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Datasheet for the decision
of

Case Number: T 0782/14 - 3.3.06
Application Number: 07114167.5
Publication Number: 1889653
IPC: B01D71/10, B01D71/12, C08B15/00
Language of the proceedings: EN

Title of invention:
Crosslinked cellulosic nanofiltration membranes

Patent Proprietor:
MILLIPORE CORPORATION

Opponent:
SARTORIUS STEDIM BIOTECH GMBH

Headword:
Nanofiltration membrane/MILLIPORE

Relevant legal provisions:
EPC Art. 52(1), 54
RPBA Art. 12(4)
Keyword:
Novelty - main request (no)
Auxiliary claim requests filed with statement of grounds - admitted (no) - claim requests could and should have been filed in opposition proceedings

Decisions cited:
T 1685/07, T 2513/11

Catchword:
DECISION of Technical Board of Appeal 3.3.06
of 30 November 2017

Appellant: MILLIPORE CORPORATION
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Decision under appeal: Decision of the Opposition Division of the
European Patent Office posted on 20 January 2014
revoking European patent No. 1889653 pursuant to
Article 101(3) (b) EPC.

Composition of the Board:
Chairman B. Czech
Members: M. Maremonti
C. Heath
Summary of Facts and Submissions

I. The appeal lies from the decision of the Opposition Division to revoke the European Patent No. 1 889 653.

II. The patent was granted with a set of fourteen claims, independent claim 1 reading as follows:

"1. A nanofiltration membrane comprising a porous support and a crosslinked cellulose membrane integral with the support layer, wherein said membrane retains solutes of greater than 200 Daltons, wherein said membrane comprises a cellulose ultrafiltration membrane reacted with a multifunctional crosslinking reagent through hydroxyl groups in the anhydroglucose units, under conditions whereby sufficient hydroxyl groups are left unreacted to provide a hydrophilic membrane and wherein said membrane is organic solvent resistant".

Claims 2 to 9 are dependent on claim 1 and are directed to specific embodiments of the membrane of claim 1.
Claims 10 to 14 are directed to a method of removing organic solutes using the membrane of claim 1.

III. The following documents were cited inter alia during the opposition procedure:

E3: DE 10 2004 053 787 A1;

E4: US 5,522,991 A;

E10: A. P. Broek et al, "Characterisation of Hemodialysis Membranes by Inverse Size Exclusion Chromatography"; Journal of Membrane Science, 99, 1995; pages 217 to 228; and

The Opposition Division came inter alia to the following conclusions:

- Article 100(c) EPC did not prejudice the maintenance of the patent as granted.

- The subject-matter of granted claim 1 lacked novelty over document E3.

- The subject-matter of claim 1 according to the then pending first auxiliary request did not involve an inventive step in view of E3 taken as the closest prior art, in combination with E4.

IV. In its statement of grounds, the Appellant (Patent Proprietor) contested the reasoning given by the Opposition Division and defended the patent as granted. It nevertheless also filed two sets of amended claims as auxiliary requests I and II.

V. In its reply, the Respondent (Opponent) rebutted the arguments of the Appellant and maintained that the subject-matter of claim 1 as granted extended beyond the content of the application as filed and that its subject-matter lacked novelty and inventive step. It also submitted that claim 1 according to auxiliary request I was objectionable under Article 123(2) EPC as well as for lack of novelty and inventive step. Claim 1 of auxiliary request II was also objectionable under Article 123(2) EPC and for lack of inventive step.

VI. The parties were summoned to oral proceedings. In preparation therefor, the Board issued a communication stating its preliminary opinion on certain points, inter alia calling into question novelty over E3 and indicating that the admittance of the two auxiliary claim requests filed with the statement of grounds
might be subject to the Board's discretion under Article 12(4) RPBA.

VII. In a further letter dated 06 October 2017, the Respondent asked for the non-admittance of auxiliary claim requests I and II into the proceedings pursuant to Article 12(4) RPBA. It maintained all its previous objections and raised a further objection under Article 123(2) EPC against claim 1 of auxiliary request II.

