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Case Number: T 0656/09 - 3.3.07

Application Number: 03809353.0

Publication Number: 1558369

IPC: B01D 61/44, C12P 7/56

Language of the proceedings: EN

Title of invention:
Method of separating multivalent ions and lactate ions from a fermentation broth

Applicants:
PURAC Biochem BV

Opponent:
-

Headword:
-

Relevant legal provisions:
EPC Art. 56

Relevant legal provisions (EPC 1973):
-

Keyword:
"Inventive step (no) - obvious solution - Main Request and Auxiliary Request"

Decisions cited:
-

Catchword:
-

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Case Number: T 0656/09 - 3.3.07

D E C I S I O N
of the Technical Board of Appeal 3.3.07
of 20 July 2012

Appellants: (Applicants)
PURAC Biochem BV
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NL-4206 AC Gorinchem (NL)

Representative:
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Decision under appeal:
Decision of the Examining Division of the European Patent Office posted 3 November 2008 refusing European patent application No. 03809353.0 pursuant to Article 97(2) EPC.

Composition of the Board:

Chairman: J. Riolo
Members: G. Santavicca
M.-B. Tardo-Dino

Summary of Facts and Submissions

I. The appeal lies from a decision of the Examining Division, posted on 3 November 2008, refusing European patent application 03 809 353.0, originating from international application PCT/EP2003/050759 (international publication number WO 2004/037397 A1) filed on 27 October 2003 and claiming priority from EP 02 079 493.9 of 28 October 2002.

II. The decision under appeal was based on an amended Claim 1 submitted with letter of 19 June 2008 as well as on unamended original Claims 2 to 14 (Main Request). Claim 1 read as follows:

Main Request

"1. A method of separating multivalent ions and lactate ions from a fermentation broth comprising a multivalent ion lactate salt by using an electrodialysis or electrolysis apparatus, comprising the steps of introducing the broth wherein the multivalent ion concentration is at least 0.1 mole/L, the lactate ion concentration is less than 300 g/L, and less than 10 mole% of the lactate ions are negatively charged ions other than lactate ions, into a first compartment of the electrodialysis or electrolysis apparatus, which compartment is limited by an anion—selective or non-selective membrane and a cathode, and if containing extra membranes said extra membranes are selected from an anion-selective, a non-selective, and a bipolar membrane, and in which first compartment the multivalent ion is converted to obtain a residual stream comprising the hydroxide of the multivalent ion,

and the lactate ion is transported through the anion—selective or non-selective membrane into a second compartment limited by the anion—selective or non—selective membrane and an anode, after which the lactate ion is neutralized to lactic acid."

III. In the decision under appeal, it was *inter alia* held that:

(a) As regards interpretation, Claim 1 excluded the presence of a cation-selective membrane in the first compartment, not however in the second compartment. Also, the conversion of the multivalent ion to obtain the corresponding hydroxide took place in the first compartment. Finally, the other negatively charged ions represented less than 10 mole% of the lactate ions.

(b) The subject-matter of Claim 1 was novel over the embodiment of Figure 1(a) of D1, the only distinguishing feature of Claim 1 being the concentration of multivalent ions in the fermentation broth of at least 0.1 mole/l. That concentration was a distinction also over either of D2 and D3.

(c) As regards inventive step:

(i) the closest prior art was described in D1 or D3.

(ii) The problem addressed by the application was the provision of a method for directly separating multivalent ions and lactate ions from a fermentation broth by electrodialysis without the need for introducing additional chemicals (as in D1) or using extra steps (such as in D3) for removal of multivalent ions, for preventing fouling.

(iii) Still according to the application, that problem had been solved by avoiding the use of cation-selective membranes in the first compartment and by allowing precipitation of the hydroxides of the multivalent ions.

