Datasheet for the decision of 19 November 2008

Case Number: T 1765/06 - 3.3.05
Application Number: 99913995.9
Publication Number: 1064086
IPC: B01J 8/00
Language of the proceedings: EN
Title of invention:
Continuous slurry polymerization volatile removal
Patentee:
CHEVRON PHILLIPS CHEMICAL COMPANY LP
Opponent:
INEOS Manufacturing Belgium NV
Headword:
Continuous separation/CHEVRON
Relevant legal provisions:
EPC Art. 54(1)(2), 56, 83
Relevant legal provisions (EPC 1973):
-
Keyword:
"Disclosure (sufficient)"
"Inventive step (yes): change from semi-continuous into continuous mode - non obvious technical solution"
Decisions cited:
-
Catchword:
-
Case Number: T 1765/06 - 3.3.05

DECISION
of the Technical Board of Appeal 3.3.05

Appellant: INEOS Manufacturing Belgium NV
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Composition of the Board:
Chairman: G. Raths
Members: J.-M. Schwaller
S. Hoffmann
Summary of Facts and Submissions

I. This appeal was lodged by the opponent (hereinafter the appellant) against the decision of the opposition division rejecting the opposition against the European patent No. 1 064 086, independent claims 1 and 5 of which read as follows:

"1. An apparatus for removing and recovering polymerization liquid medium from a polymer produced in a reactor as a polymer slurry of particulate polymer solids suspended in a liquid medium comprising an inert diluent and unreacted monomers, the apparatus comprising,

   a discharge valve for continuously discharging a portion of said polymer slurry from said reactor into a first transfer conduit;

   said first transfer conduit communicating said polymer slurry therein into a first flash tank having a bottom communicating to a first flash tank exit seal chamber of a length (l) and a diameter (d) which provides a volume sufficient to maintain a volume of polymer solids/slurry sufficient to maintain a pressure seal in said seal chamber;

   said seal chamber having a seal chamber exit reducer, defined by substantially straight sides inclined at an angle to that of horizontal equal to or greater than the angle of slide of the polymer solids which remain after removal of about 50 to 100% of the inert diluent therefrom which communicates a continuous flow of concentrated polymer solids/slurry from said first flash tanks exit seal chamber to a second transfer conduit which communicates said continuous
flow of concentrated polymer solids/slurry to a second flash tank; and

said second flash tank operating at a substantially lesser pressure than that of said first flash tank such that essentially all of any remaining inert diluent and monomer is vaporized and communicated to a diluent and monomers recovery system through a flash tank overhead exit and essentially dried polymer solids are communicated to a dryer/storage tank.

5. A process for producing polymer comprising producing a polymer slurry in a liquid medium which comprises:

reacting a monomer in a hydrocarbon diluent inert to polymerization to form a polymerization effluent;

continuously discharging said polymerization effluent through a discharge valve into a first transfer conduit;

heating said polymerization effluent in said first transfer conduit to a temperature below the fusion temperature of the polymer;

continuously communicating said polymerization effluent through said first transfer conduit to a first flash tank wherein the pressure in said first flash tank and the temperature of said heated polymerization effluent are such as to produce as a vapor from about 50% to about 100% of the liquid medium and said vapor is condensable, without compression, by heat exchange with a fluid having a temperature in the range of about 65°F (18°C) to about 135°F (57°C);

continuously condensing said vapor obtained in said first flash step, without compression, by heat exchange with a fluid having a temperature in the range of about 65°F (18°C) to about 135°F (57°C);
continuously discharging from said first flash tank concentrated polymer solids/slurry to a second flash tank through a seal chamber wherein said seal chamber has a length \((l)\) and a diameter \((d)\) such as to maintain a volume of concentrated polymer solids/slurry in the said seal chamber sufficient to maintain a pressure seal;

continuously communicating said concentrated polymer solids/slurry to a second flash tank through a seal chamber exit reducer defined by substantially straight sides inclined at an angle to that of horizontal equal to or greater than the angle of slide of the polymer solids which remain after removal of about 50 to 100% of the inert diluent therefrom;

