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Datasheet for the decision of 4 September 2013

Case Number: T 0224/09 - 3.3.03

Application Number: 99122561.6

Publication Number: 1002806

IPC: C08F6/00

Language of the proceedings: EN

Title of invention:
Production process for hydrophilic polymer

Patent Proprietor:
NIPPON SHOKUBAI CO., LTD.

Opponent:
BASF Aktiengesellschaft

Headword:

Relevant legal provisions:
EPC Art. 54, 56

Keyword:
Novelty - (yes) Main Request
Inventive step - (yes) Main Request

Decisions cited:

Catchword:

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Case Number: T 0224/09 - 3.3.03

D E C I S I O N
of Technical Board of Appeal 3.3.03
of 4 September 2013

Appellant: (Patent Proprietor)	NIPPON SHOKUBAI CO., LTD. 1-1, Koraibashi 4-chome Chuo-ku Osaka-shi, Osaka 541-0043 (JP)
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Appellant: (Opponent)	BASF Aktiengesellschaft -Patentabteilung - C6- Carl-Bosch-Strasse 38 D-67056 Ludwigshafen (DE)
Representative:	Thalhammer, Wolfgang Reitstötter, Kinzebach & Partner (GbR) Patentanwälte Sternwartstrasse 4 81679 München (DE)
Decision under appeal:	**Interlocutory decision of the Opposition Division of the European Patent Office posted on 11 December 2008 concerning maintenance of the European Patent No. 1002806 in amended form.**

Composition of the Board:

Chairman: B. ter Laan
Members: D. Marquis
C. Brandt

Summary of Facts and Submissions

I. The appeals by the patent proprietor and the opponent lie from the decision of the opposition division posted on 11 December 2008 maintaining European patent N° 1 002 806 (based on application number 99 122 561.6) in amended form.

II. The patent was granted with a set of 5 claims of which independent claims 1 and 2 read as follows:

"1. A production process for a hydrophilic polymer, comprising the steps of: obtaining a hydrogel polymer by aqueous solution polymerization of a hydrophilic monomer; and then drying the hydrogel polymer; with the production process being **characterized in that** the drying step includes the steps of:

partially drying the hydrogel polymer under normal pressure at a material temperature of not higher than 90°C until the water content of the hydrogel polymer reduces to 15-40 weight %; and then
heat-aging the hydrogel polymer by keeping the hydrogel polymer for not shorter than 10 minutes in a state where the change of the water content of the hydrogel polymer is within 5 weight % and where the material temperature is in the range of 70-120°C; and then finish-drying the hydrogel polymer until the water content of the hydrogel polymer is in a range of 0-10 weight %."

"2. A production process for a hydrophilic polymer, comprising the steps of: obtaining a hydrogel polymer by aqueous solution polymerization of a hydrophilic monomer; and then drying the hydrogel polymer;

with the production process being **characterized in that** the drying step includes the steps of:

partially drying the hydrogel polymer under normal pressure at a material temperature of not higher than 90°C until the water content of the hydrogel polymer reduces to 15-40 weight %; and then
heat-aging the hydrogel polymer by keeping the hydrogel polymer for not shorter than 10 minutes in a state where the water content of the hydrogel polymer is in the range of 15-40 weight % and where the material temperature is in the range of 70-120°C; and then
finish-drying the hydrogel polymer until the water content of the hydrogel polymer reduces to a desired value."

Claims 3 to 5 were directed to preferred embodiments of claims 1 or 2.

III. A notice of opposition against the patent was filed in which the revocation of the patent was requested on the grounds according to Article 100(a) EPC (lack of novelty and lack of inventive step).

IV. By a decision announced orally on 11 November 2008, the opposition division maintained the patent in amended form on the basis of the auxiliary request filed during the oral proceedings. The decision was based *inter alia* on the following documents:

D2: Modern Superabsorbent Polymer Technology, Wiley-VCH, ISBN 0-471-19411-5, pages 44, 72, 73 and 87 to 93.
D3: EP 0 289 338 A2
D5: Experimental evidence. Example 8 of D3.
D6: Experimental evidence. Example 9 of D3.

