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Datasheet for the decision of 8 September 2021

Case Number: T 1304/17 - 3.3.03

Application Number: 04798045.3

Publication Number: 1689790

IPC: C08F2/00

Language of the proceedings: EN

Title of invention:
PROCESS FOR PRODUCING POLYMERS

Patent Proprietor:
Ciba Specialty Chemicals Water Treatments Limited

Opponent:
SNF SAS

Relevant legal provisions:
EPC Art. 54

Keyword:
Novelty - (no)
Late-filed evidence - admitted (yes)

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Case Number: T 1304/17 - 3.3.03

D E C I S I O N
of Technical Board of Appeal 3.3.03
of 8 September 2021

Appellant: (Patent Proprietor)	Ciba Specialty Chemicals Water Treatments Limited Cleckheaton Road, Low Moor Bradford, West Yorkshire BD12 0JZ (GB)
Representative:	BASF IP Association BASF SE G-FLP-C006 67056 Ludwigshafen (DE)
Appellant: (Opponent)	SNF SAS ZAC de Milieux 42163 Andrézieux (FR)
Representative:	Lavoix 62, rue de Bonnel 69448 Lyon Cedex 03 (FR)
Decision under appeal:	**Interlocutory decision of the Opposition Division of the European Patent Office posted on 12 April 2017 concerning maintenance of the European Patent No. 1689790 in amended form.**

Composition of the Board:

Chairman	D. Semino
Members:	D. Marquis
	W. Ungler

Summary of Facts and Submissions

I. The appeals of the patent proprietor and of the opponent lie from the decision of the opposition division posted on 12 April 2017 concerning maintenance of European patent No. 1 689 790 according to the claims of auxiliary request 1 filed during the oral proceedings before the opposition division on 9 February 2017 and a description adapted thereto.

II. Claim 1 as granted read as follows:

"1. A process for preparing a polymer of an ethylenically unsaturated monomer, which monomer is (meth)acrylamide, in which the monomer is obtained from a biocatalysed reaction,
in which the ethylenically unsaturated monomer is prepared by providing a substrate that can be converted into the ethylenically unsaturated monomer, which substrate is (meth)acrylonitrile,
contacting the substrate with a biocatalyst, which biocatalyst comprises cellular material in the form of whole cells or fractured cells and fermentation broth, and thereby converting the substrate into the ethylenically unsaturated monomer in the presence of the cellular material and the fermentation broth, which biocatalyst comprises a nitrile hydratase enzyme,
forming the polymer by polymerising the ethylenically unsaturated monomer or a monomer mixture comprising the ethylenically unsaturated monomer, wherein there is no removal of the cellular material and the fermentation broth from the ethylenically unsaturated monomer."

III. The decision of the opposition division was based on the granted claims as the main request and on auxiliary

request 1 filed during the oral proceedings before the opposition division.

IV. Claim 1 of auxiliary request 1 corresponded to claim 1 as granted with the addition at the end of the claim of the feature "in which the polymer is a water-soluble high molecular weight polymer exhibiting an intrinsic viscosity (IV) of at least 3 dl/g (measured using a suspended level viscosimeter in 1 M sodium chloride at 25°C)".

V. The decision of the opposition division was based *inter alia* on the following documents:

E1: JP H10-316714
E1a: English translation of E1
E5: US 4 343 900
E7: US 4 421 855

VI. As far as it is relevant to the present appeal, the decision of the opposition division can be summarized as follows:

- E1/E1a did not disclose a two-step process according to granted claim 1 wherein cellular material and fermentation broth had not been removed. Paragraph 15 of E1/E1a disclosed at least two alternative ways of carrying out the polymerization step. The expression "without further modification" described a step wherein the acrylamide solution was used without further modification, but that paragraph also disclosed a step wherein the acrylamide solution was modified, at least by increasing the acrylamide concentration by a condensation process. Moreover, paragraph 15 did not mention the presence of cellular material

and fermentation broth. If present, these components had to come from the biocatalyst mixture used in the first reaction step. However, the biocatalyst was not exclusively defined as comprising cellular material and fermentation broth, but other embodiments were possible according to paragraph 14. Thus, the biocatalyst could be devoid of the cellular material and fermentation broth, for instance, when "immobilized bacteria separated from a culture medium" or "an extract" or "an extract immobilized on a carrier" were used.

- Therefore, in view of the alternative embodiments disclosed in paragraphs 14 and 15 it was neither directly nor unambiguously disclosed - beyond any doubt - that E1/E1a disclosed a two step polymerization process comprising the feature "no removal of cellular material and fermentation broth" prior to the polymerization step.

- On that basis novelty over document E1/E1a was acknowledged.

