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D E C I S I O N
of 22 March 2001

Case Number: T 0842/98 - 3.3.3

Application Number: 91200625.1

Publication Number: 0448177

IPC: C08G 7/02

Language of the proceedings: EN

Title of invention:
Preparation of polyketone polymers

Patentee:
SHELL INTERNATIONALE RESEARCH MAATSCHAPPIJ B.V.

Opponent:
BP International Limited

Headword:
-

Relevant legal provisions:
EPC Art. 56

Keyword:
"Inventive step - ex post facto analysis"

Decisions cited:
-

Catchword:
-



Case Number: T 0842/98 - 3.3.3

D E C I S I O N
of the Technical Board of Appeal 3.3.3
of 22 March 2001

Appellant: SHELL INTERNATIONALE RESEARCH
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Representative: -

Respondent: BP International Limited
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Decision under appeal: Decision of the Opposition Division of the
European Patent Office posted 29 June 1998
revoking European patent No. 0 448 177 pursuant
to Article 102(1) EPC.

Composition of the Board:

Chairman: R. J. Young
Members: B. L. ter Laan
J. C. M. De Preter

Summary of Facts and Submissions

I. Mention of the grant of European patent No. 0 448 177 in respect of European patent application No. 91 200 625.1, filed on 20 March 1991, claiming priority from an earlier application in the Netherlands (NL 9000677 of 22 March 1990), was published on 27 December 1995 (Bulletin 95/52) on the basis of a set of ten claims of which Claim 1 read:

"Process for the preparation of polyketone polymers, characterized in that

- a) linear polymers of carbon monoxide with one or more olefinically unsaturated compounds, in which polymers the units originating from carbon monoxide and the units originating from the olefinically unsaturated compound(s) occur in a substantially alternating order, are prepared by contacting the monomers at elevated temperature and pressure, in the presence of a diluent in which the polymers are insoluble or virtually insoluble, with a suitable catalyst,
- b) wherein the preparation is carried out continuously and during the stationary period the catalyst addition rate has a value k and the diluent addition rate has a value v ,
- c) wherein the running-in procedure is carried out by adding the feed streams to a reactor containing monomers and diluent and which is at the temperature and pressure chosen for the stationary period, and
- d) wherein at the beginning of the running-in period a catalyst addition rate is used which is lower than k and/or a diluent addition rate which is higher than v , and

- e) wherein during the running-in period the catalyst addition rate is raised and/or the diluent addition rate is reduced, this being done such that at the end of the running-in period they have substantially the values k and v respectively."

Claims 2 to 10 referred to preferred embodiments of the process according to Claim 1.

- II. On 24 September 1996 a Notice of Opposition against the granted patent was filed, in which the revocation of the patent in its entirety was requested on the grounds set out in Article 100(a) EPC.

The opposition was supported, *inter alia*, by the following documents:

D1: EP-A-0 336 459 and

D2: EP-A-0 305 011.

- III. By a decision taken on 26 March 1998 and issued in writing on 29 June 1998 the Opposition Division revoked the patent. That decision was based on the set of claims as granted.

The Opposition Division held that the claimed subject-matter lacked an inventive step (Article 56 EPC). Since D2, like the patent in suit, concerned a continuous process for the polymerization of CO/olefin copolymers, that document was considered to be the closest prior art. D2 did not specify the start-up procedure. The differences in the patent in suit between the Examples, in which terpolymers were described, and Comparative Examples, in which copolymers were prepared, did not

allow any conclusions as to any technical effect being achieved due to the specified start-up procedure, so that the problem to be solved was seen as to provide an alternative start-up procedure to D2. D1 disclosed a semi-batch process for the polymerization of CO/olefin copolymers, in which the amount of catalyst was increased stepwise until its final value. Since a semi-batch process and a continuous process were technically equivalent, the differences between batch and continuous processes would not deter the skilled person from considering features of a semi-batch process for application in a continuous process in order to provide an alternative start-up procedure. Consequently, the claimed subject matter lacked an inventive step.

- IV. On 20 August 1998 the Appellant (Proprietor) lodged an appeal against the above decision and paid the prescribed fee simultaneously. The Statement of Grounds of the Appeal, which was filed on 20 October 1998, was accompanied by a declaration of a Mr W.H. Hesselink, containing a calculated further example, and one of a Prof. Dr K.R. Westerterp, the latter concerning the behaviour of a continuous process during stationary and running-in periods.

