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Bezeichnung der Erfindung: Method and apparatus of distillation for readily polymerizable liquid
Title of invention:
Titre de l'invention :

Klassifikation / Classification / Classement : C07C 67/54

ENTSCHEIDUNG / DECISION

vom / of / du 23 February 1988

Anmelder / Applicant / Demandeur :

Patentinhaber / Proprietor of the patent / Titulaire du brevet : Asahi Kasei Kogyo Kabushiki Kaisha

Einsprechender / Opponent / Opposant : Degussa AG

Stichwort / Headword / Référence : Distillation/Asahi Kasei Kogyo

EPÜ / EPC / CBE Article 56

Kennwort / Keyword / Mot clé : "Inventive step (denied)"

Leitsatz / Headnote / Sommaire

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Case Number : T 282/86 - 3.3 1

D E C I S I O N
of the Technical Board of Appeal 3.3.1
of 23 February 1988

Appellant : (Opponent)
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Representative :

Respondent : (Proprietor of the patent)
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Representative :
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Decision under appeal :
Decision of the Opposition Division of the European Patent Office of 15 April 1986 posted on 24 June 1986 rejecting the opposition filed against European patent No. 0 046 980 pursuant to Article 102(2) EPC.

Composition of the Board :

Chairman : K.J.A. Jahn
Members : R.W. Andrews
C.V. Payraudeau

Summary of Facts and Submissions

I. The mention of the grant of the patent No. 0 046 980 in respect of European patent application No. 81 106 631.5, filed on 26 August 1981 and claiming priority of 29 August 1980 from two prior applications in Japan, was announced on 14 December 1983 (cf. Bulletin 83/50) on the basis of seven claims. The independent Claims 1 and 6 read as follows:

"1. A method for distilling a readily polymerizable liquid by vaporizing the polymerizable liquid from a liquid phase containing the same in an evaporator by externally heating the liquid phase characterized in that it comprises the steps of:

(a) converting the vapor of the polymerizable liquid to a superheated state by externally heating, and introducing the vapor of the polymerizable liquid to the inlet of a condenser, while the vapor is maintained at the superheated state; and, then,

(b) condensing the vapor in the condenser while a portion of the condensed fraction is circulated to the inlet of the condenser, whereby the condensation surface thereof is wetted.

6. An apparatus for carrying out the distillation of a readily polymerizable liquid according to Claim 1 comprising:

an evaporator, external heating means, a conduit pipe and a condenser for condensing the evaporated vapor of the liquid, characterised by

(a) an evaporator (10) provided with at least two external heating means (16) and (11) which are

located above and below the upper end (14) of the heating jacket (11) which is located above the evaporating surface (13) of the liquid to be evaporated therein,

(b) a conduit pipe (21) provided with the heating means (23) for connecting the evaporator (10) and the condenser (22); and

(c) a circulating means for circulating a portion of the condensed fraction of the condenser (22) to the vapor inlet of the condenser (22), wherein the condensation surface of the condenser is wetted."

II. On 24 August 1984 the Appellant filed a notice of opposition requesting the revocation of the patent on the grounds of lack of inventive step in the light of the disclosure in US-A-3 988 213 (1).

III. By a decision of 15 April 1986, posted on 24 June 1986, the Opposition Division rejected the opposition on the basis that at least the step of condensing the superheated vapor of the readily polymerisable liquid in a condenser, the condensation surfaces of which are wetted by a recycled portion of the condensed liquid fed to the inlet thereof, could not be derived in an obvious manner from the prior art. Therefore, the Opposition Division decided that this feature alone was a sufficient basis to justify the conclusion that the subject-matter of Claims 1 and 6 involved an inventive step.

IV. An appeal was lodged by the Opponent against this decision on 20 August 1986 with the payment of the appropriate fee. A statement of grounds of appeal was submitted on 24 October 1986.