In the decision it was held that the main request (claims as granted) was not novel over D3 as shown by D5 and D6. The auxiliary request however met the requirements of Articles 84, 123(2) and 123(3) EPC and was also novel and inventive because none of the cited documents disclosed or suggested that the combination of features of claim 1 and/or claim 2 would result in a production process for a hydrophilic polymer having low residual monomer content and absorption capacity change ratio together with low extractable content change ratio. In particular D2 taught away from heat-aging the hydrogel polymer for not shorter than 10 minutes.

V. On 21 January 2009, the patent proprietor lodged an appeal against the decision of the opposition division and paid the prescribed appeal fee on the same day. The statement setting out the grounds of the appeal was filed on 20 April 2009. The patent proprietor requested that the patent be maintained on the basis of the main request (claims as granted) or any of seven auxiliary requests filed with the statement of grounds of the appeal.

VI. On 20 February 2009, the opponent lodged an appeal against the decision and paid the prescribed appeal fee on the same day. The statement setting out the grounds of the appeal was filed on 14 April 2009. The opponent requested that the patent be revoked.

VII. With a letter dated 9 September 2009, the patent proprietor submitted further arguments and filed nine auxiliary requests.

VIII. By letter dated 1 September 2009, the opponent requested the dismissal of the proprietor's appeal.

Three new documents were filed, of which two were experimental reports D7 and D8.

IX. On 26 April 2013, the parties were summoned to oral proceedings to be held on 4 September 2013.

X. By letter of 5 August 2013, the proprietor submitted further arguments concerning novelty and inventive step of the main request.

XI. Oral proceedings were held on 4 September 2013.

XII. The patent proprietor's arguments may be summarised as follows:

- Novelty

D3 did not disclose the heat-aging step as set out in the claims of the patent in suit. D3 did not disclose the water content and the temperature of the hydrophilic polymer either, both of which were essential features of claims 1 and 2. The repetition of examples 8 and 9 of D3 in D5 and D6 could not be used to provide the information missing in D3 because the drying process disclosed in those documents was not the same as that used in D3. In particular the drying apparatus was significantly different. D3 did also not provide the process and product information necessary to perform its examples 8 and 9. D5 and D6 were therefore based on assumptions regarding essential parameters such as the determination of the water content and the temperature of the polymer at the start of the drying process. D7 and D8 were also no accurate repetition of examples 8 and 9 of D3, so that there was no evidence that D3 disclosed all the present features in combination.

- Inventive step

D2 was not the closest prior art as it did not disclose a three-step drying process nor the combination of parameters as claimed in the patent in suit. D2 only disclosed the drying process of a hydrogel.

Starting from D3, the problem to be solved was to provide, with good productivity, hydrophilic polymers having a low residual monomer content, while preventing the absorption capacity and the extractable content of the polymer from being deteriorated due to drying. Examples 1 and 8 and comparative example 15 of the patent in suit showed that that problem was effectively solved.

The solution given in claims 1 and 2 of the patent in suit was not obvious in view of the cited prior art because none of the documents provided a motivation to prevent the deterioration of the absorption capacity and the extractable content of the polymer by means of the claimed measures.

XIII. The opponent's arguments may be summarised as follows:

- Novelty

D3 took away the novelty of claims 1 and 2. Examples 8 and 9 of D3 had been repeated and the results of the measurements of the polymer temperature as well as its water content during the drying process were provided in D5 and D6. The drying process disclosed in D5 and D6 was essentially the same as that described in D3. Any differences did not significantly influence the drying process and the properties of the polymers. The

temperature of the polymer at the start of the drying process was not relevant, as confirmed by a comparison of the drying profile of the polymer of D6 with that of D7. Although the temperature of the polymer at the start of the drying process was different for those two polymers, the product temperature and the water content were very similar after 10 minutes into the drying process. D5 and D6 therefore disclosed valid repetitions of examples 8 and 9 of D3 and showed that the drying profile of the hydrogel polymer could be seen as a three-step drying process so that all the features of present claims 1 and 2 were implicitly present in D3. Therefore D3 destroyed the novelty of the claimed processes.