(iv) However, the electrodialysis cell of the embodiment of Figure 1(a) of D1 did not contain cation-exchange membranes, hence was identical to one of those used in the method of Claim 1. Furthermore, by mentioning that precipitation of multivalent cation hydroxides within the salt/base loop or on the membranes was minimized, D1 also made known a possible precipitation of compounds of multivalent cations in the first compartment. Since the embodiment of Figure 1(a) of D1 was identical to that of the application, and since D1 taught how to prevent fouling of the membrane by avoiding precipitation of the multivalent cations, doubts were cast on whether the problem addressed by the application had effectively been solved. These doubts were not removed by the examples of the application, because none of them referred to scaling or fouling as determined in D1.

(v) Thus, the problem effectively solved over D1 was to provide a method for the separation of lactate ions and multivalent ions from a fermentation broth containing high amounts of multivalent ions.

(vi) Although D1 disclosed low amounts of multivalent ions, it did not limit the upper concentration thereof. Therefore, the

skilled person would obviously consider also using the method of Figure 1(a) of D1 when higher concentrations of multivalent ions were present.

(d) So the claimed method did not involve an inventive step and the application should be refused.

IV. In their statement setting out the grounds of appeal, the appellants enclosed two sets of amended claims as their new Main Request and Auxiliary Request, respectively made up of 11 and 10 claims. Claim 1 of each of these claims requests read as follows:

Main Request

Claim 1 of the new Main Request is identical to Claim 1 of the Main Request underlying the decision under appeal.

Auxiliary Request

"1. A method of separating multivalent ions and lactate ions from a fermentation broth comprising a multivalent ion lactate salt by using an electrodialysis or electrolysis apparatus, comprising the steps of introducing the broth wherein the multivalent ion concentration is at least 0.1 mole/L, the lactate ion concentration is less than 300 g/L, and less than 10 mole% of the lactate ions are negatively charged ions other than lactate ions, into a first compartment of the electrodialysis or electrolysis apparatus, which compartment is limited by an anion-selective or non-selective membrane and a cathode, and if containing extra membranes said extra membranes are selected from

an anion-selective, a non-selective, and a bipolar membrane, and in which first compartment the multivalent ion is converted to obtain a residual stream comprising the hydroxide of the multivalent ion which is at least partially present as solid in slurry, and the lactate ion is transported through the anion-selective or non-selective membrane into a second compartment limited by the anion-selective or non-selective membrane and an anode, after which the lactate ion is neutralized to lactic acid."

V. In their statement setting out the grounds of appeal, the appellants have in particular argued as follows:

(a) As regards the amendments, the new requests only contain method claims, Claims 12 to 14 as filed having been cancelled. Claim 1 of the Auxiliary Request, compared to Claim 1 of the Main Request, included the additional features of Claim 7 as filed originally, which thus had been cancelled.

(b) Since Claim 1 recited that the multivalent ions were converted to their respective hydroxides in the residual stream comprising the multivalent ions, the addition of a chelator, as in D1, was implicitly excluded, which fact applied *a fortiori* when the hydroxides precipitated and were present as solids in a slurry, as defined in the Auxiliary Request.

(c) As to inventive step, there was agreement with the decision under appeal as regards problem (a method of separating lactate and multivalent ions from fermentation broths) and solution (avoidance of use of cation-selective membrane and allowance of precipitation of calcium hydroxide).

(d) However, D1 did not address the separation of multivalent and lactate ions from a fermentation broth but the separation of monovalent and lactate ions therefrom, as illustrated in D1 by e.g. the use of the letter M, meaning sodium, potassium or ammonium. So D1 related to the conversion of monovalent (particularly ammonium) lactate to lactic acid, whereas the present application related to the conversion of multivalent lactate (calcium or magnesium) lactate to lactic acid, which required different techniques.

(e) Also, since D1 only mentioned the prevention of fouling on the membranes, it in fact related to a different problem than that of preventing fouling of the electrodes, as did the present application.

(f) D1 illustrated only one embodiment without cation-selective membranes and preferred embodiments with cation-selective membranes, so it taught away from using electrolysis cells without cation-selective membranes.

(g) Therefore, the claimed method was not obvious, which applied *a fortiori* to the claimed method of the Auxiliary Request, and the decision under appeal should be reversed.