VIII. In a further letter dated 30 October 2017, the Appellant inter alia expressed its disagreement with the preliminary opinion of the Board regarding novelty over E3, rebutted the objections raised by the Respondent and presented its arguments in support of the admittance of its auxiliary claim requests I and II.

IX. By letter dated 28 November 2017, the Appellant withdrew its request for oral proceedings and announced that its representative would not attend the oral proceedings.

X. Oral proceedings before the Board were held on 30 November 2017 in the absence of the Appellant. The debate focused on the interpretation of claim 1 (main request) and on the novelty of its subject-matter over the disclosure of document E3.

XI. Final Requests

The **Appellant** requested in writing that the decision under appeal be set aside and that the patent be maintained as granted (Main Request) or on the basis of the claims of one of auxiliary requests I and II, both filed with the grounds of appeal. The Appellant also requested the remittal of the case
for the examination of inventive step in the event that the Main Request met the novelty requirement.

The Respondent requested that the appeal be dismissed. It also requested non-admittance of Appellant's Auxiliary (claim) requests I and II.

XII. The **Appellant**'s arguments of relevance for the present decision can be summarised as follows.

**Novelty - Main Request**

- Document E3 did not disclose a "*nanofiltration membrane*" within the meaning of claim 1 at issue.

- E3 did not disclose that the membrane described therein was capable of retaining multivalent ions and, in general, small molecules having a molecular weight (MW) as low as 200 Da, as required by claim 1 at issue.

- The membrane of E3 was not said to contain "*sufficient hydroxyl groups left unreacted to provide a hydrophilic membrane*", as required by claim 1 at issue.

- The membrane of E3 was not "*organic solvent resistant*" within the meaning of claim 1.

- E3 did not unambiguously disclose that the membrane described in paragraph [0050] of E3 is "*integral*" with the fabric support, as required by claim 1 at issue.

- The ultrafiltration membrane used in E3 as the starting material was subjected to a tempering step before being cross-linked. Such a tempering step was not part of the production method of the
nanofiltration membrane according to claim 1 at issue, and implied differences in the membrane structure.

Admittance of Auxiliary Requests I and II

- The Respondent only raised an objection more than three years after its reply to the statement of grounds of the Appellant. This late-filed objection should thus not be considered.

- Auxiliary claim Requests I and II were duly filed within the appeal period.

- The Appellant had been confronted with the situation that the subject-matter of claim 1 was deemed to lack novelty for the first time during the oral proceedings before the Opposition Division.

- There had been no indication that the Opposition Division would admit any further auxiliary request during oral proceedings, let alone a request including features taken from the description.

- It was unrealistic from a practical point of view to consider that the Appellant could have filed Auxiliary Requests I and II at issue already before the Opposition Division.

XIII. The Respondent essentially counter-argued as follows.

Main Request - lack of novelty - claim 1

- Filtration membranes having a molecular weight cut-off (MWCO in the following) in the range of from 200 to 1000 Da had to be considered as "nanofiltration membranes" within the meaning of
the patent in suit, particularly in view of paragraph [0003].

- E3, describing such membranes, thus disclosed a "nanofiltration membrane" within the meaning of claim 1 at issue.

- The feature of claim 1 stating "wherein said membrane retains solutes of greater than 200 Daltons" had not to be understood in the sense that the MWCO of the membrane had to be 200 Da. As a matter of fact, no such a membrane was disclosed in the patent in suit.

- This feature had to be interpreted in the sense that the claimed membrane had to be suitable for retaining multivalent ions and organic molecules with a molecular weight in the range of from 200 to 1000 in accordance with the conventional definition of a nanofiltration membrane.

- The appropriateness of this understanding of claim 1 was also apparent in view of claim 9, dependent on claim 1 and requiring retention of solutes with a size greater than 400 Da.