- Inventive step

D2 was the closest prior art. D2 indicated that a drying temperature of 60 to 100°C and a water content of 10 to 60% were favourable for a low residual monomer content. Table 1 of the patent in suit did not allow a comparison of the different compositions because the absolute values of the absorption capacity and extractable content were not disclosed. The technical problem solved in the patent in suit was therefore to provide an alternative process for the production of hydrophilic polymers, in particular regarding the drying step. In view of the technical details disclosed in D2, present claim 2 was an arbitrary choice within the possibilities offered by D2. Moreover, in order to arrive at a further lowering of the rest monomers, the skilled person would use a longer drying time than in D2. Therefore, starting from D2, the technical problem solved in the patent in suit was to provide an alternative process and the claimed solution to that problem was obvious in view of D2.

Starting from example 11 of D3, which disclosed a two-step drying process, the technical problem was only the reduction of the extractable content change ratio. The residual monomer and the absorption capacity of the polymer obtained in comparative example 15 of the patent in suit were comparable to those of the polymer of example 11 of D3. According to D2, a higher temperature resulted in a higher extractable content ratio, so that the skilled person would be motivated to vary the drying temperature in order to influence the extractable content of the polymer. The absorption would only be measured on a dried polymer. The subject-matter of claims 1 and 2 of the patent in suit therefore lacked an inventive step over D3 in combination with D2.

XIV. The patent proprietor requested that the decision under appeal be set aside and that the patent be maintained as granted (main request), or alternatively that the patent be maintained on the basis of any of the auxiliary requests 1 to 9 filed with letter of 9 September 2009.

XV. The opponent requested that the decision under appeal be set aside and that the European patent No. 1002806 be revoked. The opponent further requested not to admit the auxiliary requests 1 to 7 and 9 as filed with letter dated 9 September 2009 to the proceedings.

Reasons for the Decision

1. The appeal is admissible.

Main request (as granted)

2. Novelty

2.1 D3 discloses a method for the production of a hydrophilic polymer having a small residual monomer content from a hydrated gel polymer by drying the hydrated gel polymer characterised in that the drying is achieved by contacting the gel polymer with a gas containing steam and having a dew point in the range of 50° to 100°C at a temperature in the range of 80° to 250°C (claim 1).

In Examples 8 and 9 of D3 a method is disclosed for the production of a hydrophilic polymer by reacting acrylic monomers containing sodium acrylate, acrylic acid and methylene-bis-acrylamide in a kneader. As polymerization initiators, ammonium persulfate and sodium hydrogensulfite were added to the contents of the kneader. The resulting hydrated gel polymer was then removed from the kneader. This polymer was found to contain unaltered acrylic acid and sodium acrylate in a total concentration of 10000 ppm. After polymerization, the polymer was dried in a hot-air drier by heating it with gases kept at a temperature of 120°C (Example 8) or 100°C (Example 9) and adjusted to a dew point of 80°C (Example 8) or 60°C (Example 9) until the water content thereof fell to 10% by weight, to give rise to a water-swellable polymer. The dried product was then transferred to a heat exchanger and crushed to obtain a water-swellable polymer powder

which was tested for residual monomer content and adsorption ratio.

It was not disputed that the production process of examples 8 and 9 of D3 does not disclose i) the water content of the hydrogel polymer and ii) the material temperature of the polymer during the drying procedure which are essential features of both claims 1 and 2 of the main request.

2.2 D5 and D6 describe a repetition by the opponent of the process for the production of a hydrophilic polymer of example 9 and example 8 of D3, respectively. Acrylic monomers containing sodium acrylate, acrylic acid and methylene-bis-acrylamide as in examples 9 and 8 of D3, were reacted in a kneader. As polymerization initiators, ammonium persulfate and sodium hydrogensulfite were added to the contents of the kneader. After polymerization, the resulting hydrated gel polymer was removed from the kneader and dried on a conveyor belt drier. Temperature and dew point of the drying gas were set before the drying process.