VI. The appellants were summoned to oral proceedings. In a communication of the Board in preparation for oral proceedings, dated 5 March 2012, the Board enclosed two documents, as follows:

D7: Perry's Chemical Engineers' Handbook (D7), 6th edition, 17-38; and,

D8: YH Kim and SH Moon, *Lactic acid recovery from fermentation broth using one-stage electrodialysis*,

Journal of Chemical Technology and Biotechnology, 76:169-178, 2001, and

drew the attention of the appellants to a number of points (such as amendments, clarity, novelty and inventive step) that needed to be discussed, in particular to the following points:

(a) As regards clarity and interpretation of Claim 1, *inter alia*, having regard to the feature "wherein the multivalent ion is converted to obtain a residual stream comprising the hydroxide of the multivalent ion", it was not clear that this feature limited in any way the amount and/or the state (dissolved, dissociated or precipitated) of the hydroxide of the multivalent ion. Nor could that feature be interpreted as meaning that the hydroxide of the multivalent ion was actually separated from the fermentation broth. Hence, it was not clear that multivalent ions were actually separated from the fermentation broth.

(b) As regards the closest prior art for assessing inventive step, not only D1 and D3 but also D8 may be considered, which concerns direct separation of acid lactic from a fermentation broth containing multivalent ions.

(c) As to the problem solved, the examples of the application represented very particular situations within the breadth of Claim 1, in particular because no operating conditions were specified in Claim 1. Nor was it apparent that any of these examples were comparative over e.g. D1 or D8. So the problem effectively solved could only be seen to consist in the mere provision of further processes for the separation of acid lactic from a fermentation broth containing multivalent ions.

(d) As regards the character of the process of Claim 1, since D8 disclosed the claimed steps and D7 disclosed the possible use of non-selective membranes, it might be considered as having been obvious for the skilled person at the priority date of the application.

VII. By letter of 3 May 2012, the appellants informed the Board that they would not attend the scheduled oral proceedings. However, no reference whatsoever was made to the communication by the Board, nor to the documents annexed thereto. In a telephone enquiry on 23 May 2012 by the Registry, the appellants declared that the communication by the Board in preparation of the oral proceedings had not been received.

VIII. Oral proceedings took place on 24 May 2012 in previously announced-absence of the appellants, pursuant to Rule 115(2) EPC. After deliberation by the Board, the following decision was made:

(e) to continue the appeal proceedings in writing;

(f) to enclose the communication by the Board that had not been received, including the annexes thereto, in the minutes of the oral proceedings;

(g) to invite the appellants to reply or inform the Board whether a decision according to the state of the file was requested, within two months of the notification of the minutes and annexes.

IX. By letter of 3 July 2012, the appellants requested a decision according to the state of the file.

X. The appellants requested that the decision under appeal be set aside and that a patent be granted on Claims 1

to 11 of the Main Request, filed with letter of 26 February 2009, alternatively on Claims 1 to 10 of the Auxiliary Request filed with the same letter.

Reasons for the Decision

1. The appeal is admissible.

Main Request

Amendments

2. In the decision under appeal, no objections were raised against the amendments made to the claims of the Main Request. The Board has no reason to deviate from that decision. Since the appeal fails for lack of an inventive step of the method of Claim 1, the Board need not give further details on the amendments made. Therefore, the Board is satisfied that Claim 1 of the Main request fulfils the requirements of Article 123(2) EPC.

Clarity and scope of Claim 1

3. In the decision under appeal, no objections were raised against the clarity of the claims of the Main Request but an interpretation was given for some of the features of Claim 1. The Board has no reason to deviate from the decision, with the exceptions indicated in the Board's communication, in particular (Point VI.a, *supra*) that neither the state nor the amount of the hydroxide was defined, hence that those hydroxides were not necessarily separated from the fermentation broth.

Since the appeal fails for lack of an inventive step of the method of Claim 1, the Board need not give further details on clarity. Therefore, the Board is satisfied that Claim 1 of the Main request fulfils the requirements of Article 84 EPC.