continuously exposing the remaining liquid medium in said concentrated polymer solids/slurry to a further pressure reduction from a higher pressure of from about 140 psia \((9.65 \times 10^5 \text{ Nm}^{-2})\) to about 315 psia \((2.17 \times 10^6 \text{ Nm}^{-2})\) in said first flash tank to a lower pressure of from about 15 psia \((1.03 \times 10^5 \text{ Nm}^{-2})\) to about 35 psia \((2.41 \times 10^5 \text{ Nm}^{-2})\) in said second flash tank wherein the pressure of said second flash tank and the temperature of said heated concentrated polymer slurry are such as to produce as a vapor substantially all of the remaining diluent and monomer and said vapor is condensable with compression and cooling; and

discharging from said second flash tank polymer solids which are substantially free of diluent or unreacted monomer.”

II. During the opposition procedure, the parties inter alia relied upon the documents:

The contested decision can be summarized as follows:

The examples and the content of paragraph [0028] of the description would allow the ordinary skilled worker to carry out the claimed invention.

The apparatus disclosed in D1 would not be suitable for a continuous flow of polymer particles between the flash tanks, because the pressure seal provided by a suitable volume of polymer solids/slurry would not be maintained in case of continuous flow.

Starting from D1 as the closest state of the art, the problem to be solved would be seen in the reduction or elimination of pressure variations and of the risk of plugging both in the settling leg and in downstream equipment. The non-obvious solution to this problem consisted in:

(a) a discharge valve in the apparatus for continuous removal of polymer slurry from the reactor, instead of a settling leg; and
(b) a continuous communication between the first and second flash tank by means of a seal chamber wherein a volume of the polymer solids/slurry served as the pressure seal,

Feature (b) contributed to the solution of the problem because a direct consequence of the continuous removal of polymer solids/slurry was that there were no extended settling times during which plugging in the product separation zone might occur. The danger of plugging was therefore at least reduced as compared to a process with intermittent settling periods.

IV. In its grounds of appeal dated 24 January 2007, the appellant objected to the contested patent under Article 100(a) and (b) EPC, arguing in particular that the subject-matter of claim 1 lacked novelty over D1 and that the subject-matter of independent claims 1 and 5 further lacked an inventive step over D1 in combination with either of D4, D5 or D6.

V. Under cover of the letter dated 15 June 2007, the respondent requested to reject the appeal as inadmissible. It also submitted observations in response to the grounds of appeal as well as a declaration and a set of amended claims as an auxiliary request.

VI. On 12 November 2008, the respondent submitted a new auxiliary request in replacement to the aforementioned one.

VII. At the oral proceedings, which took place on 19 November 2008, the admissibility of the appeal was
no longer contested by the respondent and the appellant declared that it no longer maintained its novelty objection based on document D1.

VIII. The appellant's arguments can be summarised as follows:

- The specification provided none of the details needed to achieve the "desiderata" features set out in the claims 1 and 5, namely to maintain "a volume of polymer ... sufficient to maintain a pressure seal" and to maintain "a continuous flow ... from first flash tank to second flash tank". Furthermore, it could be seen from D3 that the specification did not indicate the design criteria necessary to maintain the polymer slurry under mass flow conditions. Therefore the patent in suit was objectionable under Article 100(b) EPC.

- The subject-matter claimed lacked an inventive step, because the continuous removal of a polymer reaction slurry from the polymerization reactor was a standard for a long time, as could be seen from D4, D5 and D6. Furthermore the feature "maintaining a volume of polymer ... sufficient to maintain a pressure seal" which was functional in nature was obvious in the light of D1.

IX. The appellant requested that the decision under appeal be set aside and that the patent be revoked.

The respondent requested that the appeal be dismissed or alternatively, that the patent be maintained on the basis of the claims according to the auxiliary request filed under cover of the letter dated 12 November 2008.
Reasons for the Decision

1. Disclosure of the invention

1.1 According to the case law of the boards of appeal, in order to establish insufficiency of disclosure, the burden of proof is upon the opponent to show that the skilled reader of a disputed patent, using his common general knowledge, would be unable to carry out the invention claimed.

