2 of D9.

Inventive step

(e) The closest prior art document D3 disclosed porous polymer beads prepared from a divinylbenzene copolymer wherein the vinyl groups were chemically modified with more hydrophilic functional groups. According to the examples of the patent in suit, "80% DVB" was used, meaning that the divinylbenzene beads were prepared using a

divinylbenzene copolymer, not a homopolymer, so that no effect of the use of homopolymers was shown. In support, two datasheets of commercial technical grade 80% divinylbenzene which also contained 18% ethylstyrene and <0,5% diethylbenzene (D10 and D11) were cited. Therefore, the problem to be solved was merely to provide alternative functionalised polymer beads to those of D3.

(f) According to D3, both divinylbenzene copolymers and homopolymers could be used, the latter not being preferred only because they were expensive (page 2, line 20 to page 3, line 6). It was therefore obvious to prepare beads having a divinylbenzene homopolymer backbone. The teaching of D3 was to graft divinylbenzene backbone polymers with nitrogen-containing monomers. To use N-methyl-N-vinylacetamide as the graft monomer was known from e.g. D12, and could not render the claimed subject-matter inventive either.

(g) During the oral proceedings before the Board the appellant agreed that it was technically possible to obtain divinylbenzene not being a mixture of isomers.

XI. The respondent had, in writing, essentially argued as follows:

Main request

Amendments

(h) A list of the basis for the amendments was provided and it was concluded that the requirements of Art. 123(2) EPC were met.

Auxiliary request I

Amendments

(i) Art. 123(2) EPC was complied with by the claims for the same reasons as for the main request.

(j) The amendment of "µm" in ":m" in the patent specification was based on the text of the application as filed. Further considering that the diameter of the polymer beads was specified nowhere in the claims, said amendment satisfied both Art. 123(2) EPC and Art. 123(3) EPC.

Novelty

(k) None of the cited documents disclosed functional polymeric beads prepared from a divinylbenzene homopolymer. Furthermore, none of these documents disclosed the specific combination of a divinylbenzene backbone modified using a N-methyl-

N-vinylacetamide functional monomer. The requirements of Art. 54 EPC were met.

Inventive step

(l) D3 was the closest prior art document. The examples of the patent in suit, Examples A-C (filed with letter dated 16 December 2011) and those filed with letter of 19 November 2007 showed that the claimed polymer beads provided improved recovery rates with the separation of analytes as compared to those of D3. The expression "80% DVB" as used in the examples of the patent in suit, referred to the solution containing the divinylbenzene (DVB), the remaining 20% being benzoyl peroxide and toluene. Therefore, the problem of improving the recovery rate had been effectively solved by the beads as claimed. D3 recommended the use of divinylbenzene copolymers instead of homopolymers and, thus, taught away from the subject-matter being claimed. Finally, none of the cited documents led to the specific combination of a divinylbenzene homopolymer backbone functionalised with N-methyl-N-vinylacetamide monomers. The claimed subject-matter was therefore inventive.

XII. The appellant (opponent) requested that the decision under appeal be set aside and that the European patent No. 1 313 781 be revoked.

The respondent (patent proprietor) requested in writing that the appeal be dismissed and that the patent be maintained as decided by the Opposition Division or,

alternatively, that the patent be maintained on the basis of one of the auxiliary requests filed with letter dated 16 December 2011.

XIII. The Board announced its decision at the end of the oral proceedings.

Reasons for the Decision

1. The appeal is admissible.

Main request

2. Amendments

2.1 Claim 1 corresponds to claim 3 as originally filed amended by specifying in step b) that "the functional monomer is selected from the group consisting of N-methyl-N-vinylacetamide and styrene sulfonic acid".

The only basis in the claims for polymer beads prepared from styrene sulfonic acid monomers in step b) is found in Claim 10 of the application as filed, wherein styrene sulfonic acid is disclosed in a list of equivalent alternative functional monomers. However, since claim 10 depends on claim 1 as filed (dealing with **poly**vinylidene monomers in step a)), it fails to disclose the specific combination of **di**vinylidene monomers in step a) and styrene sulfonic acid in step b) according to present claim 1.

In the description of the application as filed, divinyl benzene is indicated as one of two types of preferred

monomers for step a): di- and trivinylbenzene (see e.g. page 5, lines 25-27; page 6, lines 28-30). Styrene sulfonic acid is further cited within a list of equivalent alternative functional monomers on page 7, line 24 as originally filed. The specific combination of divinyl benzene for the backbone and styrene sulfonic acid as functional monomer, however, does not emerge from the application as filed and can only be obtained after combining parts of those two passages.

Among the examples of the application as filed, only example 12 deals with beads prepared from divinyl benzene in step a) and styrene sulfonic acid monomers in step b). However, it is derivable from the preparation method used that said beads are porous (see page 8, line 30 to page 9, line 2). The subject-matter of present claim 1 is not specific in that respect, but from page 3, line 4 of the patent specification (corresponding to page 3, lines 16-19 of the application as filed) it has to be seen as directed to both porous and non-porous beads. Therefore, the subject-matter of claim 1 corresponds to a generalisation that extends beyond example 12.