In this statement and during the oral proceedings held on 23 February 1988 the Appellant argued that the difference between the apparatus of the disputed patent and that disclosed in document (1) is an inevitable consequence of changing from a rectification process to a simple distillation process. Thus, if the perforated trays are removed from the column of document (1) a distillation column is obtained whose inner wall is heated to a temperature high enough to prevent the condensation of any vapor on its rough surface. The Appellant also contended it would be obvious to heat the pipe connecting the evaporator to the condenser since condensation of vapor with the resulting polymer formation would quickly block the pipe in view of its narrow cross-section.

With regard to step (b) of the claimed process the Appellant alleged that document (1) teaches that the vapor of the readily polymerisable liquid should be only condensed on metal surfaces which are continuously wetted. Furthermore, EP-A-0 014 041 (2) discloses that the problem of polymer formation during the purification of readily polymerisable monomers may be solved by contacting a liquid stream of cold purified monomer with the vapors of the hot crude monomer so as at least partially condense them in the cold purified monomer stream.

V. The Respondent denied that the removal of the perforated trays from the rectification column of document (1) would result in the invention according to the disputed patent. Thus, according to the patent-in-suit the wall of the evaporator, which corresponds to the reboiler of document (1), is provided with separate heating means for that part in contact with the liquid and that part in contact with the vapor. Moreover, the vapor is superheated and is maintained in this state until it is shock-cooled in the condenser whose surfaces are continuously wetted by cold purified liquid, optionally containing a polymerisation inhibitor. The Respondent also contended that the surfaces

of the trays of the rectification column of document (1) are wetted by a solution of polymerisation inhibitor, whereas the presence of a polymerisation inhibitor was not essential in the process according to the patent-in-suit.

Document (2) was irrelevant since the vapor is produced by flash evaporation and is not superheated when contacted with the cold purified monomer. Moreover, there was no teaching in this document that the vapor should only be condensed on metal surfaces which are wetted.

The following statement was entered in the minutes of the oral proceedings: "The representative of the patentee declares that the claims do not cover rectification but only encompass a single stage or simple distillation".

VI. The Appellant requested that the decision under appeal be set aside and the patent-in-suit revoked. The Respondent requested that the appeal be dismissed.

Reasons for the Decision

1. The appeal complies with the requirement of Articles 106 to 108 and Rule 64 EPC and is, therefore, allowable.

2. In agreement with the above-mentioned declaration by the Patentee's Representative the Board considers that Claims 1 to 5 of the patent-in-suit must be construed as covering only a simple distillation process. Thus, all the vapor produced by heating the readily polymerisable liquid is conveyed to the condenser and condensed; i.e. no separation taking place in the vapor state.

3. The patent-in-suit relates to a process for the purification of readily polymerisable liquids by distillation and an apparatus for carrying out the claimed

process. The distillation of these types of liquids is known in the art. However, it was found that the condensed monomeric liquid being unstable gave rise to polymer formation nucleating on the interior surface irregularities of the distillation apparatus and that once polymerisation was initiated in this manner it proceeded at an accelerated rate resulting in loss of product and in the shut-down of the distillation unit.

In the light of this prior art the technical problem underlying the disputed patent may be seen in providing a process for the purification of readily polymerisable liquids by distillation in which undesired polymerisation of the monomer is prevented. According to the disputed patent this technical problem is essentially solved by superheating the vapor of the readily polymerisable liquid and condensing the superheated vapor in a condenser whose surfaces are continuously wetted by the introduction of some of the condensed purified liquid monomer to its inlet.

In view of the results in the Examples, the Board is satisfied that the technical problem is credibly solved.

4. After examination of the cited prior art the Board has reached the conclusion that this technical teaching is not disclosed in any of them and the claimed subject-matter is, therefore, novel. Since novelty is not disputed it is not necessary to consider this matter in detail.

5. It still remains to be examined whether the requirement of inventive step is met by the claimed subject-matter.

5.1 Document (1) discloses a process for the purification of certain polymerisable vinyl monomers by distillation in a column having perforated plates whose inner wall is heated to a sufficiently high temperature to prevent the

condensation of the vapor of the polymerisable vinyl monomer thereon. The temperature of the inner wall of the column should be maintained at a temperature up to 30°C higher than that of the vapor (cf. Claim 1). From the teaching of this document a skilled person would realise that during the distillation of readily polymerisable liquids it is necessary to prevent the vapor condensing on the surface of the column wall where polymerisation of the condensed monomer can occur.