1.2 In the present case, the appellant contested the sufficiency of disclosure, arguing - without however providing any evidence in support of its allegations - that the specification provided none of the details needed to achieve the "desiderata" features set out in the claims 1 and 5 (see item VIII. above). Specifically, it objected to the absence of an indication concerning the size of the orifice of the seal chamber exit reducer. It also submitted document D3 in order to show that the specification did not indicate the design criteria necessary to maintain the polymer slurry under mass flow conditions, so as to maintain a pressure seal and avoid the tendency of funnel flow in the seal chamber.

1.3 The board cannot accept these arguments because it appears to be plainly within the competence of the skilled person to calculate or to find out by routine experimentation the size of the orifice of the seal chamber exit reducer in order to reach the desired effect - namely to maintain a volume of concentrated polymer solids/slurry in the seal chamber sufficient to
maintain a pressure seal and to maintain a continuous flow of polymer solids/slurry through the seal chamber and the seal chamber exit reducer. Furthermore, both independent claims 1 and 5 and the description, in particular the Examples, disclose sufficient details as regards the design of the apparatus necessary for achieving said desired effect, in particular the requirement of a seal chamber having a length \((l)\) and a diameter \((d)\) and a seal chamber exit reducer having substantially straight sides inclined at an angle to that of horizontal equal to or greater than the angle of slide of the polymer solids which remain after removal of about 50 to 100% of the inert diluent.

The board is further convinced that it is within the competence of the skilled person to optimize the design of the seal chamber and of its exit reducer so as to maintain the polymer slurry under mass flow conditions, i.e. without breaking the pressure seal, in particular by following the advice given in the excerpt D3 of a standard engineering textbook, which clearly and unambiguously teaches (page 7-25, left column, 5th paragraph) that Jenike's method (i.e. the one described in D3) "allows the chemical engineer to design bulk-storage vessels and to weigh cost vs. performance with a high level of confidence that, if the conditions in the real storage system are the same as those prevailing during the test, the product will flow. It is up to the engineer, though, to establish the bounds of conditions the product will encounter and to make appropriate tests".

1.4 For the above reasons, the board has no doubt that the invention as defined by the apparatus and process
according to present claims 1 and 5 is disclosed in a manner sufficiently clear and complete to be carried out by a person skilled in the art (Article 83 in conjunction with Article 100(b) EPC).

2. **Novelty**

The claimed subject-matter complies with the requirements of Articles 52(1) and 54(1)(2) EPC, because D1 does not directly and unambiguously disclose that the valve located, on the one hand, between the polymerization reactor and the first flash vessel and the valve located, on the other hand, between the two flash vessels are both suitable for being operated in a continuously open mode.

3. **Inventive step**

3.1 The contested patent relates to an apparatus and a process for recovering polymer solids from a polymerization effluent comprising a slurry of polymer solids in a liquid comprising an inert diluent and unreacted monomers, the slurry being discharged to a first flash tank wherein from about 50% to about 100% of the liquid medium are vaporized, said flash tank communicating the concentrated polymer solids/slurry into a second flash tank wherein essentially all of any remaining inert diluent and/or unreacted monomer is vaporized and removed overhead (paragraphs [0008] and [0009] of the patent in suit).

3.2 In agreement with the parties, document D1 is taken as the starting point for assessing inventive step, as it concerns a method and apparatus for recovering polymer
solids from a polymerization zone effluent comprised of a slurry of the polymer solids in a liquid diluent, said method comprising vaporizing the diluent by exposing the effluent to a pressure drop, at least partially, in a first flash zone of a cyclone type with an extended solids receiving zone; separating the diluent vapor from the effluent in the first flash zone; removing the diluent vapor from the first flash zone; condensing the diluent vapor; recycling at least part of the condensed diluent to the polymerization zone; allowing the polymer solids to pass into the extended solids receiving zone where they are held until it is at least partially full; thereafter, passing the polymer solids from the extended solids receiving zone to a second flash zone where they are exposed to a second pressure drop which vaporizes residual diluent remaining with the polymer solids; and separating the residual diluent vapor from the polymer solids (column 2, lines 1 to 38).