In D5 and D6, the water content in the gel used as starting point for the monitoring of the water content was stated to be performed according to the method found on page 6 of the contested patent (Water content). During the drying process, the water content and the material temperature of the hydrogel polymer were monitored, with the following results:

	D5	
Drying time [min.]	Product temperature [°C]	Water content [%]
0	25	59,9
15	63	51,8
30	63	41,5
40	63	33,7
50	70	26,2
60	81	19,6
75	95	12,5
90	97	8,6

	D6	
Drying time [min.]	Product temperature [°C]	Water content [%]
0	58	60,3
10	88	47,1
20	89	29,4
25	92	21,9
30	100	15,6
35	110	10,9
40	115	8,0
50	119	5,7
60	120	5,0

2.3 The opponent could not explain why the product temperature at the start of the drying procedure (drying time t = 0 min) differed in D5 (25°C) and in D6 (58°C). It could also not be clarified if those values had to be selected before the drying process or if they were the result of the production process of the hydrophilic polymer. In any case, examples 8 and 9 of D3 did not mention the temperatures of the hydrophilic polymer at the start of the drying process, so that it

was not known if either of D5 and D6 properly reflected the processes of those examples.

2.4 The opponent argued that the product temperature at the start of the drying process had no influence on the drying process as a whole. In support he submitted D7 in which a temperature of 33°C was used at the start of the drying step, showing no significant difference of the product temperature and water content after 10 minutes of drying, compared to D6. However, D7 is not a repetition of Example 8 of D3, which was not disputed by the opponent, and the reaction conditions are different from those of D6 and D5. The same is true for D8. Therefore, on the basis of D7 and D8 no conclusions can be drawn regarding the effect of the starting temperature on the drying process as a whole. As a consequence, it cannot be concluded that the product temperature and water content of the hydrogel polymer during drying as measured in D5 and D6 actually correspond to the values achieved in examples 9 and 8 of D3, respectively.

2.5 In the absence of any evidence that the production processes of examples 8 and 9 of D3 disclose all the features of the processes of claims 1 and 2 of the patent in suit, it cannot be concluded that the claimed processes lack novelty over D3.

2.6 Also in view of the other cited documents, the Board accepts that the subject-matter claimed in the main request is novel, so that the requirements of Article 54 EPC are met.

3. Inventive step

3.1 The patent in suit relates to a production process for a hydrophilic polymer such as a water-soluble or water-swellable polymer, and to a process for producing a hydrophilic polymer having a low residual monomer content while retaining the properties of a hydrogel polymer resultant from polymerization (paragraph [0001]).

3.2 D2 is a general publication on modern superabsorbent polymer technology. It is stated in D2 that drying is an essential step in the manufacturing processes of such polymers. D2 in particular discloses the effect of the continuation of the polymerization during drying (page 44, Chapter 2.5.4.1). However, the provided pages (pages 44, 72, 73, 87 to 93) do not mention a multi-step drying process of the hydrogel polymer.

3.3 D3 aims at providing a hydrophilic polymer having a small residual monomer content and a method for the production of such a polymer from a hydrated gel polymer (page 2, lines 47 to 50). A drying temperature of 80 to 250°C is mentioned (page 4, lines 29 to 31). The drying can be carried out continuously or in more steps (page 4, lines 42 to 47). Example 11 of D3 discloses a drying process in two steps, in which in a first step the polymer is dried with gas at a temperature of 120°C until the solids content reached 80%. Then the drying is continued to a water content of not more than 10% by applying hot air at a temperature of 180°C.

3.4 D3 explicitly mentions a multi-step drying process of a hydrogel polymer, it is directed to a similar purpose as the patent in suit and it requires less structural

modifications in order to arrive at the production process claimed in the main request than D2, which does not mention a multi-step drying process of the hydrogel polymer. Therefore, D3 is considered to be the closest prior art.

3.5 The technical problem mentioned in the patent in suit was to provide hydrophilic polymers and in particular water-absorbent resins having a residual monomer content of not higher than 300 ppm, an absorption capacity change ratio not higher than 10 under no load and an extractable content change ratio not higher than 100 (paragraph [0029]).

The question to be answered is whether that problem has been solved vis-à-vis the closest prior art document D3.

3.6 The solution to the problem cited above resides in the process steps defined in claims 1 and 2 and more specifically in the heat-aging step performed by keeping the hydrogel polymer for not shorter than 10 minutes in a state in which either the change of the water content of the hydrogel polymer is within 5 weight % and the material temperature is in the range of 70-120°C (claim 1) or by keeping the hydrogel polymer for not shorter than 10 minutes in a state where in which the water content of the hydrogel polymer is in the range of 15-40 weight % and the material temperature is in the range of 70-120°C (claim 2).