- The membrane of E3 was "hydrophilic", as required by claim 1 at issue. In fact, the examples of E3 were carried out using aqueous solutions. Moreover, the examples of E3 showed that by increasing the crosslinking time from 24 to 168 hours the permeate flux was reduced and vitamin B12 retention was increased. This meant that at least the membranes obtained after only 24 hours crosslinking still had hydroxyl groups left unreacted, as required by claim 1 at issue.
- The membranes of E3 were also "organic solvent resistant" as required according to claim 1 at issue. This was evident not only from the fact that the same membrane material (cellulose-based) as according to the contested patent was used, but also from the observation that the steps carried out in producing the membrane of E3 were performed in organic solvents.

- Paragraph [0050] of E3 clearly referred to a membrane that is integral with the fabric support as evidenced by the term "vliesverstärkt" used in this paragraph.

- The presence of a tempering step in the production of the ultrafiltration membrane of E3 was irrelevant for the question of novelty of claim 1, which referred to a cellulose ultrafiltration membrane in general.

Admittance of Auxiliary claim Requests I and II

- Auxiliary Requests I and II were not filed in opposition.

- These requests were divergent from each other and gave rise to additional issues under Article 123(2) EPC.

- It was not relevant that the objection against the admittance of these requests was only raised at a later stage of the appeal proceedings. The decision on the (non-)admittance of late-filed requests was always a matter for the Board's discretion under Article 12(4) RPBA.
Reasons for the Decision

Main request - Lack of novelty

1. Document E3

1.1 E3 indisputably discloses (paragraphs [0050] and [0051]; page 8, Table 1) crosslinked cellulose hydrate semipermeable membranes comprising a porous support layer ("vliesverstärkte Membran"). Said membrane is obtained by subjecting a supported cellulose acetate membrane to a treatment involving tempering and saponification followed by crosslinking with a butanediol diglycidyl ether solution in a solvent comprising diglyme.

Some of the membranes were crosslinked for 24 hours and display \( R_{812} \) (vitamin B12 rejection) values ranging from 92.0 % to 98.8 %, corresponding to "cut-off classes" of 500 Da or 1 kDa (E3, paragraph [0042]).

1.2 The Board thus holds that these membranes display all the features of claim 1, even those not explicitly mentioned in E3.

2. According to the Appellant this was not the case, since in its view the membrane disclosed in E3 differed from the membranes according to claim 1 at issue in several aspects as follows.

2.1 First, E3 did not disclose a "nanofiltration membrane" within the meaning of claim 1 at issue.

On the one hand, the term "nanofiltration" was not mentioned as such in E3, which only related to ultrafiltration membranes. On the other hand, E3 did not contain information indicating that the membranes disclosed therein were capable of retaining multivalent
ions and, in general, small molecules having a molecular weight (MW) as low as 200 Da, as implied by the feature "nanofiltration membrane" of claim 1 at issue.

2.2 Second, the membrane of E3 did not contain "sufficient hydroxyl groups left unreacted to provide a hydrophilic membrane" as required by claim 1 at issue.

2.3 Third, E3 did not mention that the membrane disclosed was "organic solvent-resistant", as required by claim 1 at issue.

2.4 Fourth, the example described in paragraph [0050] of E3 did not clearly and unambiguously disclose that the membrane used therein was "integral" with the fabric support as required by claim 1 at issue.

2.5 Fifth, the ultrafiltration membrane used as starting material according to paragraph [0050] of E3 was subjected to a tempering step before being crosslinked. However, such a tempering exerted a decisive influence on the cut-off value of the resulting membrane as evidenced by Table 1 on page 8 of E3. No such tempering step was involved in preparing the nanofiltration membrane according to the patent in suit. The membrane of claim 1 therefore implicitly differed structurally from the crosslinked ultrafiltration membrane disclosed in E3.