The Appellant argued, in substance, as follows:

- (a) The claimed subject-matter was inventive. In particular, D2, which was considered to be the closest document, taught that the properties of the polymer, in particular the bulk density, could be influenced by adjusting certain process parameters in the stationary phase of a continuous process.

- (b) The problem to be solved by the patent in suit was to find an alternative way of increasing the bulk density of polyketone polymers produced by a continuous process, at the same time attaining a high slurry concentration.

- (c) The Examples, Comparative Examples and the calculated example filed with the Statement of Grounds of Appeal, showed that that problem was solved by the claimed process. The differences in detail between those examples did not influence the bulk density to the extent that they did not provide a valid comparison, as could be seen from the Examples in D2.

- (d) The solution provided was not obvious since the skilled person would have had to take two barriers to solve the above-defined problem: first the selection of the running-in period for applying measures and second the selection of which measures were to be taken. Since D2 referred solely to the stationary period, it contained no incentive regarding measures to be taken during the start-up period. D1 referred to batch and semi-batch processes, which were not the same as the start up period of a continuous process. Therefore, neither of D1 or D2, taken alone or together, would suggest the combination of measures as claimed with a view to enhancement of the bulk density, so that the claimed process was inventive.

In support of the arguments reference was made to the two declarations filed with the Statement of Grounds of Appeal.

V. The Respondent, in its response, cited seven new documents and argued along the following lines:

- (a) Starting from D2, the problem to be solved was to find an alternative way of increasing the bulk density of polyketone polymers in a continuous process.
- (b) The alleged effect was the mere consequence of reaching the stationary state of the continuous process, the definition of that state being, according to D2, that the suspension withdrawn from the reactor had a constant copolymer content and the copolymers contained therein had a constant bulk density.
- (c) By adjusting parameters during the stationary phase of the continuous process as described in D2, the process would move out of the stationary phase into a transitional state until a new stationary phase was reached. The parameters which could be adjusted in a continuous process were monomer feed rate, diluent feed rate, catalyst feed rate, product take-off rate, temperature and pressure. In order to maintain the stationary phase, the process parameters should be kept more or less constant. Therefore, the skilled person would regard the start up period of a continuous process as a transitional state and would consider that any measure increasing the bulk density of a copolymer when moving from one steady state to another via a transitional state would also be effective during the start up of a continuous process. Furthermore, the skilled person would also consider that only a limited number of

process parameters could be adjusted, some of which were interrelated. Therefore, his attention would be focused on the diluent addition rate, suspension withdrawal and catalyst addition rate.

Regarding D2, the prolongation of the residence time, which resulted in an increased bulk density, could only be accomplished by reducing the withdrawal of polymer suspension, which again would lead to an increase in the quantity of catalyst present in the reactor and hence a change from the steady state into a transitional state. The skilled person would realise that any measure leading to an increased bulk density when moving from a steady state to another via a transitional state, would also be effective during start-up and which he would therefore try. Hence, the claimed process was not inventive over D2 alone.

The same was true for a combination of D2 with D1, which taught an increase of bulk density by introducing a relatively low amount of catalyst at the beginning of the polymerization and to add more catalyst at a later stage and which the skilled person would find when looking for any measures known to improve the bulk density.

The difference between batch, semi-batch and continuous processes were not such that a skilled person would not adopt measures proposed for a batch process in a continuous process, as could be seen from six of the seven newly cited documents. Moreover, it was not relevant that the polymer formed during a semi-batch process remained in the reactor, which, according to the Appellant, formed

an essential difference between semi-batch and continuous processes to the extent that the skilled person would not think of applying measures from a batch process to a continuous process. In reality some of the polymer formed at the start-up of a continuous process would also remain in the reactor. Support for this could be found in the seventh of the newly cited documents. Also, if the withdrawal of polymerisation mixture in a continuous process was not initiated as from the beginning, which was not required by the claim and which would be considered because there was no polymer yet in the reactor, the semi-batch and continuous process would resemble each other even more closely. Since, however, a semi-batch start-up involved the removal of polymer at a later stage but before a steady state had been reached, the removal of polymer during start-up was also obvious over a combination of D1 and D2. It was noted that the Appellant had not provided any evidence that the immediate withdrawal of the polymerisation mixture had any influence on the bulk density.

VI. Oral proceedings were held on 22 March 2001. As announced in their letters dated 4 December 2000 (Respondent) and 7 February 2001 (Appellant), neither of the parties appeared. Both parties requested a decision based on the written submissions.