3.7 The drying process of comparative example 15 of the patent in suit can be seen as representative of example 11 of the closest prior art D3 because the same drying steps were applied. Since the same product (production

example 1) was dried according to claims 1 and 2 of patent in suit (examples 1 and 8) and according to D3 (comparative example 15), those examples provide a proper comparison regarding the effect of the drying steps on the product properties.

3.8 In examples 1 and 8 of the patent in suit, the hydrophilic polymer was dried for 20 minutes at a temperature of 80°C with a through-flow batch type drying oven (partial drying step). The material temperature was 70°C and the water content 25 weight %. Next, the resultant aggregated semi-dry product was placed into a stainless beaker and then heated at 100°C for 1 hour (heat-aging step). After being heated, the semi-dry product had a material temperature of 95°C and a water content of 23 weight %. Thereafter, the lumpish aggregate was finish-dried at 150°C for 30 minutes with the through-flow batch type drying oven used in the first drying step. Examples 1 and 8 of the patent in suit show that the processes according to claims 1 and 2 lead to hydrophilic polymers with a residual monomer content of 130 ppm and 290 ppm, an absorption capacity change ratio under no load of 2,4 % and 2 % (the value of 0 % for example 1 in Table 1 being obviously wrong) and an extractable content change ratio of 26 % and 18,5 % (Table 1).

3.9 In comparative example 15 the hydrophilic polymer was dried in a through-flow batch type drying oven until the water content reduced to 20 weight % by blowing a gas having a temperature of 120°C and a dew point of 80°C. The polymer thus obtained had a residual monomer content of 280 ppm while the absorption capacity change ratio under no load was 30 % and the extractable content change ratio 381 %.

3.10 The examples show that the drying process according to the patent in suit (examples 1 and 8) leads to a powder displaying smaller changes in absorption capacity under no load and extractable content ratio after drying than the powder obtained by the drying process of comparative example 15, which is representative of the closest prior art D3.

Therefore, the problem effectively solved by the subject-matter of claims 1 and 2 over D3 may be seen as to provide hydrophilic polymers having a lower absorption capacity change ratio under no load and a lower extractable content change ratio after drying.

3.11 It remains to be decided whether the solution to that problem, as defined in claims 1 and 2, was obvious in view of the prior art.

3.12 The absorption ratios of the polymer powders obtained in D3 are reported in Table 1. In D3 however, the absorption capacity of the polymers before drying was not determined so that no comparison of the absorption capacity before and after drying is possible. Hence, D3 does not indicate that the absorption properties of the hydrophilic polymer may be deteriorated by drying and therefore does not suggest that deterioration can or should be reduced. Also, the extractable content of the hydrophilic polymers is not mentioned in D3. Therefore, there is no hint in D3 at solving the problem defined above by applying the specific drying steps of present claims 1 and 2.

3.13 D2 also does not point to the solution according to the patent in suit. D2 reviews drying processes of hydrophilic polymers, particular emphasis being put on the reduction of residual monomer content. The effect

of the drying temperature on the swelling capacity and extractable content is analysed in Table 3.4 on page 89. However, D2 does not disclose any measurement of the polymer properties before and after drying so that there is no indication in D2 of the possible deterioration of the absorption properties and extractable content of the polymer and no motivation to prevent such a deterioration. Moreover, D2 does not describe drying in multiple steps. Therefore, D2 does not contain any hint to change the drying process described in D3 so as to arrive at the subject-matter of present claims 1 and 2.

3.14 For those reasons, the subject-matter of claims 1 and 2 of the main request is inventive.

3.15 Also starting from D2 as the closest document, as the opponent did, one would not arrive at any other conclusion since neither of D2 or D3, taken alone or in combination, discloses the specific combination of drying steps of present claims 1 or 2.

3.16 Therefore, the main request fulfils the requirements of Article 56 EPC.

4. Since the main request is allowable, there is no need to go into the admissibility of any of the auxiliary requests.

Order

For these reasons it is decided that:

1. The decision under appeal is set aside.

2. The patent is maintained unamended.

The Registrar: The Chairman:

E. Goergmaier B. ter Laan

Decision electronically authenticated