The subject-matter of claim 1, in particular the combination of divinyl benzene monomer in step a) and styrene sulfonic acid in step b) is, thus, not directly and unambiguously derivable from the application as filed.

2.2 The requirements of Art. 123(2) EPC are therefore not met so that the main request is not allowable.

Auxiliary request I

3. Amendments

3.1 Claim 1 corresponds to claim 3 as originally filed amended by specifying in step b) that "the functional monomer is N-methyl-N-vinylacetamide".

3.2 Both divinylbenzene monomer for step a) and N-methyl-N-vinylacetamide for step b) are disclosed as preferred monomers in the original description (page 5, lines 25-27; page 6, lines 28-30; page 7, lines 12-13). Hence, the combination of those monomers is directly and unambiguously supported by the application as filed.

No other objection under Art. 123(2) EPC was raised by the appellant and the Board sees no reason to take a different view. In particular, page 4, line 33 of the patent specification according to auxiliary request 1 now mentions ":m", thus returning to the original disclosure.

Therefore, Art. 123(2) EPC is complied with.

3.3 The amendments amount to a limitation of the definition of the monomer of each of steps a) and b) recited in the set of granted claims, i.e. divinylbenzene and N-methyl-N-vinylacetamide, respectively, thus leading to a limitation of the scope of the subject-matter claimed.

Page 4, lines 33 of the granted patent reads "The polymer beads **can have** a diameter in the range of 3 to about 100 µm…" (emphasis by the Board) which implies

that the limitation of the diameter is an optional feature of the polymer beads. Hence, an amendment of this passage can not modify the scope of the subject-matter claimed.

In decision T 142/05, it was considered that the deletion in the description of an important desired property of the claimed subject-matter led to an extension of the scope of protection. That is, however, a different situation from the case at issue in which the diameter range of the beads is an optional, hence not an important feature. Therefore, decision T 142/05 is not relevant for the present case.

For these reasons, the argument of the appellant that the amendment broadened the scope of the claimed subject-matter can not be followed and Art. 123(3) EPC is complied with.

4. Novelty

4.1 The polymer beads defined in the claims are prepared by polymerising N-methyl-N-vinylacetamide to bond covalently to a homopolymer obtained from a divinylbenzene monomer. There is no reason to deviate from the literal wording of the claims, according to which a divinylbenzene "homopolymer" is formed i.e. a polymer derived from a single type of divinylbenzene monomer, thus excluding divinylbenzene "copolymers".

4.2 None of the documents cited in the proceedings discloses a divinylbenzene homopolymer according to present claim 1.

4.2.1 In particular, example 2 of D9, relied upon by the appellant in the written proceedings, deals with a poly(styrene-co-divinylbenzene) grafted with poly(methacrylic acid) (page 14, lines 16-17) i.e. a backbone made of a divinylbenzene copolymer, which is not a homopolymer according to the subject-matter now being claimed.

4.2.2 Document D3 discloses the preparation of porous hydrophobic divinylbenzene copolymers having pendant vinyl groups, the latter being thereafter chemically modified so as to form different functional groups having a greater hydrophilicity and greater biocompatibility than those of the vinyl groups (claims 1-3). The claims of D3 specifically deal with divinylbenzene copolymers. In the examples of D3 a mixture of divinylbenzene (para- and meta-isomers) and p-ethylstyrene or butylacrylate was used, i.e. a divinylbenzene copolymer was prepared. Also, N-methyl-N-vinylacetamide is not mentioned as a suitable functional monomer (see e.g. claims 4-6 and page 6, lines 1-12 or the examples of D3). Hence, D3 does not disclose a divinylbenzene homopolymer and the use of N-methyl-N-vinylacetamide as functional monomer.

4.3 Therefore, the subject-matter of claim 1 is novel. Consequently, each of claims 2-20, which refer directly or indirectly to the subject-matter of claim 1, also fulfil the requirements of Art. 54 EPC.

5. Inventive step

5.1 Closest prior art

5.1.1 The patent in suit relates to functionalised polymeric beads. Such beads are known from D3, which deals with a method of production of polymeric particles for purification of physiological liquids of an organism e.g. hemodialysis. The particles are made of functionalised divinylbenzene copolymers. Both parties and the opposition division considered D3 as closest prior art. There is no reason to deviate from that view.

5.2 Problem to be solved

5.2.1 The problem addressed by the patent in suit is to provide highly crosslinked preformed rigid particles having hydrophilic, cation or anion exchange properties, suitable for separation applications such as liquid chromatography and solid phase extraction (paragraphs [0001], [0004] and [0005] of the patent in suit).