VII. The Appellant requested that the decision under appeal be set aside and the patent be maintained as granted.

The Respondent requested that the appeal be dismissed.

Reasons for the Decision

1. The appeal is admissible.

Procedural matters

2. Both parties had indicated that they would not attend the Oral Proceedings. In accordance with Rule 71(2) EPC, the proceedings therefore continued without them.
3. In its reply to the Statement of Grounds of Appeal, the Respondent cited seven new documents which had not been mentioned in the proceedings before. The Board came to the conclusion that these documents were not more relevant to the case in issue than the documents already on file, and were consequently not decisive for the outcome of the case. Therefore, the newly cited documents were disregarded (Article 114(2) EPC).

Novelty

4. The subject-matter claimed in the patent in suit was held to be novel by the Opposition Division and the Respondent did not make any novelty objection either. In the light of the cited documents, the Board sees no reason to depart from that view. Consequently, the subject-matter claimed in the patent in suit is held to be novel.

Problem and solution

5. The patent in suit concerns the preparation of polyketone polymers. Such preparation is described in D2, which the Board, in common with the parties and the Opposition Division, regards as the closest state of the art.

5.1 D2 describes a process for the preparation of copolymers, characterized in that a mixture of carbon monoxide with one or more olefinically unsaturated compounds is copolymerized by continuously contacting the mixture in steady state with a solution of a palladium-containing catalyst composition in the presence of liquid diluent in which the copolymers are almost or entirely insoluble, the quotient of the quantity of copolymer formed per hour and the quantity of copolymer in the reactor at any moment being less than 0.2 (Claim 1).

In the worked examples, a carbon monoxide/ethene copolymer (Example 1) and carbon monoxide/ethene/propene terpolymers (Examples 2 and 4) were prepared under conditions such that the quotient was 0.04, 0.02 and 0.04, leading to bulk densities of 0.54, 0.53 and 0.55, respectively, whereas the Comparative examples (Examples 3 and 5) were run with quotients of 0.26 and 0.28, resulting in bulk densities of 0.18 and 0.16, respectively (column 8, lines 20 to 42).

5.2 The object of D2 is to increase the bulk density of polyketones prepared in a continuous process (column 2, lines 8 to 33). The Appellant stated that the problem to be solved by the patent in suit was to find an alternative way of increasing the bulk

density of polyketones, whilst at the same time attaining a high slurry concentration, in line with page 3, lines 5 to 8 of the patent specification (Statement of Grounds of Appeal, page 2, point 3.1). The Respondent did not disagree with that view (Letter of 21 April 1999, page 2, second paragraph). Therefore, the technical problem to be solved by the patent in suit can be defined as to provide an alternative way of increasing the bulk density of polyketones.

- 5.3 The solution proposed according to claim 1 of the patent in suit is to operate the continuous process with a stationary state in which the catalyst addition rate has a value "k" and the diluent addition rate has a value "v" and (i) to choose, at the beginning of the running-in period, the catalyst addition rate and/or the diluent addition rate such that, at the beginning of the running-in period a catalyst addition rate is used which is lower than "k" and/or a diluent addition rate is used which is higher than "v", and (ii) during the running-in period, to raise the catalyst addition rate and/or reduce the diluent addition rate such that at the end of the running-in period they have substantially the values "v" and "k", respectively.

- 5.3.1 The examples in the patent in suit, in particular Examples 3 to 5, demonstrate that polyketones with a high bulk density at a high slurry concentration are produced using the above-described procedure.

- 5.3.2 Although there had been extensive discussions before the Opposition Division as to whether the examples and comparative examples were sufficiently similar,

particularly in terms of the monomers used in the polymerization mixture and the process conditions applied, to demonstrate convincingly that the claimed process did in fact lead to an increased bulk density of the polymer, and the Appellant spent a considerable part of its Statement of Grounds of Appeal on the subject (pages 2 to 5, point 4), the Respondent did not pursue the argument further before the Board.