3. The Board, however, does not find any of these arguments convincing for the following reasons.

3.1 The feature "nanofiltration"

3.1.1 It is indisputably common general knowledge that the term "nanofiltration" identifies a filtration process lying in between reverse osmosis and ultrafiltration in
terms of the size of the solutes retained. More particularly, the term "nanofiltration membrane" normally designates membranes used to remove molecules in the molecular weight range of from 200 and 1000 Da, as acknowledged in the patent in suit (paragraph [0003]). Moreover, according to document E11 (page 302, penultimate line), considered by both parties to represent relevant common general knowledge, nanofiltration membranes are defined as having a pore size < 2 nm. According to E11 (page 286, Figure VI-3), vitamin B12 is retained by nanofiltration membranes. In this connection, the Board notes that the vitamin B12 molecule, used as tracer in membrane characterization, is considered to have a radius of 0.78 nm, as apparent e.g. from by E10, page 221, Table 1.

3.1.2 The supported crosslinked cellulose hydrate membranes of E3 (paragraphs [0027], [0040], [0050] to [0053]) are obtained by subjecting a cellulose acetate precursor membrane to tempering and saponification step, and then to crosslinking. The molecular weight cut-off (MWCO) values of the membranes obtained after crosslinking for 24 hours (see 1, supra) are of the same order of magnitude as the ones obtained after crosslinking according to the contested patent (page 8, Table 1, third column) which display MWCO values from 544 to 1113 Da, corresponding to the molecular weight at 90% rejection, determined using dextrans (see paragraphs [0052] to [0055] of the patent in suit).

3.1.3 Bearing in mind the usual meaning of the term "nanofiltration" (3.1.1, supra), the cellulose hydrate membranes of E3, as obtained after crosslinking for 24 hours, the Board holds are undoubtedly "nanofiltration membranes" within the meaning of claim 1 of the patent in suit.
3.2 The feature "retains solutes of greater than 200 Daltons"

3.2.1 The Board holds that bearing in mind the relevant common general knowledge (see 3.1.1, supra), this feature must be understood as setting a lower limit to the molecular weight range of solutes retained by the claimed membrane. This understanding is fully in line with the indications in paragraph [0003] of the patent in suit, according to which nanofiltration membranes are (emphasis added) "used to remove multivalent ions and small organic molecules in the molecular weight range of approximately 200-1000 Daltons".

The Board thus holds that any membrane able to retain to a significant extent substances with a molecular weight in this range meets the requirement defined by this feature.

3.2.2 However, paragraph [0030] of the contested patent, stating that "solute greater than about 200 kD [sic! - read 200 Da] are retained (rejected) on the upstream side of the membrane" could give the impression that the feature in question actually requires the MWCO of the claimed membrane to be set at 200 Da.

Such a narrow interpretation of the feature in question is, however, not supported by the whole content of the patent in suit.

- As a matter of fact, although "Membrane 1" described in Example 1 of the patent appears to substantially reject MgSO₄ and raffinose (page 8, Table 2), it is not shown that said membrane, or any other membrane disclosed in the patent, has an
MWCO as low as 200 Da (see the higher MWCO values indicated in Table 1 of the patent).

Moreover, claim 9, which is dependent on claim 1 and thus directed to a more preferred embodiment, requires specifically that the claimed membrane "retains solutes of a size greater than 400 Daltons" (emphasis added by the Board). The Board holds that the wording of claim 9 actually confirms that the membranes according to claim 1 need not have a MWCO value as low as 200 Da.

3.2.3 Given the results in terms of $R_{B12}$ and MWCO as reported in E3 (see 1, supra), the Board holds that at least the membrane of E3 crosslinked for 24 hours and displaying a $R_{B12}$ of 98.8%, corresponding to the cut-off class 500 Da (i.e. $R_{B12} > 96 \%$), undoubtedly qualifies as a "nanofiltration membrane" meeting the requirement "retains solutes of greater than 200 Daltons".

3.3 The features regarding crosslinking "under conditions whereby sufficient hydroxyl groups are left unreacted to provide a hydrophilic membrane"

3.3.1 The experimental comparison of membranes crosslinked for 24 hours and for 168 hours, respectively, as reported in Table 1 of E3 clearly shows that an increase in the crosslinking time generates a decrease in the flow-rate across the membrane and an increase in the $R_{B12}$ values.