In the embodiment illustrated in its Figure 1, polymerization is carried out in a loop reactor (10) and the polymer effluent is removed therefrom to a settling leg (22), from which the effluent is passed via a valve to conduit (24) and into flash vessel (28) (column 2, lines 49 to 60). The pressure drop within the flash vessel is such that the major portion of diluent associated with the polymer solids leaving the settling leg is vaporized and removed as vapor via conduit (30) (column 4, lines 5 to 9 and 23 to 25). Flash vessel (28) is in the form of a cyclone having downstream an extended solids reservoir (32) wherein the polymer solids settle (column 2, lines 49 to 65). When the extended solids reservoir is at least
partially filled, its content flows via conduit (48) into a lower pressure second flash vessel (50). The passage of the polymer solids from said extended solids reservoir into conduit (48) is controlled by valve (46) which is fully open when the polymer solids flow to lower pressure flash vessel and fully closed at other times (column 3, lines 9 to 17).

It is uncontested that the aforementioned process and apparatus do not work in a continuous mode, since the polymer solids/slurry and the concentrated polymer solids/slurry are both collected in the settling leg (22) and in the extended solids reservoir (32), respectively, before being periodically transferred to the first and second flash vessels (28) and (50), respectively.

3.3 The problem underlying the patent in suit in the light of document D1 is to eliminate the intermittent high pressure pulses within the equipment and the plugging in the equipment downstream the polymerization reactor (see also paragraphs [0010] to [0012] of the patent in suit) while maintaining the pressure difference between the flash vessels.

3.4 As a solution to this problem, the patent in suit proposes an apparatus and a process according to claims 1 and 5, respectively.

The apparatus according to claim 1 is characterized by:

(a) a discharge valve for continuously discharging a portion of the polymer slurry from the polymerization reactor;
(b) the first flash tank having at its bottom an exit seal chamber of a length \( l \) and a diameter \( d \) sufficient to maintain a volume of polymer solids/slurry sufficient to maintain a pressure seal, the seal chamber having an exit reducer defined by substantially straight sides inclined at an angle to that of horizontal equal to or greater than the angle of slide of the polymer solids which remain after removal of about 50 to 100% of the inert diluent therefrom, said reducer communicating a continuous flow of concentrated polymer solids/slurry to the second flash tank.

The process according to claim 5 is characterized by:

(c) continuously discharging the polymerization effluent through a discharge valve and continuously communicating it to the first flash tank;

(d) continuously discharging the concentrated polymer solids/slurry from the first flash tank through a seal chamber having a length \( l \) and a diameter \( d \) such as to maintain a volume of concentrated polymer solids/slurry sufficient to maintain a pressure seal and continuously communicating the concentrated polymer solids/slurry to a second flash tank through a seal chamber exit reducer defined by substantially straight sides inclined at an angle to that of horizontal equal to or greater than the angle of slide of the polymer solids which remain after removal of about 50 to 100% of the inert diluent therefrom.
3.5 The board is satisfied that the technical problem as defined under point 3.3 is effectively solved, because a process working in a continuous operating mode is manifestly less sensitive to plugging and avoids intermittent high pressure pulses (see patent in suit, paragraphs [0010] to [0012]) in comparison with the same process working in a semi-continuous mode, such as the one described in D1. The same remark applies to the apparatus according to claim 1, the design of which allows continuous operation.

3.6 The question which thus remains to be answered is whether the solution as proposed by the subject-matter claimed is obvious or not in view of the cited prior art.

3.7 Concerning appellant's argument that the continuous removal of the polymer reaction slurry from the polymerization reactor was a standard for a long time, the board observes that D4 (column 2, lines 52 to 59), D5 (column 6, lines 3 to 9) and D6 (column 4, lines 53 to 57) indeed disclose the continuous withdrawal of the polymer product from the reaction zone, however as can be seen hereinafter the disclosure of these documents is not sufficient to render obvious the subject-matter of present claims 1 and 5.

3.8 D6 (column 1, lines 5 to 8; claim 1) in fact relates to a pressure responsive system for controlling removal of a reaction slurry from a polymerization reactor, said method comprising controlling the flow rate of the reaction slurry in a primary effluent stream in a continuous manner by manipulating a v-notch valve; and
automatically initiating withdrawal of said reaction slurry through a secondary effluent conduit if said primary effluent conduit becomes plugged with said reaction product.

