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D E C I S I O N
of 13 October 1998

Case Number: T 0665/97 - 3.3.3

Application Number: 93903632.3

Publication Number: 0623153

IPC: C08G 63/06

Language of the proceedings: EN

Title of invention:

Continuous process for the manufacture of lactide and lactide polymers

Applicant:

Cargill, Incorporated

Opponent:

-

Headword:

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Relevant legal provisions:

EPC Art. 56, 123(2)

Keyword:

"Main request: Inventive step - (no) obvious combination of known features"

"Auxiliary request: Amendments not clearly and unambiguously derivable from the original application"

Decisions cited:

T 0169/83

Catchword:

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Case Number: T 0665/97 - 3.3.3

D E C I S I O N
of the Technical Board of Appeal 3.3.3
of 13 October 1998

Appellant: Cargill, Incorporated
15407 McGinty Road West
Wayzata, MN 55391-2399 (US)

Representative: Smulders, Theodorus A.H.J., Ir.
Vereenigde Octrooibureaux
Nieuwe Parklaan 97
2587 BN 's-Gravenhage (NL)

Decision under appeal: Decision of the Examining Division of the
European Patent Office posted 30 December 1996
refusing European patent application
No. 93 903 632.3 pursuant to Article 97(1) EPC.

Composition of the Board:

Chairman: C. Gérardin
Members: H. H. Fessel
J. Stephens-Ofner

Summary of Facts and Submissions

I. European patent application No. 93 903 632.3, published with the international publication number WO 93/15127 was filed on 22 January 1993 on the basis of a set of 19 claims, of which Claim 1 read as follows:

"A process for the continuous conversion of a crude lactide acid feed in a hydroxylic medium to polylactide, said process comprising the steps of:

- (a) providing a source of lactic acid in a hydroxylic medium;
- (b) concentrating the lactic acid in the hydroxylic medium by evaporating a substantial portion of the hydroxylic medium to form a concentrated lactic acid solution;
- (c) polymerizing lactic acid in the concentrated lactic acid solution of step (b) by further evaporation of the hydroxylic medium to form polylactic acid molecules having an average molecular weight of between about 100 and about 5000;
- (d) forming a crude lactide in the presence of catalyst means for catalyzing the depolymerization of the polylactic acid molecules to form lactide molecules;
- (e) purifying the crude lactide formed in step (d) to form a substantially purified lactide by distilling the crude lactide; and
- (f) reacting the substantially purified lactide of step (e) to form polylactide."

II. The application was refused by a decision of the Examining Division of 5 December 1996, posted 30 December 1996, based on a set of 22 claims filed during oral proceedings, of which Claim 1 read as follows:

"A process for preparing a polylactide; the process including: providing crude lactide mixture including meso-lactide and at least one of the L-lactide and D-lactide; said process being characterized of steps of:

- (a) purifying the crude lactide mixture into at least one purified polymer grade lactide fraction by distillation, without a step of solvent extraction or recrystallization; and
- (b) reacting the purified lactide fraction to form polylactide."

Dependent Claims 2 to 16 related to specific embodiments of the subject-matter of Claim 1. The subject-matter of Claim 17 was a process for preparing polymer grade lactide from crude lactide mixture including meso-lactide, L-lactide, D-lactide, water and lactic acid. Claims 18 to 22 concerned preferred embodiments of the process according to Claim 17.

The reason for the refusal was lack of inventive step of the subject-matter claimed in Claims 1 to 22. The most relevant prior art was said to be represented by

- D1: EP-A-0 261 572;
- D2: EP-A-0 275 581;
- D3: DE-B-1 234 703 and
- D4: E. S. Lipinsky and R. G. Sinclair, "Is Lactic Acid a Commodity Chemical?", Chem. Eng. Progress, August 1986, pages 26 to 32).

The distinguishing features of the claimed subject-matter versus that prior art were that D1 did not describe a process in which a mixture of meso- and D- or L-lactide was used and D2 did not describe the purification of lactide by distillation.

Starting from D2 as closest prior art the problem to be solved was seen in the provision of a simpler, more economical process for the preparation, purification and polymerization of lactide. The solution given in the application in suit was the distillation of crude lactide instead of relying on the extraction method disclosed in D2.