Novelty

4. According to the decision under appeal, the method of Claim 1 of the Main Request is novel over D1, D2 and D3. The Board has no reason to deviate from this decision. As regards D8, mentioned in the communication by the Board, it does not disclose the composition of the broth as defined in Claim 1 of the Main Request, in particular the amounts of multivalent ions and negatively charged ions other than lactate ions. Hence, the method of Claim 1 of the Main Request is novel (Article 54(1)(2) EPC).

Closest prior art

5. The present application concerns a method of separating multivalent ions and lactate ions from a fermentation broth (Title).

5.1 The objective set out in the present application (page 3, lines 5-9) is to provide a method of directly separating multivalent ions and lactate ions from a fermentation broth comprising a multivalent ion lactate salt by using an electrodialysis or electrolysis apparatus, without the need for an extra step for removal of multivalent ions or the introduction of additional chemicals for preventing fouling by the precipitation of complexes formed by multivalent ions.

5.2 D8 (title) relates to lactic acid recovery from fermentation broth using one-stage electrodialysis.

5.3 The objective of D8 (page 170, left column, lines 16-22) is the development of a simplified electrodialysis (ED) process for lactic acid recovery, i.e. a one-stage ED for recovery of free lactic acid from fermentation broth directly without pretreatment, such as desalting ED or ion-exchange for removal of hardness metals and foulants, thus, like the present application.

5.4 Therefore, the present application and D8 definitely belong to the same technical field, concern the same separation and address the same objective.

5.4.1 Instead, D1 and D3, considered as closest prior art in the decision under appeal, comprise pre-treatment of the incoming broth to reduce hardness metals such as calcium and magnesium, hence do not deal with direct recovery of lactic acid from fermentation broth.

5.4.2 Consequently, D8 discloses the closest prior art.

The disclosure of D8

6. D8 (Abstract) relates to an investigation on one-stage electrodialysis (ED) for lactic acid recovery with two- and three-compartment water-splitting ED (WSED) using various ion-exchange membranes in order to overcome the inefficiency of two-stage ED.

6.1 The distinction between one-stage ED according to D8 and two-stage ED according to the prior art is apparent

from the process flow sheet of Figure 1 of D8, reproduced hereinafter. This process flow sheet deals with the development of ED processes for recovery of lactic acid. In particular (paragraph bridging pages 169 and 170), route (a) represents a known two-stage ED using *inter alia* an ion-exchange column for removing hardness materials such as calcium and magnesium ions, to prevent membrane fouling due to hydroxide precipitates. Route (b) represents a known simplification of the process of route (a), in which only a nanofiltration step is used before the ED. Finally, route (c) represents the process of D8, which does not use any pretreatment for removal of hardness materials, such as calcium and magnesium ions. Hence, route (c) corresponds to the method of the present application.

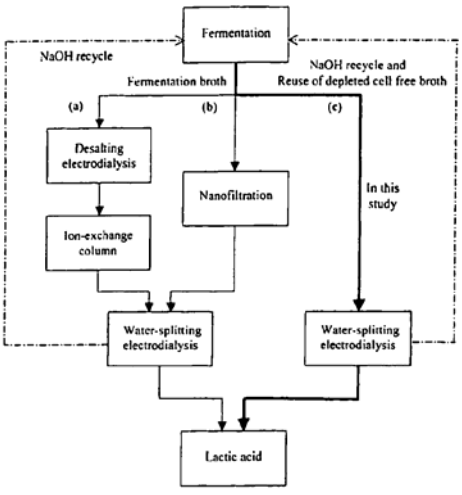

Figure 1. Process flow sheet of the integrated processes for lactic acid recovery ((a) two-stage electrodialysis,[14] (b) hybrid of nanofiltration and water-splitting electrodialysis,[15] (c) one-stage electrodialysis).

6.2 The one-stage ED disclosed by D8 is applicable to conventional fermentation processes for producing lactic acid as calcium, ammonium or sodium salts, wherein the fermentation broth contains organic and inorganic ingredients (page 169, left column, last

paragraph). Hence, recovery of lactic acid from fermentation broths containing calcium lactate is contemplated by the process of D8.