3.3.2 For the Board, absent any specific counter-argument, these results clearly indicate that an increase in the crosslinking time leads to a kind of tightening of the membrane, only attributable to the additional crosslinking of further hydroxyl groups of the
cellulosic membrane material. Therefore, at least in the case of the shorter crosslinking duration of 24 hours, a certain number of hydroxyl groups of the membrane of E3 must have been left unreacted, as required by claim 1 of the contested patent.

3.4 The feature "organic solvent resistant"

3.4.1 The Board holds that in the context of the subject-matter of the patent in suit this relative feature can only mean that the claimed membrane must be able to be operated in environments comprising organic solvents.

3.4.2 According to E3 (cf. paragraphs [0018], [0025], [0027] and example 1 in [0050]), the various steps the membranes disclosed therein are subjected to in the course of the process leading to the ultimately obtained membrane, i.e. tempering, saponifying and crosslinking, may be carried out in organic solvent solutions. More particularly, the crosslinking of the membranes disclosed in paragraph [0050] of E3 is carried out in a liquid comprising substantial amount of diglyme, i.e. an organic solvent.

3.4.3 The Board thus concludes that the requirement in question is also met by the membrane of E3 obtained after 24 hours crosslinking duration and having an $R_{B12}$ value of 98.8%.

3.5 The feature "integral with a support layer" (emphasis added)

3.5.1 E3 does not expressly mention membranes which are "vliesverstärkt" without the fleece being integral with the actual separation membrane. From paragraphs [0015] ("mit integrierer Vliesverstärkung") and [0040] ("integral vliesverstärkt") and from claim 15
("integral vliesverstärkt") of E3, it can, instead, be
gathered that the precursor membrane ("vliesverstärkt")
referred to in paragraph [0050] of E3 is implicitly a
composite membrane comprising a porous fleece support
material "integral" with, i.e. penetrated by, the
cellulosic material forming the actual separation
membrane.

3.5.2 The membranes exemplified in paragraph [0050] of E3
thus also meet this requirement of claim 1 as granted.

3.6 Different method for the preparation of the membrane

3.6.1 The patent in suit does indeed not disclose a tempering
step, as required according to E3, to be carried out in
preparing the membrane according to the invention. Such
a step is, however, not excluded by the wording of
claim 1 as granted, which merely requires that "a
cellulose ultrafiltration membrane [is] reacted with a
multifunctional crosslinking reagent". This wording
imposes no limitations in terms of method steps that
may be carried out before the (final) crosslinking.

3.6.2 Hence, the mere fact that the membrane of E3 obtained
after 24 hours crosslinking duration and having a \(R_{B12}\)
of 98.8% is obtained by tempering and saponifying a
cellulose acetate ultrafiltration membrane, does not
justify considering that its structure must be
different from the structure of membranes defined by
claim 1.

4. In summary, none of the features of claim 1 invoked by
the Appellant distinguishes the membranes of claim 1
from (at least) the membrane disclosed in paragraph
[0050] of E3 obtained after 24 hours crosslinking
duration and having a \(R_{B12}\) of 98.8%. 
In the Board's judgement, the subject-matter of claim 1 thus lacks novelty over the disclosure of document E3 (Articles 52(1) and 54 EPC).

5. The main request of the Appellant cannot be allowed.

(Non)-admissibility of Auxiliary Requests I and II

6. The amended sets of claims according to Auxiliary claim Requests I and II (AR1, AR2 hereinafter) were filed for the first time with the the Appellant's statement of grounds.

6.1 As justification for the filing of AR1 and AR2 only in appeal, the Appellant stated that the Opposition Division had not issued a preliminary opinion before the oral proceedings. The Patent Proprietor had thus only been confronted with the finding that the subject-matter of granted claim 1 lacked novelty over E3 at the oral proceedings before the Opposition Division.

The Appellant also submitted that there had been no indication that the Opposition Division would admit any further auxiliary request during said oral proceedings, in particular a request including features taken from the description. It was "unrealistic from a practical point of view" to consider that auxiliary requests as now on file should already have been filed in the first instance proceedings.