The Examining Division held that solution not to involve any inventive step since distillation was, together with recrystallization (as disclosed in D1) and extraction (as disclosed in D2), one of the most common ways to purify a chemical compound. The prejudice alleged by the Applicant was not accepted since in D4, a document dealt with in oral proceedings, it was taught that lactide obtained by distillation from polylactic acid could be further purified by redistillation.

III. On 5 March 1997 the Appellant (Applicant) filed a Notice of Appeal together with payment of the prescribed fee.

With the Statement of Grounds of Appeal filed on 7 May 1997 a new set of 20 claims was filed based on the claims of the decision under appeal, but amended in that feature (a) of Claim 1 now read:

"purifying the crude lactide mixture by distillation in a packed column into at least three component streams, an overhead low-boiling stream, a high-boiling bottom stream, and an intermediate boiling sidedraw stream including purified polymer grade lactide, without a step of solvent extraction or recrystallization; and".

In Claim 16 that feature now read:

"purifying the crude lactide mixture into at least one purified lactide fraction by distillation in a packed column to provide at least three component streams, an overhead low-boiling stream, a high-boiling bottom stream, and an intermediate boiling sidedraw stream providing polymer grade lactide".

The claims were said to result from a combination of Claims 1 and 3, respectively 17 and 20 of the set of claims underlying the decision under appeal in conjunction with the features described on page 46, line 19.

The Appellant alleged that Article 113(1) EPC had been violated since the decision was based on a document, on which the Appellant had had no chance to comment.

Regarding the issue of inventive step, he argued that there was a general prejudice against distillation of crude lactide to form a polymer grade lactide and that, in addition thereto, he had, surprisingly, found that for the continuous distillation of crude lactide in a packed column, the point of highest lactide purity is not at the bottom of the column, but is at an intermediate point above the bottom of the column.

IV. Oral proceedings were held on 13 October 1998 at the Appellant's request.

- (i) At the beginning of the hearing the Appellant was informed that in the preliminary view of the Board there did not seem to be any violation of Article 113(1) EPC, since the Appellant had not only been given ample opportunity to comment, but actually made observations on D4, as could be seen from items 10 and 11 of the minutes. Thereafter, the Appellant decided to abandon that procedural issue.

Regarding the wording of the claims the Board expressed serious doubts whether the requirements of Article 123(2) EPC were met. A detailed discussion, however, did not seem to be necessary, since the general subject-matter of the application, i.e. the purification of the crude lactide mixture by distillation, would appear not to involve an inventive step.

- (ii) The Appellant then filed a new set of 18 claims as main request, of which Claim 1 reads as follows:
"A process for preparing a polylactide, the process including providing a crude lactide mixture including water, lactic acid, meso-lactide and at least one of the L-lactide and D-lactide; said process being characterized by the steps of:
(a) purifying the crude lactide mixture by distillation, without a step of solvent

extraction or recrystallization of the crude lactide mixture, into at least one purified polymer grade lactide fraction having a concentration of lactide of at least 99 % by weight; and

(b) reacting the purified polymer grade lactide fraction to form polylactide."

In addition to an objection under Article 123(2) EPC raised in general terms that the above process could not be derived clearly and unambiguously from the process specified in the application as originally filed, the Board reiterated its previous position that the general subject-matter of the application in suit did not seem to involve an inventive step and that this conclusion, consequently, also applied to the process as defined in Claim 1.

(iii) This led the Appellant to file following Claim 1 as the basis for an auxiliary request:

"A process for preparing a polylactide from lactic acid in solution with a hydroxylic medium, generating a crude lactide mixture including water, lactic acid, meso-lactide and at least one of the L-lactide and D-lactide; said process being characterized by the steps of:

(a) continuously purifying the crude lactide mixture by distillation in a distillation system to separate the lactide from water and lactic acid, without a step of solvent extraction or

recrystallization of the crude lactide mixture, into at least one purified polymer grade lactide fraction having a concentration of lactide of at least 99 % by weight; and

(b) reacting the purified polymer grade lactide fraction to form polylactide,

wherein the crude lactide mixture is introduced into the distillation system at a point above the bottom outlet."