5.3.3 From the Examples of D2 it can be seen that the presence or absence of propene monomer in the polymerization mixture does not influence the resulting polymer bulk density to the extent of the difference shown between the polymers in the examples of the patent in suit. More specifically, a comparison of Example 1 (CO/ethene copolymer) with Examples 2 and 4 (CO/ethene/propene/terpolymers) on the one hand and of (comparative) Example 3 (CO/ethene copolymer) with (comparative) Example 5 (CO/ethene/propene/terpolymer) on the other, shows that the bulk densities obtained with copolymers or terpolymers, also in view of other differences in process parameters, do not differ to a great extent. Consequently, the large increase in bulk density shown by the polymers in the illustrative examples of the patent in suit over the polymers of the comparative examples represent a convincing demonstration of the relevant effect.

5.3.4 Therefore, the Board comes to the conclusion that the above-defined problem is effectively solved by the claimed measures.

Inventive step

6. It remains to be decided whether the claimed subject-matter is obvious having regard to the documents on file.

6.1 The object of D2 is to increase the bulk density of polyketones prepared in a continuous process (column 2, lines 8 to 33), which is achieved by having a low quotient of the quantity of copolymer formed per hour and the quantity of copolymer in the reactor at any moment. This quotient can be decreased by reducing the quantity of polymer formed per hour by e.g. employing less or a less active catalyst or by reducing the temperature and/or the pressure. The quotient can also be reduced by raising the quantity of polymer in the reactor, which can be achieved by prolonging the residence time of the copolymer in the reactor (column 3, lines 1 to 11).

6.1.1 As can be seen from the wording of Claim 1 and the examples, the process of D2 concerns the steady state of a continuous polymerization process. It does not describe a changing system, nor the change of one steady state to another. The comparative terms "reduced" and "increased" refer to the parameters of known processes or processes outside the scope of D2 with which the process parameters of D2 are compared. They do not refer to actively increasing or reducing the values of those parameters whilst the continuous process is running. Neither does D2 contemplate a change from one steady state to another. There is no indication that first a "normal" steady state should be attained and then the conditions should be changed so as to arrive at the different conditions within the scope of D2. On the contrary, its teaching merely concerns the conditions of the steady state phase of

a continuous process. It is silent regarding the conditions of the start-up phase as well as any influence of the start-up conditions on polymer properties. For those reasons, the Board cannot concur with the Respondent's arguments that the skilled person would regard the measures according to D2 as moving from one steady state to another.

6.1.2 Therefore, D2 by itself does not contain any hint to the solution of the technical problem.

6.2 The addition, at the beginning of the polymerization, of only part of the final amount of catalyst is known from D1, which describes a process for the preparation of polymers, characterized in that a mixture of carbon monoxide with one or more olefinically unsaturated compounds is polymerized by contacting the mixture in a reactor with a solution of a palladium-containing catalyst composition in a diluent in which the polymers are insoluble or virtually insoluble, that the preparation is carried out batchwise, that 25-85% of the total amount of catalyst composition to be employed is present in the reactor at the outset of polymerization and that the remainder of the catalyst composition to be employed is added to the reactor only after the polymer concentration has reached a value of at least 0.2 g per 100 g polymer suspension (Claim 1).

In the comparative examples (Examples 1 to 4) and worked examples (Examples 5 to 7), carbon monoxide/ethene/propene terpolymers were prepared under conditions such that the amount of catalyst present at the beginning of the reaction was 100%, 95%, 15%, 50%, 72%, 83% and one third, respectively,

in Examples 2 to 7 the remainder being added after the polymerization concentration had reached a concentration of 6.0, 1.0, 0.1, 2.1, 0.3 and 2.0 g per 100 g polymer suspension, respectively. During polymerization the pressure was kept constant by the introduction of an ethylene/carbon monoxide mixture.

6.2.1 The object of D1, like that of D2 and the patent in suit, is to increase the bulk density of the polymer (column 1, line 35 to column 2, line 49), in particular a bulk density above 0.2 g/ml (column 2, lines 40 to 49). This is achieved by adding only part of the catalyst at the beginning of the polymerization and adding the remainder after the polymerization concentration has become high enough.

6.2.2 Since D1 concerns a batch process, however, it contains no information regarding continuous processes.

6.3 The Respondent's line of argument was mainly based on the allegation that the skilled person, when looking for measures useful to improve the bulk density of polyketones, would inevitably find D1 and would apply data obtained from a batch process to a continuous process.

6.3.1 Regarding the differences between batch and continuous processes, both D1 (column 1, lines 20 to 30) and D2 (column 1, lines 16 to 30) offer the following, identical, information:

"Batchwise preparation of the copolymers is carried out by introducing catalyst into a reactor containing diluent and monomers and which is at the desired

temperature and pressure. As polymerization proceeds, the pressure drops, the concentration of the copolymers in the diluent increases and the viscosity of the suspension rises. Polymerization is continued until the viscosity of the suspension has reached such a high value that continuing the process further would create difficulties in connection with heat removal."