As indicated at column 1, lines 17 to 25, a polymerization process typically employs a relatively high pressure continuous reactor which may have a downwardly depending settling leg to facilitate removal of the produced polymer slurry. The slurry of produced solid polymer and diluent is thereafter discharged into a separation chamber where the slurry is flashed to a low pressure.

D6 (column 4, lines 53 to 57) further discloses that the continuous removal of the reaction effluent stabilizes the polymerization reactor pressure as well as the feed flow and other reactor conditions.

D6 does however not disclose a two-stage flash separation process, let alone a two-stage flash separation process operating at two different pressures and working in a continuous manner, such as the one defined in present independent claims 1 or 5.

So, even if the skilled person faced with the problem defined under item 3.3 would take into consideration the aforementioned passage of D6 (column 4, lines 53 to 57) and contemplate the continuous removal of the reaction slurry as representing a promising way of solving said problem, it would however arrive neither at the subject-matter of claim 1, nor at that of claim 5, since D6 - which only envisages "a separation chamber where the slurry is flashed to a low pressure",.
i.e. a **one-stage** flash separation process - neither discloses, nor suggests the features referred to as (c) and (d), respectively, in item 3.4 supra.

### 3.9

Concerning the appellant's argument that the wording of independent claims 1 and 5 regarding the feature "maintaining a volume of polymer ... sufficient to maintain a pressure seal" was functional in nature, and that this functional feature was obvious in the light of D1 - which would implicitly disclose the use of the concentrated polymer solids slurry leaving the extended solids reservoir as a pressure seal - the board observes that the disputed independent claims 1 and 5 not only disclose the above functional features, but these features are also defined in concrete technical terms. In particular, they require that the first flash tank includes a seal chamber having a length (l) and a diameter (d) and a seal chamber exit reducer defined by substantially straight sides inclined at an angle to that of horizontal equal to or greater than the angle of slide of the polymer solids which remain after removal of about 50 to 100% of the inert diluent therefrom. These features are however neither disclosed, nor suggested in D1.

Concerning the appellant's argument that the concentrated polymer solids slurry leaving the extended solids reservoir and flowing to the lower pressure flash vessel would work as a pressure seal maintaining the pressure difference between the two vessels for the period of time during which the valve (46) between the two vessels is maintained open, this might well be possible. However, D1 explicitly requires that the transfer of the concentrated polymer solids slurry from
the higher pressure flash vessel (28) to the lower pressure flash vessel (50) be carried out batchwise and with the valve separating both flash vessels being closed during the collection of the polymers solids (column 4, lines 36 to 41 and 50 to 53), i.e. exactly the opposite to what is required in the subject-matter of claims 1 and 5. So, it must be concluded that D1 did not plainly recognize the benefits of a polymer solids pressure seal. Since D1 furthermore does also not give any hint how to maintain the pressure difference between said flash vessels when the valve (46) would be permanently kept open, the board does not see how D1 would teach the use of a polymer solids slurry pressure seal in a process working in a continuously operating mode, such as the one defined in present claims 1 and 5.

3.10 Concerning documents D4 and D5, it is observed that apart from the disclosure that the withdrawal of the polymer reaction product from the polymerization zone may be carried out either continuously or periodically (D4: column 2, lines 58 and 59; D5: column 6, lines 6 to 9), these documents - alike D6 - do also not disclose a two-stage flash separation process, let alone a two-stage flash separation process operating at two different pressures and working in a continuous mode, such as the one defined in present independent claims 1 or 5, so that the reasons given with respect to document D6 in items 3.8 and 3.9 supra apply mutatis mutandis to the content of documents D4 and D5.

3.11 The remaining documents cited during the opposition proceedings do not contain further information which would point towards the claimed solution of the technical problem stated under point 3.3 supra.
Accordingly, for the reasons indicated above, the subject-matter of independent claims 1 and 5, and by the same token that of dependent claims 2 to 4 and 6 to 15, which include all the features of either claim 1 or claim 5, cannot be considered as being obvious to a person skilled in the art in view of the cited prior art. Therefore, the subject-matter of claims 1 to 15 involves an inventive step within the meaning of Articles 52(1) and 56 EPC.

Order

For these reasons it is decided that:

The appeal is dismissed

The Registrar: C. Vodz

The Chairman: G. Raths