In order to meet the requirements of Article 123(2) EPC the Appellant was invited to indicate the support in the original files for the disclosure of the amendments, especially for the location of the introduction of the crude lactide mixture into the distillation system. The Appellant considered Figures 2, 11 and 12 to provide adequate support for that feature.

- V. The Appellant requested that the decision under appeal be set aside and that a patent be granted on the basis of the main request or the auxiliary request both submitted during oral proceedings.

Reasons for the Decision

1. The appeal is admissible.

Main request

2. *Article 123(2) EPC*

As mentioned above, the Board questioned in general terms, e.g. without going into a detailed discussion of the support of the claimed subject-matter, the derivability of the two-step process according to the main request from the six-step process according to the application as originally filed. However, since it was clear from the Appellant's written submissions that the inventiveness of the distillation step was the key issue for the outcome of the case, it did not deem appropriate to first concentrate on the exact wording of Claim 1.

3. *Novelty*

Novelty was not disputed and since the Board is also of the opinion that none of the documents D1 to D4 discloses the combination of features specified in the present claims, there is no need to discuss that matter any further.

4. *Inventive step*

The application in suit concerns a continuous process for the manufacture of lactide and lactide polymers and is now with the amended claims restricted to the preparation of lactide polymers.

- 4.1 Such a process is disclosed in D2 which the Board, like the Appellant and the Examining Division, regards as representing the closest state of the art. This citation describes the preparation of polylactic acid (polylactide within the terminology used in the present application) from a crude lactide (i) which is first dissolved in an organic solvent which is liquid under the given conditions and not miscible with water, (ii) the solution being then extracted with water, and (iii) the lactide isolated from the organic solvent layer (Claim 1). Although the lactide purified by this extraction method has a high degree of purity which makes the resulting high molecular weight polylactic acids suitable as biodegradable polymers for pharmaceutical and surgical applications (column 4, lines 5 to 32), this process cannot be carried out at industrial scale and does not allow continuity.
- 4.2 In line with the introductory statement in the application in suit (page 6, lines 19 to 37) the technical problem may thus be seen in the provision of a simpler, more economical process for the preparation, purification and polymerization of lactide or, if defined in less ambitious terms, to provide an alternative method for the production of polylactide.
- 4.3 This problem is said to be solved with the means specified in Claim 1, especially by purifying the lactide mixture by distillation without a step of solvent extraction or recrystallization.

4.4 In view of the description (page 4, lines 23 to 36), especially the examples which show that this process provides a large scale, commercial manufacture of biodegradable polymers possessing physical properties suitable for applications in medical as well as non-medical products, the Board is satisfied that the above defined technical problem is effectively solved by the given means.

5. It remains to be decided whether the combination of features specified in Claim 1, in particular feature (a), i.e. purification by distillation, regarded by the Appellant as the only critical feature, is obvious having regard to the documents relied upon by the Examining Division.

5.1 As mentioned above, D2 describes a method for preparing high molecular weight polylactide in which a crude lactide mixture is purified by extraction from an organic solvent and then polymerized. No alternative method is envisaged in that citation, whose teaching must be regarded as an attempt to improve prior art methods based on one or several recrystallization step(s) (column 1, line 37 to column 2, line 9) and wherein, consequently, the solvent extraction must be regarded as an essential feature.

Thus the person skilled in the art would have no reason to depart from that teaching, which means that D2 considered in isolation cannot lead to the solution of the above defined technical problem.

5.2 D1 concerns the preparation and purification of lactide in large industrial scale. According to a preferred embodiment (i) optically pure lactic acid is dehydrated in the presence of Sn catalyst to form polylactic acid of average M_w between 400 and 2000, (ii) the polylactic acid is heated at low pressure in the presence of the

same catalyst to form a mixture which contains lactide, (iii) the lactide is distilled off this mixture, and (iv) the lactide is further purified by recrystallization (Claim 4 and example 2 in conjunction with page 4, lines 15 to 31). This method, which is based on a final recrystallization step, corresponds thus to the prior art referred to in D2. It is evident that this disclosure, whether considered in isolation or in combination with D2, cannot lead a skilled person to the claimed subject-matter which requires a different purification step.