6.3 The electrodialysis apparatus shown in Figure 2(a) of D8 is reproduced hereinafter.

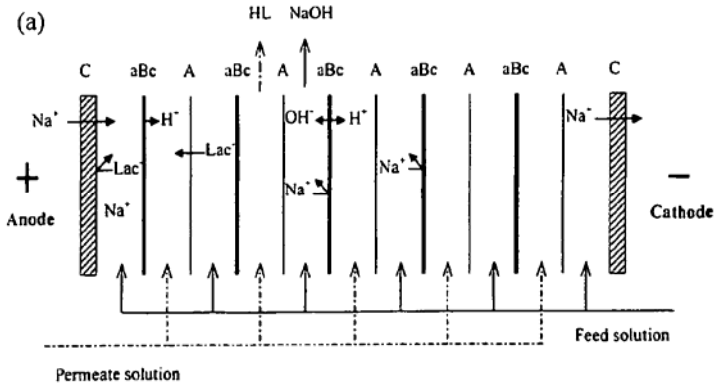

Figure 2. (a) Principle of two-compartment water-splitting electrodialysis; (b) principle of

6.4 It comprises, in addition to the usual cathode and anode, only anion-selective membranes (A) and bipolar membranes (aBc). The compartments defined in Claim 1 are apparent from the Figure 2(a). So all of the structural features defined in the claims of the Main Request are present in the said embodiment of D8.

6.5 The fermentation broth used as feed solution in the investigation of D8 contained high levels of foulants, such as residual glucose and protein, colloids, and hardness metals such as calcium and magnesium (i.e. as multivalent ions) (page 170, right column, lines 9-12). Hence, the fermentation broth is used as such.

6.6 However, as regards the composition of that fermentation broth, D8 merely discloses a content of lactate of 80-100 g/l (which satisfies the condition

set out in Claim 1 of the Main Request of less than 300 g/l). Although D8 (page 170, right column, first paragraph) also refers to one of the references (17) concerning the process that generates the fermentation broth, the Board has not succeeded at obtaining any translation thereof, which could have been sent to the appellants.

6.7 Thus, the method of Claim 1 of the Main Request is novel over D8 as regards the defined concentrations for the multivalent ion and the negatively charged ions other than the lactate ions.

Solved problem

7. The application as originally filed acknowledges the prior art disclosed by D1 and D3, dealt with in the decision under appeal, not however D8.

7.1 The problem addressed in the application as filed was to provide a method of directly separating multivalent ions and lactate ions from a fermentation broth comprising a multivalent ion lactate salt by using electrodialysis or electrolysis apparatus, without the need for an extra step for removal of multivalent ions or the introduction of additional chemicals for preventing fouling by the precipitation of complexes formed by multivalent ions (Page 3 of the international application as published, first full paragraph).

7.2 Since the method of D8 (Figure 1 route (c)) does not require an extra step for removal of multivalent ions nor the introduction of additional chemicals for preventing fouling by the precipitation of complexes

formed by multivalent ions, the problem has to be reformulated, as D8 is taken as the closest prior art.

7.3 The appellants have not replied to the communication by the Board, thus have not wished to discuss a different problem over D8.

7.4 The examples of the application as filed respectively concern:

(a) Example 1 is based on the use of a cell comprising a single ACM membrane operated according to the given operating conditions, including the use of a 12 wt.% calcium lactate solution as the catholyte, with addition of an acid (lactic acid) in order to keep its pH below 10. Hence, in this example (unless it is comparative) chemicals are added to control the operation of the cell. It is not clear whether the maintained pH prevents precipitation and/or reduces scaling on the electrodes (cf. page 3, lines 1-3). Example 1 mentions circulation rates of 117 and 105 l/h, respectively for anolyte and catholyte. The visual determination of no scaling concerns an operating time of up to 5 h.

(b) Example 2 concerns a repetition of Example 1 at higher temperature and without addition of an acid, so by allowing precipitation of calcium hydroxide. The circulation rates are of 114 (anolyte) and 120 l/h (catholyte). Despite formation of calcium hydroxide slurry, the electrodes remain visually free from scaling. The lactic acid concentration increased less than that in Example 1.

(c) Example 3 deals with an electrolysis cell provided with a non-selective porous membrane and used in combination with a feed container and a

fermentator. Calcium hydroxide was used to perform neutralization during the fermentation process.

7.5 Thus, these examples represent very particular situations within the breadth of Claim 1, as:

(a) Claim 1 encompasses the use of broths having multivalent ion concentrations any higher than 0.1 mole%, not only calcium, and any kind and amounts of residual organic or biological foulants.

(b) The slurry mentioned in Claim 7 may contain any amount of any kind (not only calcium) of multivalent ions.

(c) The amount and nature of the precipitated material, if any, is not limited by the features of Claim 1.

(d) The broth is treated in apparatuses having any anode or cathode and any anion-selective or non-selective membranes.

(e) No operating conditions such as temperature or circulation rates are specified in Claim 1, although they have an influence on whether the slurry has enough time to deposit and scale.

(f) Finally, none of these examples is comparative over D8, which discloses similar circulating rates (page 171, right column, lines 15-17), i.e. about 101 and 103 for permeate and feed solution.

7.6 It is thus not apparent that the problem solved could be formulated in terms of an improvement over the closest prior art D8. Based upon the original information present in the application as filed, the problem solved was to provide a further process for direct recovery of lactic acid from fermentation broths.

Character of the solution

8. The operating conditions that distinguish the method of Claim 1 of the Main Request from that of D8 are a multivalent ion concentration of at least 0.1 mole/l and less than 10 mole% of negatively charged ions other than lactate ions.

8.1 The first distinguishing feature makes clear that no pretreatment of the incoming broth is carried out. This however is already known from D8 (route (c) of Figure 1), in a context where it is stressed that the incoming contains high levels of foulants, *inter alia* calcium and magnesium ions. Hence, this measure was obvious from D8.

8.2 As regards the second distinguishing feature, the application as originally filed never describes it in more particular details, so no effect whatsoever resulting therefrom has ever been invoked. In any case, D8 does not set any limitations on the composition of the broths and specifically mentions those containing calcium lactate, as in the present application.

8.3 The method of Claim 1 of the Main Request was obvious over D8 (Article 56 EPC) and lacks the inventive step required by Article 52(1) EPC.

8.4 Therefore, the Main Request is rejected.

Auxiliary Request

Amendments

9. Compared to Claim 1 of the Main Request, Claim 1 of the Auxiliary Request comprises the additional features of Claim 7 as originally filed, namely that the hydroxide of the multivalent ion is at least partially present as solid in slurry. The amendments are clear (Article 84 EPC) and based on the application as filed (Article 123(2) EPC).

Inventive step

10. Since D8 stresses the direct recovery of lactic acid from fermentation broths containing high levels of foulants, whereby the fermentation broth may contain calcium lactate, the amendments do not change the closest prior art, still disclosed by D8, nor the problem solved.

10.1 Claim 1 does not define how much calcium and magnesium hydroxides are precipitated, i.e. even very low amounts are encompassed thereby, let alone whether the solid part of the slurry is separated or simply swept away. Nor is any circulating rate defined, e.g. circulating rates suitable to quickly sweep away the slurry before it has time to deposit and scale are encompassed thereby. D8 discloses circulation rates which are comparable to those illustrated in the examples of the present application.

10.2 So the additional measure too appears to be obvious from D8.

10.3 Consequently, the method of Claim 1 of the Auxiliary Request lacks an inventive step.

10.4 The Auxiliary Request is thus rejected.

Conclusion

11. None of the claims requests submitted by the appellants fulfils the requirements of the EPC. So they must be rejected.

12. All of the objections on which the rejection of the Main and Auxiliary requests are based were raised as such in the communication by the Board in preparation for oral proceedings (Point VI., *supra*). The appellants did not make any observations thereon.

Order

For these reasons it is decided that:

The appeal is dismissed.

The Registrar: The Chairman:

S. Fabiani J. Riolo