Applying this teaching to the solution of the technical problem underlying the patent-in-suit as defined above, the skilled person would take steps to prevent the condensation of the vapor of the readily polymerisable monomer on the walls of the distillation vessel and the conduit connecting this vessel to the condenser by maintaining those surfaces in contact with the vapor at a temperature above the dew point of the vapor at the prevailing pressure. Since it is generally recognised in the art that polymerisation in the gaseous phase only occurs at very high temperatures the skilled person would not be deterred from maintaining the vapor of the readily polymerisable liquid at a temperature of up to 50°C higher than the dew point of the vapor at the working pressure, as suggested by a preferred embodiment of the disputed patent.

5.2 This document also discloses that a rectification column containing perforated trays was used in preference to either a packed or bubble cap tray column because it was found to be essential that the condensation of the vapor of the polymerisable vinyl monomer should only take place on metal surfaces which are continuously wetted by a solution of a polymerisation inhibitor in the monomer. Thus, the top and bottom of the trays on which the condensation occurs are always wetted by the said solution and the risk of polymerisation of the monomer is thereby minimised (cf. column 2, line 56 to column 3, line 11).

Although reference in this document is only made to the perforated trays being wetted with a solution containing a polymerisation inhibitor it would be clear to the skilled person from studying Figure 1 of this document and the Examples that, in view of the low volatility of the exemplified polymerisation inhibitors, hydroquinone and phenothiazine, the perforate trays above the feed inlet, particularly the ones at the head of the column, would be actually wetted by substantially pure liquid monomer. This would provide the skilled person with the incentive to carry out the condensation of the vapor of an easily polymerisable liquid in the presence of the pure liquid monomer.

Further encouragement to adopt this procedure to solve the problem underlying the disputed patent of preventing the condensed vapor from polymerising is provided by the teaching of document (2). This document discloses a process for purifying readily polymerisable vinyl monomers with boiling points above 170°C at atmospheric pressure by partially vaporising the crude monomer and condensing at least some of the vapor by contacting it with a stream of cold purified monomer which optionally contains a polymerisation inhibitor (cf. Claims 1 and 6).

Thus, the two techniques applied to solve the problem underlying the disputed patent of ensuring that the vapor of the readily polymerisable liquid does not condense on the surfaces of the distillation vessel and the conduit connecting this vessel to the condenser by external heating and condensing the vapor on surfaces wetted by the cold purified monomer are disclosed in documents (1) and (2).

Although the present process is a simple distillation process (cf. The declaration of the Patentee's Representative and paragraph 2 above) whereas documents (1)

and (2) disclose a rectification process and a flash evaporation process respectively, all three processes are related to each other insofar as they are processes for the purification of easily polymerisable liquids involving the vaporisation of the crude liquid monomer. Therefore, the skilled person would realise that the techniques disclosed in these prior art documents for preventing polymer formation during purification may be applied to a distillation process.

In the Board's judgement, therefore, the subject-matter of Claim 1 of the patent-in-suit does not involve an inventive step.

Dependent Claims 2 to 5, which relate to preferred embodiments of Claim 1, do not contain any independent inventive features and are, therefore, also unpatentable in the absence of an allowable Claim 1.

Claim 6 and its dependent Claim 7, which are directed to an apparatus for carrying out the distillation of a readily polymerisable liquid according to Claim 1, are also unallowable. The subject-matter of these claims does not involve an inventive step since it would be obvious to furnish the necessary technical measures to render a conventional distillation apparatus suitable for carrying out the distillation process according to the patent-in-suit. Moreover, it was neither asserted nor can it be recognised, that this subject-matter possess any inventive merit in its own right.

Order

For these reasons, it is decided that:

1. The decision of the Opposition Division is set aside.

2. The patent is revoked.

The Registrar: The Chairman: