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Datasheet for the decision of 22 January 2009

Case Number: T 1221/06 - 3.3.09

Application Number: 95939403.2

Publication Number: 0744435

IPC: C08J 9/06

Language of the proceedings: EN

Title of invention:
Water-absorbent resin, process for production thereof, and water-absorbent resin, composition

Patentee:
NIPPON SHOKUBAI CO., LTD.

Opponent:
BASF Aktiengesellschaft

Headword:
-

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

Relevant legal provisions (EPC 1973):
-

Keyword:
"Main request: Inventive step (no) - Priorities not valid"
"Auxiliary request: Inventive step (yes) - Priorities valid"

Decisions cited:
-

Catchword:
-

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Case Number: T 1221/06 **-** 3.3.09

D E C I S I O N
of the Technical Board of Appeal 3.3.09
of 22 January 2009

Appellant: (Patent Proprietor)	NIPPON SHOKUBAI CO., LTD. 1-1, Koraibashi 4-chome Chuo-ku Osaka-shi Osaka-fu 541 (JP)
Representative:	Jacob, Reuben Ellis R G C Jenkins & Co. 26 Caxton Street London SW1H 0RJ (GB)
Respondents: (Opponent)	BASF Aktiengesellschaft -Patentabteilung - C6- Carl-Bosch-Strasse 38 D-67056 Ludwigshafen (DE)
Representative:	Reitstötter - Kinzebach Patentanwälte Postfach 86 06 49 D-81633 München (DE)
Decision under appeal:	**Decision of the Opposition Division of the European Patent Office orally announced on 5 May 2006 and posted 7 June 2006 revoking European patent No. 0744435 pursuant to Article 102(1) EPC.**

Composition of the Board:

Chairman: P. Kitzmantel
Members: W. Ehrenreich
M-B. Tardo-Dino

Summary of Facts and Submissions

I. Mention of the grant of European patent No. 0 744 435 in respect of European patent application No. 95 939 403.2, filed on 8 December 1995 in the name of *Nippon Shokubai Co., Ltd.* as International application No. PCT/JP95/02523 (published as WO-A 96/017884) was announced on 3 September 2003 (Bulletin 2003/36).

The patent, entitled *"Water absorbent resin, process for production thereof, and water-absorbent resin composition"* was granted with thirty-seven claims. Claims 1, 5 and 10 read as follows:

"1. A porous water-absorbent resin having an average particle diameter measured by JIS standard sieves of 200 to 600 μm, an elevated surface cross-link density, an average pore diameter measured by electron microscopy of 10 to 500 μm, an absorbent capacity (measured as specified in the description) of not less than 25 g/g 60 minutes after the initiation of absorption under pressure, a water-soluble component content (measured as specified in the description) of not more than 15 weight percent, and a residual monomer content (measured as specified in the description) of not more than 500 ppm.".

"5. A water-absorbent composition, comprising:

a porous water-absorbent resin according to any of claims 1 to 4; and
an inorganic powder.".

"10. A process for producing a porous water-absorbent resin, comprising the steps of:

dispersing a solid blowing agent having an average particle diameter (measured with a laser-type particle size distribution apparatus) of 1 to 100 µm in an aqueous monomer solution containing an unsaturated monomer and a cross-linking agent; and polymerizing the unsaturated monomer.".

Claims 2 to 4 were dependent on Claim 1, Claims 6 to 9 were dependent on Claim 5 and Claims 11 to 33 were, either directly or indirectly, dependent on Claim 10.

The set of claims as granted further included independent Claims 34 and 35, which were directed to an acrylic salt of an azo compound of formulae (1) or (2) as specified in Claim 17, as well as independent Claim 36 (and Claim 37 dependent thereupon) concerning a water-absorbent product including the composition according to Claims 6 to 9.

II. Notice of opposition was filed by

BASF Aktiengesellschaft

on 28 May 2004.

The opposition was based on Article 100(a) EPC, it being alleged that the claimed subject-matter was not novel and did not involve an inventive step. The Opponent based its objections *inter alia* on the following documents:

D3 WO-A 95/17455

D4 EP-A 0 644 207

D9 Experimental report of the Opponent reworking example 18 of D3 and showing measurement results of the absorbent capacity of the resulting water-absorbent resin

D9a CD-Rom containing a video documentation of the experiments reported in D9

D9b Microscope photograph of the blowing agent particles formed during reworking example 18 of D3.

The Opponent further alleged that the product according to Claim 1 of the patent was available to the public before the effective filing date, basing its submission on documents D5 to D8.

Furthermore, the validity of the priority JP 30518594 dated 8 December 1994 was contested.

In the oral proceedings before the Opposition Division, which were held on 5 May 2006, the Opponent raised further objections under the opposition grounds according to Articles 100(b) and 100(c) EPC.

III. With its decision orally announced on 5 May 2006 and issued in writing on 7 June 2006 the Opposition Division revoked the patent.

The decision was based on a set of eleven claims (replacing the set of claims as granted and the auxiliary request filed with the letter dated 7 February 2006), submitted during the oral proceedings as basis for the Proprietor's sole request, Claims 1 to

9 corresponding to granted Claims 1 to 9 and Claims 10 and 11 corresponding to granted Claims 36 and 37.

The Opposition Division did not admit the late filed opposition grounds under Articles 100(b) and 100(c) into the proceedings and did not accept the alleged prior public use as relevant prior art. A reasoned statement in this respect was given in the decision.

Furthermore, the Opposition Division held that the claimed subject-matter was not entitled to the priority dates of either 8 December 1994 (JP 30518594) or 24 March 1995 (JP 6542795). It was held that no disclosure was to be found in these priority documents about the average particle diameter range of 200 to 600 microns for the claimed water-absorbent resin particles. Therefore, D3, published after the two priority dates, and D4, published after the first but before the second priority date, were considered to represent prior art according to Article 54(2) EPC for the product claimed in Claim 1.

The Opposition Division considered the claimed water-absorbent resin novel over the prior art but did not acknowledge its inventive step over a combination of D3 with D4.

As to inventive step it was argued that example 18 of D3 disclosed a porous water-absorbent resin which was prepared by polymerizing and crosslinking sodium acrylate in the presence of a solid blowing agent precursor. In the Opposition Division's view the experimental report D9, reworking example 18 of D3, the video clip D9a and the microscope photographs D9b

demonstrated that the absorbent capacity of the resulting resin was in the range claimed in Claim 1 and the particle size of the solid blowing agent was in the technically relevant range of 1 to 100 microns.
The only distinguishing feature, namely the elevated surface crosslink density obtained by an additional surface crosslinking step, was considered obvious in view of D4, which taught that surface-crosslinking enhanced the absorbent capacity under pressure of water-absorbent resins.

IV. On 4 August 2006 the Patent Proprietor (hereinafter: the Appellant) lodged an appeal against the decision of the Opposition Division. The Statement of the Grounds of Appeal was submitted on 17 October 2006.
Enclosed with the grounds of appeal were three sets of claims as bases for a new main request and auxiliary requests 1 and 2.
Further sets of claims as bases for auxiliary requests 3 to 8 were submitted with the letter dated 24 November 2008.

In the oral proceedings before the Board, held on 22 January 2009, the Appellant withdrew the main request and auxiliary requests 1 and 2 in reaction to the provisional opinion of the Board expressed in its communication of 13 January 2009 questioning the admissibility of these requests under Rule 80 EPC.

The Appellant filed a new main request on the basis of the set of process Claims 1 to 24 according to the previous auxiliary request 3, amended by deletion of the passage *"separating a hydrogel product from the*

polymerization medium and" from Claim 1 and by deletion of Claims 23 and 24.
Furthermore, a new auxiliary request on the basis of process Claims 1 to 24 according to the previous auxiliary request 4 was filed wherein Claim 1 was amended as above, Claims 7, 17, 20 and 23, 24 were deleted and the subsequent claims were renumbered accordingly.

Auxiliary requests 5 to 8 were withdrawn.

Claims 1 and 7 according to the new main request read as follows:

"1. A process for producing a porous water-absorbent resin, comprising the steps of:

dispersing a solid blowing agent having an average particle diameter (measured with a laser-type particle size distribution apparatus) of 1 μm to 100 μm in an aqueous monomer solution containing an unsaturated monomer and a cross-linking agent;

polymerizing said unsaturated monomer;

drying the hydrogel to produce the porous water-absorbent resin; and

treating the porous water-absorbent resin of 100 parts by weight with a surface cross-linking agent of 0.01 to 5 parts by weight to form a covalent bond."

"7. A process according to any one of claims 1 to 6, wherein the blowing agent is used in an amount of 0.005 to 25 parts by weight based on 100 parts by weight of the unsaturated monomer.".

Claim 1 of the new auxiliary request corresponds to Claim 1 of the new main request with the addition of the following feature at the end of the claim after "a covalent bond;":
"and treating an area of the surface of the porous water-absorbent resin with a cationic compound after treatment with the cross-linking agent to form an ionic bond.".
Claim 7 as above was no longer part of the set of Claims 1 to 19 according to the auxiliary request.

V. In its letter of response to the Appellant's grounds of appeal the Respondent cited the new documents

D10 WO 95/02002 (a document cited under Article 54(3) EPC)
D11 graph representing the results of a particle size analysis of $CaCO_3$ and $(MgCO_3)_4Mg(OH)_2 \cdot 5H_2O$

and submitted that the claimed process was not novel over D10.

In the oral proceedings the Respondent declared that it had no objections to the admission into the proceedings of the Appellant's late-filed new requests.
However, the validity of the two priorities for the subject-matter claimed in Claim 7 of the new main request was contested. It was argued that the amount of 0.005 to 25 parts by weight of the blowing agent according to Claim 7 was not disclosed in any of the priority documents. Therefore, D3 and D4 became prior art according to Articles 54(2) and 56 EPC with the consequence that the subject-matter of Claim 7 was rendered obvious by their combined disclosure.

No formal objections or objections as to lack of novelty or lack of inventive step were raised against the subject-matter claimed according to the new auxiliary request.
Furthermore, the objections under the opposition grounds of Articles 100(b) and 100(c) EPC were no longer maintained.

VI. The Respondent's arguments as to lack of inventive step of the subject-matter of the main request were as follows:

A process for preparing a porous water-absorbent resin was disclosed in D3. According to example 18 of this document the process comprised the following steps:

- providing an aqueous solution of acrylic acid, neutralized with NaOH;
- adding a crosslinking agent and a 2,2'-azobis(2-methylpropionamidine)dihydrochloride (ABAH) blowing agent precursor to the solution; the amount of the blowing agent being in the range given in Claim 7;
- polymerizing the unsaturated monomer;
- drying the resulting hydrogel at 150°C to obtain, after crushing and sieving, a porous water-absorbent resin of a particle size range of from 150 to 850 µm.

The average tea bag retention value of the water-absorbent resin, determined in the experiments reported in D9, was 40.2 g/g and exactly corresponded to the value given in Table 1 of D3. This value was fully

embraced by the range of not less than 25 g/g given for the water-absorbent resin in Claim 1 as granted.

The video clip D9a clearly showed that the aqueous sodium acrylate solution, after adding the ABAH precursor, became turbid within 25 to 30 seconds by precipitation of the diacrylate salt of the ABAH blowing agent at the temperature of the reaction solution of about 10°C, i.e. before the exothermic polymerization reaction started and the temperature rose to 100°C. The average particle diameter of the precipitated needle-like crystals of the diacrylate salt was within the range given in Claim 1, as could be seen from the microphotographs in D9b.

Therefore, the claimed process differed from that described in example 18 of D3 only in that the resulting porous water-absorbent resin was treated with a surface-crosslinking agent forming a covalent bond.

When starting from D3, the problem to be solved by the claimed process was to further reduce the content of water-soluble components in the water-absorbing resin and to improve its water-retention capacity.

The solution to this problem by treating the resin with a surface-crosslinking agent was, however, known from D4 which stated on page 2, lines 43 to 45, that surface-crosslinking leads to an improvement of the absorption under pressure, of the gel strength and to a reduction of the amount of extractables.

VII. The Appellant objected to the admission of documents D10 and D11 into the proceedings and provided the following counterarguments as to inventive step of the subject-matter of the main request:

The experimental report D9, reworking example 18 of D3, and the video D9a did not give any information as to the particle size of the blowing agent at the outset or during the progress of the polymerization reaction. Furthermore, it was not derivable from D9b whether the particles shown in the microphotographs were representative of the average particle diameter as required by Claim 1 (emphasis by the Board). Therefore, no indication as to the relevance of the claimed average particle size of the blowing agent during the polymerization reaction was derivable from D3.

Because, as stated in paragraphs [0041] and [0042] of the patent specification, the claimed particle size of 1 to 100 µm for the solid blowing agent was essential for adjusting the desired average pore diameter and water absorbent capacity of the resulting water-absorbent resin, the claimed process was not obvious by combining D3 with D4.

VIII. The Appellant requested that the decision under appeal be set aside and the patent be maintained on the basis of Claims 1 to 22 of the main request filed during the oral proceedings (corresponding to auxiliary request III filed with the letter of 24 November 2008 and amended during the oral proceedings), or alternatively on the basis of Claims 1 to 19 of the auxiliary request (corresponding to auxiliary request IV filed with the

letter of 24 November 2008 and amended during the oral proceedings).

IX. The Respondent requested that the appeal be dismissed.

Reasons for the Decision

1. The appeal is admissible.

2. *Admission of the new requests into the proceedings*

In view of the fact that the Respondent did not object to the admission of the new requests and because the restrictions in the claims lead to a straightforward simplification of the case, the Board exercised its discretion in accordance with Article 13(1) of the Rules of Procedure of the Boards of Appeal (OJ 11/2007, 537) and admitted the requests into the proceedings.

3. It appeared during the oral proceedings that the documents D10 and D11 were not relevant for the issues which were decisive for the decision on the main request. Thus, the Board decided not to admit them. Likewise, documents D5 to D8, on which the alleged prior public use of the product claimed in Claim 1 as granted was based, are of no relevance to the process claimed in the main and auxiliary requests. There is therefore no need to consider whether or not these documents belong to the state of the art.

Further, since the Respondent's request to consider the late-filed opposition grounds according to Articles 100(b) and 100(c) EPC has been withdrawn, this issue need also not be addressed.

4. *Priority*

4.1 Main Request

The Board agrees with the Respondent that the subject-matter of Claim 7 of the main request is not entitled to the claimed priorities. In particular, paragraph [0027] of the document JP 305185/1994, representing the first priority, and paragraph [0018] of the document JP 065427/1995, representing the second priority, do not disclose the range of 0.005 to 25 parts by weight of the blowing agent, based on 100 parts by weight of the unsaturated monomer. Instead, a range of 0.01 to 5 weight percent is disclosed in both priority documents.

Consequently, D3 and D4 become prior art according to Articles 54(2) and 56 for the subject-matter of Claim 7 of the main request.

4.2 Auxiliary Request

The first and second priorities are, however, valid for the subject-matter according to the auxiliary request, which no longer includes the embodiment of Claim 7 of the main request. This was not contested by the Respondent.

For this request D3 and D4 are therefore only prior art according to Article 54(3) EPC and cannot be considered for the assessment of inventive step.

5. *Novelty*

5.1 Main Request

After the decision of the Board not to admit D10 and D11 into the proceedings the Respondent no longer maintained its objections as to lack of novelty.
However, because, as will be shown below, the subject-matter of Claim 7 lacks an inventive step over a combination of D3 with D4, consideration of this issue is unnecessary.

5.2 Auxiliary request

As was not contested by the Respondent, the claimed process is novel over the prior art.
The combination of the process steps, including the polymerization of the unsaturated monomer and the subsequent surface crosslinking reaction followed by the treatment with a cationic compound to form an ionic bond, is not disclosed in any of the cited documents.

Inventive step

6. The patent in suit

The patent is concerned with the preparation of a porous water-absorbent resin which is *inter alia* suitable for use in sanitary materials, such as paper diapers or incontinence pads (patent specification,

paragraph [0001]). The preparation method should result in a resin having an enhanced liquid permeability and dispersion under pressure in combination with improved absorption rate and absorbent capacity after the initiation of absorption under pressure (paragraphs [0017] and [0020/21]).

7. Inventive step of the subject-matter according to the main request

7.1 The claimed process

According to Claim 7, in conjunction with Claim 1 to which it refers back, the desired properties of the resin are achieved by the following process steps:

(a) a solid blowing agent having an average particle diameter of 1 µm to 100 µm is dispersed in an aqueous unsaturated monomer solution; the amount of the blowing agent required by Claim 7 being 0.005 to 25 parts by weight based on 100 parts by weight of said monomer; the solution further contains a crosslinking agent;
(b) the unsaturated monomer is polymerized;
(c) the resulting hydrogel is dried to produce the porous water-absorbent resin; and
(d) the resin is treated with a surface crosslinking agent in an amount of 0.01 to 5 parts by weight per 100 parts by weight of the resin.

7.2 The closest prior art

D3 is representative of the closest prior art. Example 18 of D3 describes a process for the preparation of a porous water-absorbent resin including the process steps:

(a) adding an ABAH blowing agent precursor to a neutralized acrylic acid solution containing a triallyl amine crosslinker;
(b) polymerizing the acrylic monomer; and
(c) crushing and drying the resulting hydrogel to obtain, after milling and sieving, the water-absorbent resin.

The amount of the ABAH added in step (a) lies in the range claimed in Claim 7 (see Table 1).

The Board also follows the explanations of the Respondent with respect of D9, D9a and D9b (see point VI) that shortly after the addition of the ABAH precursor and before the onset of the polymerization reaction a solid precipitate of the 2,2'-azobis(2-methylpropionamidine)diacrylate blowing agent is formed, whose average particle size lies within the claimed range of 1 to 100 µm. In this regard the Respondent pointed to the scale depicted in the microphotograph D9b, which shows that the length of all crystals measured is between 30 and 90 µm, in consequence whereof the average particle size must lie within the claimed range.

The Appellant argued that it was not shown by the evidence D9a and D9b that the particle size of the blowing agent was in the claimed range during the whole polymerization process.

This argument, however, is not convincing.

Firstly, Claim 1 does not indicate that the average particle size of the blowing agent has to be in the range of 1 to 100 µm during the whole polymerization process. It only requires that the particle size is in the claimed range once the blowing agent is dispersed in the aqueous monomer solution, i.e. before the polymerization starts.

Secondly, the addition of the ABAH precursor to the monomer solution according to example 18 of D3, followed by the precipitation of the acrylate salt as shown by D9a, exactly corresponds to the technique of example 1 of the patent specification, which describes in paragraph [0134] the generation of solid particles of the blowing agent seven minutes after the addition of the ABAH precursor to the monomer solution.

7.3 Problem and solution

In the light of the above, the claimed process differs from that described in example 18 of D3 only in that the additional surface-crosslinking step (d) is performed.

A comparison between example 1 and example 6 of the patent specification shows in conjunction with Table 1 that the absence of a surface-crosslinking step

(example 1) leads to a porous (blown) water-absorbent resin having a reduced water-absorbent capacity under pressure (last but one column of the table).

Therefore, in view of this experimental evidence, the problem to be solved is seen in the provision of a process for preparing a water-absorbent resin which leads to a product having an enhanced water-absorbent capacity under pressure.

7.4 Obviousness

The solution to the problem, namely to treat the water-absorbent resin in an additional step with a surface-crosslinking agent is obvious from D4. This document, dealing with the preparation of blown microcellular superabsorbent polymers, indicates at page 4, lines 39 to 42, that surface-crosslinking of the core polymer improves its absorption under pressure.

Hence, the skilled person would arrive at the process claimed in Claim 7 by combining D3 with D4.

The subject-matter of Claim 7 therefore lacks an inventive step and the main request is therefore not allowable.

8. Inventive step of the subject-matter according to the auxiliary request

As stated above under point 4.2, documents D3 and D4, representing prior art according to Article 54(3) EPC, cannot be used for the assessment of inventive step.

According to the process of the auxiliary request the reaction step (d) is followed by a further step

(e) wherein an area of the porous water-absorbent resin is treated with a cationic compound to form an ionic bond.

This additional step, providing an alternative process for the preparation of a superabsorbent polymer, is, however, nowhere disclosed or rendered obvious from the prior art in combination with steps (a) to (d). The Respondent did not raise any objections to this conclusion.

The subject-matter of the auxiliary request is therefore based on an inventive step.

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 Claims 1-19 of the auxiliary request filed during the oral proceedings after any necessary consequential amendment of the description.

The Registrar | The Chairman

G. Röhn | P. Kitzmantel