D1 then adds:

"During batch wise polymer preparation, not only the temperature but also the pressure can be kept constant, if desired, by adding monomers to the reactor during polymerization." (column 1, lines 31 to 34).

After the above cited passage D2 goes on:

"In principle, the only parameter which remains constant in batchwise copolymer preparation is the temperature.

In continuous copolymer preparation, a diluent, monomers and catalyst are added to a reactor containing monomers and a certain volume of the diluent, and a copolymer suspension is continuously withdrawn from it. During the continuous copolymer preparation, the temperature, the pressure and the liquid volume in the reactor are kept constant. After a starting-up period in which the copolymer concentration in the suspension increases to the desired value, a stationary state (= steady state) is reached which is characterized by the suspension withdrawn from the reactor having a constant

copolymer content and the copolymers contained therein having a constant bulk density." (column 1, line 30 to column 2, line 7).

6.3.2 Therefore, from D1 and D2 it can be learned that a batch process is characterized by a constant temperature as the only constant reaction parameter, whereas in a continuous process the temperature, the pressure as well as the liquid volume in the reactor are kept constant. The start-up period in the latter process is defined as the period in which the copolymer concentration in the suspension increases, whereas in the steady state the suspension withdrawn from the reactor has a constant copolymer content. In a batch process, the pressure may also be kept constant by monomer addition (semi-batch process).

6.3.3 However, apart from its definition, nothing is said about the start-up period of continuous processes or the influence of the conditions during that phase on the properties of the reaction product. Nowhere in D1 or in D2 or in any of the other documents mentioned during the proceedings, can a basis be found for the Respondent's statement that the skilled person would consider the start-up phase of a continuous process to be the equivalent to a semi-batch process and to apply measures known for a semi-batch process to a continuous process. The fact that many documents contain general remarks such as: "the polymerisation may be carried out either batchwise or continuously" and that most processes are developed on bench scale in batch, is not sufficient to be regarded as a universal incentive to apply any arbitrary feature of a batch process to a continuous process. More in particular, such general statements cannot be seen as

an encouragement to apply specific measures used in a semi-batch process to a specific phase of a continuous process.

6.3.4 Consequently, there was nothing in the state of the art to encourage the skilled person to suppose that the conditions typical of a batch or semi-batch process would be useful in the start-up operation of a continuous process.

6.3.5 It may be that there are, in retrospect, certain similarities between the measures adopted according to the patent in suit to obtain a solution to the stated problem, and the characteristics of a batch or semi-batch process. It may even be that the stepwise increase in catalyst addition rate illustrated in the examples of the patent in suit involves the establishment of a series of steady states with transition states between them, as argued by the Respondent (submission dated 21 April 1999). Such insights are, however, dependent upon a knowledge of the teaching of the patent in suit, and do not arise from the state of the art. An argument based on such considerations is an *ex post facto* argument.

6.3.6 In particular, the finding of the decision under appeal, that "Therefore a skilled man would recognise the start-up period is [as] being technically equivalent to a semi-batch polymerisation..." is unsupported by any indication in the state of the art relied upon.

6.3.7 In summary, it has not been shown that the solution of the technical problem arises in an obvious way from the state of the art.

On the contrary, according to an uncontested statement in the declaration of Prof. Dr Ir. K. R. Westerterp, "... in a continuous process the properties of the polymer product are generally determined by the process conditions prevailing during the stationary period and not by the way in which these conditions are reached during the running-in period" (Declaration, page 1, third paragraph). Consequently, the solution provided according to the patent in suit is based on the rather surprising result, that a relevant intrinsic parameter of the product produced in the stationary state of a continuous process can be controlled by the running-in conditions, i.e those applying before the steady state was reached.

- 6.4 For the above reasons, the Board comes to the conclusion that the subject-matter of Claim 1 involves an inventive step.
7. As Claim 1 of the main request is allowable, the same is valid for dependent Claims 2 to 10, the patentability of which is supported by that of Claim 1.

Order

For these reasons it is decided that:

1. The decision under appeal is set aside.
2. The patent is maintained as granted.

The Registrar:

The Chairman:

E. Görgmaier

R. Young