5.3 In D3, which is directed to the preparation of optically active lactides, the crude lactide is recrystallized for further purification (cf. example 1). No information extending beyond that provided by D1 is thus made available, so that this citation cannot contribute to the solution of the above defined technical problem.

5.4 D4 relates to environmentally benign lactide acid polymers and to their production in large quantities at low prices for packaging and consumer goods as well as medical applications.

5.4.1 In this article emphasis is laid on the difficulties which had to be overcome for large-scale commercialization, namely the development of an advanced fermentation process technology and the development of a solvent extraction technology to obtain crude lactic acid (page 32, paragraph bridging left-hand col. and right-hand col.). Regarding the latter aspect it is specified that "Distillation can be combined with dehydration to yield lactide for use in polyester production" and that "The lactide could be further purified by redistillation or in some instances crystallization" (page 32, right-hand column, lines 6 to 12).

- 5.4.2 This explicit reference to distillation and crystallization shows that these methods are both suitable for the final purification step in the preparation of lactide. Since, from the documents discussed above, it has been established that recrystallization and solvent extraction are both suitable for that purpose, it is self evident that a skilled person faced with the above-defined technical problem would consider the distillation option.
- 5.5 In an attempt to demonstrate that it was not proper to reduce the investigations which led to the present application to a simple "could-would" alternative, the Appellant presented various arguments which either speak against or even tend to establish a prejudice against the use of distillation as the final purification step. These arguments, however, cannot be accepted for the following reasons.
- 5.5.1 The first argument was the time factor which was used to support the existence of a long felt need. In the Board's view, however, the period of 60 years wherein distillation was not used to obtain highly purified lactides cannot amount to an indication of a long felt need on which an inventive step could be based. During that period of time nobody thought to use distillation since there was no need to produce highly purified lactides economically. As specified on page 4, first full paragraph, of the present patent application, producers of medical-related biodegradable products were not concerned about low yields inevitable when using recrystallization; the same applied to extraction, because of the high margin generally expected for sales of such products. The problem of an economic production arose only in connection with large scale production of cheap biodegradable polymers. When a large scale economic production became necessary, there was a need for a continuous purification process

hardly possible with purification by recrystallization. It was thus obvious to purify by distillation instead of recrystallization known to cause high losses in the product to be recrystallized.

- 5.5.2 The interpretation which the Appellant sought to give of the critical passage in D4, namely that redistillation would apply to esters which were liquid at room temperature and recrystallization to lactides which were solid, is untenable in the light of the whole paragraph.

There, the authors explain the need to depart from the traditional methods and mention that several improvements can be envisaged. First, distillation can be combined with dehydration to yield the purified lactide monomer; alternatively, the lactic acid solution in a nonaqueous solvent can be integrated with an esterification reaction to obtain a volatile lactate ester. Thus, the final sentence, namely "The lactide or lactate ester could be further purified by redistillation or in some instances crystallization", can only mean that the starting material (lactide or lactate ester) can always be purified by redistillation and that, in some instances only, crystallization would be appropriate.

Consequently, it cannot be denied that this passage represents a clear incentive to use distillation in order to solve the technical problem.

- 5.5.3 The further argument that a skilled person would rather rely on extraction and recrystallization when the content of impurities was fairly low and consider distillation only for higher amounts is an unsupported allegation which speaks against the very teaching of D4. There is nothing in that citation which could be regarded as a limitation of the suitability of

distillation which would be related to the content of impurities. Moreover the Appellant did not provide any evidence demonstrating that a skilled person would not use such a common purification method as distillation to purify a crude lactide mixture.

5.5.4 Similarly, the arguments that the conditions existing during distillation would be similar to polymerization conditions and thereby initiate an undesirable reaction is not sustainable in view of the teaching of D4. As stated above, this document is explicitly concerned with a process for large-scale production of polylactic acids, wherein the lactide can suitably be purified by distillation. This can only mean that this method of purification is fully compatible with large-scale production, which is also the object of the application in suit.