5.3 Solution

The solution to the above problem resides in the functionalised polymer beads defined in present claim 1. The subject-matter claimed differs from D3 in that the beads are prepared using:

- a divinylbenzene homopolymer backbone;

- N-methyl-N-vinylacetamide as functional monomer.

5.4 Success of the solution - Problem effectively solved

5.4.1 In examples 2-6 and 16 and Figures 1-5 and 8-9 of the patent in suit and in Examples A-C (filed with letter dated 16 December 2011) functionalised polymer beads based on divinylbenzene are used to separate polar organic analytes, in particular cis- from trans-organic acids, the recovery being improved as compared to polymeric beads prepared using either a nonfunctionalised divinylbenzene polymer or octadecyl modified silica.

5.4.2 The divinyl benzene polymer used in the examples is described as a "DVB polymer" produced by conventional suspension polymerisation using "80% DVB", benzoyl peroxide as the initiator, and toluene as the pore forming agent (paragraph [0018] of the patent in suit). Although the patent acknowledges that its examples are illustrative of the "invention" i.e. the subject-matter claimed (see page 8, lines 23-24 of the application as filed; paragraph [0017] of the granted patent), the patent does not indicate what exactly is meant by "80% DVB". Even if 80% technical grade divinylbenzene containing ethylstyrene was commercially available at that time (D10, D11), that does not mean that that product had actually been used in the examples of the patent in suit. That situation is not changed by the experiments filed by the respondent (letter dated 19 November 2007). It is equally possible that an 80% solution in toluene was used, as stated by the respondent. Moreover, not only was it uncontested that divinylbenzene existed without any other monomers present, the appellant also agreed that it was technically possible to prepare divinylbenzene monomer

that is not a mixture of isomers. Therefore, the argumentation of the appellant that the examples of the patent in suit did not show the use of divinylbenzene homopolymer beads, can not be followed.

In view of the above, the examples of the patent in suit are considered to be illustrative of the invention i.e. beads prepared from a divinylbenzene homopolymer, in accordance with claim 1.

5.4.3 Therefore the technical problem effectively solved by the claimed subject-matter may be formulated as providing functionalised polymer beads having good separation properties of polar organic analytes including cis-trans isomers of organic acids.

5.5 Obviousness

5.5.1 It remains to be decided whether or not it was obvious to solve the above identified problem by modifying the teaching of D3 in such a way as to arrive at claim 1 i.e. whether or not it was obvious, starting from D3, to prepare polymeric beads using a divinylbenzene homopolymer backbone and N-methyl-N-vinylacetamide as functional monomer.

5.5.2 None of the documents on file deals with the separation of isomers of organic acids. Therefore, none of those documents - and in particular D3 - could suggest the solution proposed by claim 1 in order to solve that part of the problem addressed by the patent in suit.

5.5.3 D3 states in the paragraph bridging pages 2 and 3 that divinylbenzene homopolymers are rather expensive and

that the more available technical product contains up to 30-40 % of ethyl vinyl styrene. Hence, in D3 divinylbenzene copolymers are used rather than homopolymers merely for economical reasons.

D3 further discloses on page 6, lines 1-6 the modification of the divinylbenzene polymer backbone by grafting hydrophilic polymers chains by a radical polymerisation of various "water soluble" monomers. However, D3 does not disclose the monomers now being claimed, namely N-methyl-N-vinylacetamide. Hence, D3 fails to provide a hint to the specific combination of divinylbenzene homopolymer and N-methyl-N-vinylacetamide according to present claim 1, in particular to do so to in order to solve the technical problem identified in section 5.4.3 above.

5.5.4 None of the other documents cited in the proceedings discloses the use of N-methyl-N-vinylacetamide to covalently bond to divinylbenzene homopolymers for making functionalized polymer beads.

Regarding D12, that document was filed by the appellant approximately two months before the date of the oral proceedings before the Board. Considering that D12 neither discloses functionalised polymer beads, nor divinylbenzene homopolymers, nor does it deal with the separation techniques of analytes, it is not considered ***prima facie*** highly relevant. Consequently, D12 is not admitted to the proceedings (Art. 13(1) of the Rules of Proceedings of the Boards of Appeal).

5.5.5 Hence, it was not obvious to solve the above identified technical problem by preparing polymer beads as defined in claim 1.

5.6 Therefore, the subject-matter of claim 1 complies with the requirements of Art. 56 EPC. Consequently, each of claims 2-20, which depend directly or indirectly on the subject-matter of claim 1, fulfils the requirements of Art. 56 EPC.

6. Auxiliary request 1 therefore being allowable, there is no need to consider the other auxiliary request.

Order

For these reasons it is decided that:

1. The decision under appeal is set aside.

2. The case is remitted to the department of first instance with the order to maintain the patent on the basis of auxiliary request 1 filed on 16 December 2011 and a description to be adapted where necessary.

The Registrar: The Chairman:

E. Görgmaier B. ter Laan