Moreover, the Respondent only objected to the admittance of these requests more than three years after its reply to the Appellant's statement of grounds, and only after having received the Board's communication issued in preparation for the oral proceedings. Such a late objection should not be considered.
6.2 These arguments do not convince the Board that such auxiliary requests, aiming to overcome the novelty objection based on E3, could (and should) not have been filed earlier, i.e. during the opposition proceedings, and at the latest at the oral proceedings before the Opposition Division.

6.2.1 An objection for lack of novelty over document E3 had already been raised in the notice of opposition. The Appellant thus has had the opportunity to react to this novelty objection by filing amended claims as auxiliary requests at an early stage of the opposition proceedings.

6.2.2 The Appellant chose, instead, not to file any auxiliary claim request with its reply the the notice of opposition, but to defend the patent as granted. Only one month before the oral proceedings before the Opposition Division, the Appellant filed an auxiliary claim request in response to the added matter objection raised by the Opponent, claim 1 of this request comprising an additional feature taken from the description.

6.2.3 During the oral proceedings before the Opposition Division (see the minutes, points 6.7 to 6.10), the Appellant then was given the opportunity to file a new auxiliary claim request in order to overcome the novelty objection based on E3. It filed a new such claim request, incorporating into claim 1 as granted the features of granted dependent claim 5 (specifying in more detail the material of the "support layer"). This request was admitted by the Opposition Division, but the subject-matter of claim 1 as amended was then found to lack an inventive step (decision under appeal, point 5). It is, however, noteworthy that before the
decision was taken, the Opposition Division had asked the Patent Proprietor once more whether it had further requests (minutes point 6.17).

In this respect, the Board observes, moreover, that an (alleged) expectation of the Patent Proprietor that a further request incorporating some feature from the description would not be admitted by the Opposition Division cannot justify only making an attempt to this end on appeal.

6.2.4 From the case history it is thus apparent that the Appellant has had ample opportunity to file AR1 and AR2 at issue, and should have attempted to do so during the opposition proceedings, at the latest during the oral proceedings before the Opposition Division. There is nothing "unrealistic" in this finding, considering that in inter partes opposition proceedings parties usually have divergent opinions regarding at least some of the raised issues. Each party must thus envisage the possibility that the Opposition Division may adopt the view of the adverse party and must present fallback positions (claim requests) accordingly, without first awaiting the Opposition Division's decision.

6.2.5 Moreover, AR1 and AR2 diverge both

- from the earlier auxiliary claim request filed before the Opposition Division (claim 1 specifying further the "support layer" material),

as well as

- from each other,

since according to AR1 a list of alternative crosslinking reagents extracted from the
description is incorporated into claim 1, whereas according to AR2 features of dependent claims 2 and 3 relating to a "surface charge" imparted to the surface of the membrane are incorporated into claim 1.

According to the case law of the Boards of Appeal, both types of divergence justify non-admittance of claim requests, considering the consequential shifts in issues to be addressed, as in the present case (see e.g. T 1685/07 of 4 August 2010, Reasons, 6; T 2513/11 of 2 October 2014, Reasons, 3.4 to 3.6).

6.2.6 Furthermore, the amendments in the claims according to both AR1 and AR2 generated additional objections under Article 123(2) EPC raised by the Respondent, never addressed before.

6.3 Although the Respondent only asked for non-admittance of AR1 and AR2 after receiving the Board's communication in preparation for the oral proceedings, the Board has, pursuant to Article 12(4) RPBA, the discretionary power to hold inadmissible requests which could (and, in the present case, should) have been presented in the first instance proceedings.

6.4 Taking into account the above considerations the Board, in the exercise of its discretion under Article 12(4) RPBA, thus decided not to admit AR1 and AR2 into the proceedings.

Conclusions

7. None of the Appellant's requests is both admitted and allowable.
Order

For these reasons it is decided that:

The appeal is dismissed.

The Registrar: The Chairman:

D. Magliano B. Czech

Decision electronically authenticated