5.5.5 It follows from these considerations that at the priority date of the application in suit there was no prejudice against the use of distillation in the final purification step of lactide. On the contrary, the prior art provided a strong incentive to rely on that simple method in view of the continuity and large scale requirements.

5.6 For these reasons the subject-matter as defined in Claim 1 derives in an obvious manner from the prior art cited and, consequently, does not involve an inventive step.

Auxiliary request

6. *Article 123(2) EPC*

One of the amendments made in Claim 1 of the auxiliary request filed during oral proceedings consists in the addition of "wherein the crude lactide mixture is introduced into the distillation system at a point above the bottom outlet."

The Appellant argued that said insertion was supported by Figures 2, 11 and 12 and that thereby the claimed subject-matter had not be extended beyond the content of the application as filed.

In line with the approach followed in decision T 169/83 OJ EPO 1985, 193 the Board considers figures to constitute a part of the description with the consequence that even in the absence of any indication in the description it is generally possible to derive a teaching therefrom. Amendments based on features only referred to in figures must, however, be clearly shown in the drawings originally filed and clearly, unmistakably and fully derivable from the drawings in structure and function by a person skilled in the art and so relatable by him to the content of the description as a whole as to be manifestly part of the invention.

In the present case the Board considers that from the figures relied upon by the Appellant it was not clearly derivable (i) that the point of introduction of the crude lactide is above the bottom outlet, (ii) nor that the location of the introduction into the distillation system is manifestly part of the invention.

Figure 2 is a detailed schematic representation of a preferred polylactide polymer production system in accordance with the invention (cf. page 21, lines 5 to 7). As specified on pages 22 to 29 what is shown is a production system without giving any detailed information as to the exact position of inlets or outlets to tanks, reactors, distillation columns or other apparatus. This is evident if one considers feature (a) of Claim 1 of the main request filed with the Statement of the Grounds of Appeal which requires at least three component streams, i.e. an overhead, a bottom and a sidedraw stream plus one for feeding crude lactide, thus four streams altogether, whereas the schematic representation shows three streams only. From that figure the position of the streams is thus not clearly, unmistakably and fully derivable, neither can a skilled person recognize that the location of the introduction is manifestly part of the invention.

Figure 11 is a schematic representation of a preferred alternative polylactide polymer production system incorporating catalyst removal (cf. page 21, lines 30 to 32). A more detailed description of the schematic process of that figure is given on page 38, line 10 to page 40, line 31. The sites of the removals are characterized as overhead, bottom and side streams. This characterization, and not the flow chart as such, provides an information on the position of the effluents. Such an information, however, is not given for the feed stream and it is thus not clearly derivable that the crude lactide mixture is introduced at a point above the bottom outlet. Also no indication for the place of introduction is given which would be manifestly part of the invention, since this part of the description is directed to effluents more specifically.

Figure 12 is a schematic representation of a preferred alternative polylactide polymer production system incorporating a catalyst bed reactor system (cf. page 21, lines 33 to 35). Like Figure 11, Figure 12 in conjunction with page 40, line 32 to page 43, line 6 shows only a rough location of the fractionated streams by referring to them as overhead, bottom and sidedraw streams. The exact location of e.g. sidedraw streams 365 and 369 cannot be inferred from the schematic drawing of Figure 12. The same applies for feed streams 363 and 351. It is thus not disclosed that feed stream 351 is introduced at a point above the bottom.

That such a rough indication of the location within the distillation column is only given for the effluents is also clear from page 13, lines 10 to 28. The Appellant failed to indicate an appropriate passage for the introduction of the crude lactide at a point above the bottom of the outlet in the original files, from which it would appear that this feature was manifestly part of the invention as originally filed.

Consequently, the schematic flowcharts relied upon by the Appellant do not provide adequate support for the amendments in Claim 1 which consequently offend against Article 123(2) EPC.

7. For the reasons given above, the subject-matter of Claim 1 of the main request does not involve an inventive step and that of Claim 1 of the auxiliary request does not meet the requirements of Article 123(2) EPC. In the absence of any request meeting the requirements of the EPC, the appeal must be dismissed.

Order

For these reasons it is decided that:


The appeal is dismissed.

The Registrar:



E. Gorgmaier

The Chairman:



C. Gérardin