- While the process of granted claim 1 was not inventive starting from E1/E1a as the closest prior art and taking the disclosure of documents E5 and E7 into consideration, the process of claim 1 of auxiliary request 1 was inventive with respect to the same documents.

VII. Both the opponent and the patent proprietor filed an appeal against the decision of the opposition division.

VIII. The following documents were filed in appeal:

- E8 (EP 0 343 840) with letter of the opponent of 4 August 2017

- E9 (EP 0 204 555) with letter of the opponent of 22 December 2017

- E9b (F.J. Dechow, "Separation and Purification Techniques in Biotechnology", William Andrew Publishing/Noyes, 1989, pages 1-33) and E10 (H.W. Blanch & D.S. Clark, "Biochemical Engineering", Marcel Dekker New York, 1997 pages 113-117) with letter of the patent proprietor of 8 January 2018

- E11 (T. Nagasawa and H. Yamada, Pure & Appl. Chem, 1995, 67(7), pages 1241-1256), E12 (A.S. Sarac, Prog. Polym. Sci., 1999, 24, pages 1149-1204) and E13 (M.J. Caulfield et al., Chemical Reviews, 2002, 102, pages 3067-3083) with letter of the patent proprietor of 27 July 2020

IX. On 27 July 2020 the patent proprietor filed a main request that corresponded to auxiliary request 1 found by the opposition division to meet the requirements of the EPC and a new auxiliary request 1.

X. Oral proceedings were held on 8 September 2021 in the presence of both parties. The patent proprietor withdrew auxiliary request 1 filed with letter of 27 July 2020 during the oral proceedings.

XI. The arguments of the opponent, insofar as relevant to the present decision, may be summarised as follows:

Admittance of E8, E9, E9b, E10-E13

- There was no objection to the admittance of E9b and E10 into the proceedings.

- E11 to E13 were filed late in appeal and addressed an issue that had already been raised and discussed before the opposition division. These documents should therefore not be admitted into the proceedings.

Main request – Novelty over E1/E1a

- E1/E1a disclosed a method for producing an acrylamide polymer by means of an aqueous solution polymerization of acrylamide produced by an enzyme process either alone or together with another copolymerizable monomer. In that method, acrylamide was produced by hydrating acrylonitrile by means of the catalytic activity of a nitrile-hydratase. Paragraph 14 also disclosed that the nitrile-hydratase could, among other forms, be used as a culture medium. That culture medium from a micro-organism clearly contained cells and the fermentation broth. While paragraph 14 apparently disclosed a list of five forms of use of nitrile-hydratase, it ultimately corresponded to a list of two members, an embodiment in which the nitrile-hydratase contained cells and a fermentation broth (i.e. the culture medium as such was used) and embodiments in which the enzyme cells and fermentation broth had been removed from the nitrile-hydratase (i.e. the enzyme separated from the culture medium was used).

- Paragraph 15 of E1/E1a additionally recited that the aqueous acrylamide solution following the hydration reaction could be used either without further modification or after increasing the acrylamide concentration by means of a condensation procedure. Since the condensation did not amount to a removal of cells or fermentation broth it had to be concluded that in both alternatives disclosed in paragraph 15 the cells and the fermentation broth present in the acrylamide solution after hydration were also present during the polymerization reaction.

- E1/E1a also disclosed in paragraph 24 that the polyacrylamide polymers obtained had a minimum viscosity of 2000 mPa.s, which corresponded to a minimum molecular weight of 10 000 000. The application of the Mark-Houwink relationship $IV= K M^X$, for which the parameters $K=3.73\ 10^{-4}$ and $X=0.66$ were available in the art, showed that the polyacrylamides obtained in E1/E1a had an intrinsic viscosity (IV) of at least 15.55 dl/g. The water soluble high molecular weight polymer obtained in E1/E1a thus exhibited an intrinsic viscosity (IV) of at least 3 dl/g according to operative claim 1.

- In view of this Claim 1 lacked novelty over E1/ E1a.

XII. The arguments of the patent proprietor, insofar as relevant to the present decision, may be summarised as follows:

Admittance of E8, E9, E9b, E10-E13

- There was no objection to the admittance of E8 and E9 into the proceedings. E11 to E13 were filed to establish a prejudice relevant to the question of inventive step in view of the preliminary opinion of the Board. These documents should therefore be admitted into the proceedings.

Main request – Novelty over E1/E1a

- E1/E1a disclosed a process by which acrylonitrile was hydrated to acrylamide and polymerized to polyacrylamide. It was apparent from the whole of E1/E1a that any presence of impurities in the acrylamide solution was undesirable for the properties of the polyacrylamide.

- While it could be acknowledged that the polyacrylamides according to E1/E1a had an intrinsic viscosity within the range of operative claim 1 as shown by the Mark-Houwink equation, operative claim 1 was nevertheless novel over E1/E1a because the disclosure in paragraphs 13-16 described a process from which multiple selections had to be made in order to arrive at the subject-matter of operative claim 1.

- Paragraph 14 listed various forms of nitrile hydratase that could be used, a culture medium of the micro-organism, resting bacteria or immobilized

bacteria separated from a culture medium or an extracted enzyme as such or immobilized on a carrier. Out of these alternatives only the culture medium of the micro-organism contained cells and a fermentation broth. The skilled person thus would have had to select first a nitrile hydratase as a culture medium among several alternatives. Even if he had done so, the skilled person would have understood that a separation of the insoluble matter present in the culture medium was necessary prior to polymerization. That was confirmed by the examples of E1/E1a which disclosed such a separation step of the biocatalyst.

- While the condensation process referred to in paragraph 15 of E1/E1a did not constitute as such a removal of cells or fermentation broth from the aqueous acrylamide solution, the passage in paragraph 15 did not exclude necessary additional steps prior to the polymerization stage and in particular steps relating to a purification of the solution to be polymerized. In fact, should any impurities be present in the acrylamide solution obtained from the hydration reaction, E1/E1a disclosed that purification steps should take place to remove these impurities prior to the polymerization of the monomer. This was the case as the presence of impurities resulting from acrylamide production methods adversely impacted the quality of a polyacrylamide. A further selection in paragraph 15 was also necessary to arrive at operative claim 1.

- E1/E1a therefore did not disclose an instance in which acrylonitrile was converted in the presence of cellular material and fermentation broth into

the acrylamide monomer and no removal of the cellular material and the fermentation broth took place before polymerization. Hence, the subject-matter of operative claim 1 was novel in view of E1/E1a.

XIII. The appellant/patent proprietor requested that the decision under appeal be set aside and the patent be maintained on the basis of the main request filed with letter of 27 July 2020.

XIV. The appellant/opponent requested that the decision under appeal be set aside and the European patent be revoked.

Reasons for the Decision

1. Admittance of documents

1.1 Documents E8, E9, E9b and E10-E13 were submitted by the parties during the appeal proceedings. The Board decided to admit these documents into the proceedings during the oral proceedings. Since none of these documents is relevant to the present decision, there is no need to provide a fully fledged reasoning as to their admission.

Main request

2. Novelty over E1/E1a

2.1 Novelty of claim 1 of the main request was contested over the disclosure of E1, reference being made with the agreement of both parties to its translation E1a (the document is therefore cited as E1/E1a in what follows), specifically over the description of the

process in paragraphs 13-15 or E1/E1a.

2.2 E1/E1a relates to a method for producing an acrylamide polymer by effecting an aqueous solution polymerization of acrylamide produced by an enzyme process either alone or together with another copolymerizable monomer at a concentration range of 10 to 60 wt%, and then drying the obtained acrylamide polymer at 95°C or higher (paragraph 10).

2.3 The components and steps of the process according to E1/E1a are described in more details in paragraphs 13-15. It is thus specified in paragraph 13 that an acrylamide produced using an enzyme process means an acrylamide produced by hydrating acrylonitrile by means of the catalytic activity of a nitrile-hydratase. Nitrile-hydratases are defined in this paragraph as enzymes that convert nitrile compounds into the corresponding amides, and nitrile-hydratases are derived from micro-organisms which constitute a biocatalyst in the sense of operative claim 1.

2.4 Paragraph 14 further describes several forms of use of nitrile-hydratases in the context of the process of E1/E1a. It is apparent from the list provided in paragraph 14 that nitrile-hydratases can be used in a culture medium of a given micro-organism and thus in the presence of cells and fermentation broth, or in a form that additionally requires a separation of the enzyme from the culture medium it originated from (these forms being disclosed as resting bacteria or as immobilized bacteria separated from a culture medium, as an extract obtained by extracting an enzyme having nitrile-hydratase activity from resting bacteria, or such an extract immobilized on a carrier).

2.5 In order to perform the process of E1/E1a, the skilled person has therefore to perform a selection regarding the use of nitrile-hydratase, more specifically whether to use a nitrile-hydratase in the presence of cells and fermentation broth (in the culture medium) or whether to use a nitrile-hydratase in a form that involves a separation of cells and/or fermentation broth from the nitrile-hydratase.

2.6 While the nitrile-hydratase that is exemplified in E1/E1a (production example 1, paragraph 26) is one in immobilized form, obtained by isolating and washing the obtained strain of micro-organism from its culture medium, there is nothing in E1/E1a that indicates that nitrile-hydratases in their culture medium as mentioned in paragraph 14 would not be suitable in the process according to E1/E1a without having been subjected to a separation or purification treatment. Paragraphs 1 and 8 of E1/E1a mentioned by the patent proprietor only refer to the production of acrylamide polymers having high molecular weights and low contents of water-insoluble components but these paragraphs do not imply any treatment of the nitrile-hydratase used in the process. The instances in E1/E1a referring to impurities being an issue in the production of polyacrylamide in fact refer to a process of the prior art wherein copper catalysts were used instead of enzymes (paragraphs 5, 9 and 12). By contrast, the enzyme based process according to E1/E1a is disclosed in the passage bridging pages 5 and 6 as one that leads to a high purity of the acrylamide. In that regard the Board does not find in E1/E1a, an indication, even an implicit one, that the nitrile-hydratases mentioned in paragraph 13 should be purified or separated. On the contrary, it appears from E1/E1a that enzyme based processes for the production of acrylamides are less

affected by purity issues than other processes of the prior art based on copper catalysts. The selection of a nitrile-hydratase as a culture medium of micro-organism as contemplated in paragraph 14 of E1/E1a thus does not imply a separation/purification of the enzyme before or after the acrylamide solution is obtained by hydration of acrylonitrile.

2.7 The further steps of the process of E1/E1a are described in paragraph 15 which indicates the optional treatment of the aqueous acrylamide solution produced from the hydration of acrylonitrile by a condensation procedure. It is apparent from the context of E1/E1a that that passage addresses an essential feature of the process which is that the monomer concentration prior to polymerization must be in a defined range (claim 1 and paragraphs 10 and 20) and constitutes a guidance on how to proceed to increase the monomer concentration, when this is needed, by a condensation procedure. According to paragraph 15, the aqueous acrylamide solution can be used without modification or after a condensation procedure. In this respect it was acknowledged by both parties that none of these alternatives amounts to a removal of cells and fermentation broth from the biocatalyst. Both options of performing a condensation or using the aqueous acrylamide solution without modification, are thus according to operative claim 1.

2.8 It was argued by the patent proprietor on the basis of paragraph 20 that, in case the concentration of the monomer solution after its production and prior to its polymerization was above 60 wt.-%, a separation/ purification step would be required to avoid undesirable cross linking within the polymer product. However, while paragraph 20 links a too high monomer

concentration to undesirable cross linking in the polymer, there is nothing in E1/E1a that would imply a separation/purification of the aqueous acrylamide solution at any stage of the process to avoid such a too high monomer concentration resulting in a cross linking within the polymer produced. In the absence of further evidence that a separation/purification step was implied in the process disclosed in paragraphs 13-15, the Board can only conclude that there is no disclosure of a separation/purification step of the aqueous acrylamide solution prior to polymerization in paragraph 15 and therefore no removal of cells or fermentation broth at that stage.

2.9 It was also established by the opponent on the basis of paragraph 24 that the polymers produced according to the process of E1/E1a had a molecular weight of approximately 10 000 000 or higher which corresponded to an intrinsic viscosity of 15.55 dl/g or higher by application of the Mark-Houwink formula ($IV = K\ M^X$, in which IV is the intrinsic viscosity of the polymer, $K=3.73\ 10^{-4}$, $X=0.66$ and M is the molecular weight of the polymer). Since it was acknowledged by the patent proprietor that that calculation was reasonable, the Board has no reason to doubt that the Mark-Houwink formula is applicable to the polymers of E1/E1a and that the intrinsic viscosity of the polymers according to E1/E1a is 15.55 dl/g or higher. In that regard, it was established by the opponent that the polymers produced by the process of E1/E1a have an intrinsic viscosity above 3 dl/g and are thus according to operative claim 1. Also the patent proprietor confirmed at the oral proceedings that running the process according to E1/E1a with no removal of cells or fermentation broth as in claim 1 of the main request would lead to a water-soluble polymer which meets the

condition on the intrinsic viscosity according to claim 1.

2.10 The Board concludes from the above that the process of E1/E1a as disclosed in paragraphs 13-15 in the embodiment in which a nitrile-hydratase is used in a culture medium is according to operative claim 1. Claim 1 of the main request therefore lacks novelty over E1/E1a.

3. As the only request maintained by the patent proprietor in appeal does not meet the requirement of novelty, there is no need for the Board to decide on any further issue and the patent is to be revoked.

Order

For these reasons it is decided that:

1. The decision under appeal is set aside.

2. The patent is revoked.

The Registrar: The Chairman:

B. ter Heijden D. Semino

Decision electronically authenticated