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Datasheet for the decision of 9 February 2023

Case Number: T 0321/19 - 3.3.10

Application Number: 13700521.1

Publication Number: 2802559

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Language of the proceedings: EN

Title of invention:

PROCESS FOR THE SYNTHESIS OF UREA COMPRISING A PASSIVATION STREAM AT THE STRIPPER BOTTOM

Patent Proprietor:

Saipem S.p.A.

Opponent:

CASALE SA

Headword:

PROCESS FOR THE SYNTHESIS OF UREA COMPRISING A PASSIVATION STREAM AT THE STRIPPER BOTTOM/Saipem

Relevant legal provisions:

EPC Art. 56
RPBA Art. 12(4)

Keyword:

Inventive step - (yes)
Late-filed document - admitted (no)

Decisions cited:

Catchword:

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Case Number: T 0321/19 - 3.3.10

DECISION
of Technical Board of Appeal 3.3.10
of 9 February 2023

Appellant: (Opponent)

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Decision under appeal: **Decision of the Opposition Division of the European Patent Office posted on 29 November 2018 rejecting the opposition filed against European patent No. 2802559 pursuant to Article 101(2) EPC.**

Composition of the Board:

Chairman	P. Gryczka
Members:	J.-C. Schmid
	L. Basterreix

Summary of Facts and Submissions

I. The appellant (opponent) lodged an appeal against the decision of the Opposition Division rejecting the opposition against European patent No.2 802 559, independent claims 1, 12 and 14 thereof reading as follows:

"1. An enhanced process for the preparation of urea from ammonia and carbon dioxide, at high temperature and pressure, with the formation of ammonium carbamate as intermediate, comprising the following steps in the synthesis section:

(i) reacting ammonia and carbon dioxide at an overall pressure ranging from 12 to 20 MPa, with a molar ratio NH_3/CO_2, as such or in the form of ammonium carbamate, ranging from 2.1 to 6. preferably from 2.8 to 4.5, in a reaction step carried out in a suitable vertical reactor R fed with at least one ammonia stream and at least one stream of fresh carbon dioxide containing a passivation agent in such a quantity that its equivalent oxygen content is at least 0.1%, preferably from 0.15 to 0.30% in moles with respect to the moles of carbon dioxide, with the formation of a first liquid mixture containing urea, ammonium carbamate, water and ammonia, in liquid/vapour equilibrium with a gaseous phase containing at least a part of the passivation agent;

(ii) transferring said first liquid mixture to at least one decomposition-stripping step carried out in a suitable vertical apparatus, said stripper (S), operating at a pressure of at least 0.1 MPa, preferably from 0.2 to 2.0 MPa, lower than that of said reaction step (i);

(iii) heating said first liquid mixture in said decomposition-stripping step, to effect the decomposition of part of the ammonium carbamate into ammonia and carbon dioxide, and simultaneously subjecting said liquid mixture to stripping with the formation of a first gaseous mixture containing ammonia and carbon dioxide, and a second liquid mixture containing urea, water, ammonia and the non-decomposed part of the ammonium carbamate;

(iv) transferring at least a part, preferably all, of said first gaseous mixture to at least one condensation step, operating at the same pressure as the decomposition-stripping step (iii) and condensing the gaseous mixture transferred with the formation of a third liquid mixture containing ammonium carbamate, water and ammonia;

(v) transferring said third liquid mixture and the possible remaining part of said first gaseous mixture to the reaction step (i);

characterized in that at least a part of said gaseous phase in equilibrium with the first liquid mixture of step (i), is separated in a gas-liquid separator (D1) situated downstream of the reactor (R) and operating at the same pressure, to form a second gaseous mixture containing a passivation agent, which is fed to the lower section of said stripper (S)."

"12. A plant for effecting the process according to any of the claims from 1 to 11, comprising a synthesis section in which a vertical reactor (R) is in fluid connection with a vertical stripper (S) of the falling liquid film tube-bundle type,

characterized in that a gas-liquid separator (D1) is interposed between said reactor and said stripper, connected at one side to the head of the reactor (R), from which two fluid connection lines with said stripper (S), are generated, so that a transport line of a gaseous mixture connects the head of said separator (D1) with the lower section of the stripper (S) and a transport line of a liquid mixture connects the bottom of said separator (D1) with the upper section of the same stripper (S)."

"14. A method for enhancing a pre-existing process for the production of urea starting from ammonia and carbon dioxide with the intermediate formation of ammonium carbamate, which operates with a high-pressure synthesis section comprising:

- a reaction step carried out in a vertical reactor (R) fed with at least a stream of fresh carbon dioxide and at least a liquid ammonia stream, operating at an overall pressure ranging from 12 to 20 MPa, with a NH_3/ CO_2 molar ratio, as such or in the form of ammonium carbamate, ranging 2.1 to 6, preferably from 2.8 to 4.5, with the formation of an outgoing liquid mixture in the upper zone of the reactor, containing urea, water, ammonia and non-converted ammonium carbamate;

- a decomposition-stripping step of the ammonium carbamate in said liquid mixture with the separation of a gaseous stream containing carbon dioxide and the ammonia thus formed, carried out in a suitable vertical tube bundle apparatus said stripper (S) situated downstream of said reactor (R); and

- a condensation step in a condenser (C) of the gaseous stream leaving said stripper (S), with the formation of a liquid stream containing ammonium carbamate, fed as recycled product, to said first reactor,

characterized **in that** it comprises the following operations:

(a) introducing a passivation agent into said carbon dioxide stream fed to the reactor, preferably before the compression phase to the pressure of the reactor, in such an amount that its equivalent oxygen content is at least 0.1%, preferably from 0.15 to 0.30% in moles, with respect to the moles of carbon dioxide,

(b) regulating the pressure and temperature conditions of the reactor so that a gaseous phase is formed at least in the upper zone of the same, containing at least a part of the passivation agent in liquid-vapour equilibrium with said liquid mixture;

(c) positioning a gas-liquid separator (D1) between said reactor (R) and said stripper (S), operating at the same pressure as the reactor, to form a second gaseous mixture containing at least a part of the passivation agent, which is fed into the lower section of said stripper (S), the remaining liquid mixture being fed into the upper section of the same stripper;

(d) establishing the operative conditions of said decomposition-stripping step in the stripper (S) so that it is carried out at a pressure of at least 0.1 MPa, preferably from 0.2 to 2.0 MPa, lower than that of said reaction step.”

II. The appellant had filed an opposition requesting revocation of the patent-in-suit in its entirety on the grounds of lack of inventive step (Article 100(a) EPC), based *inter alia* on documents:

(1) WO-A-98/17635,
(2) EP-A-0 727 414,
(8) CN 1463964 and
(8a) English translation of document(8).

III. According to the opposition division, the subject-matter of the claims of the patent as granted involved an inventive step starting either from document (1) or (2) as the closest prior art to the invention, the problem to be solved being the provision of a process and an apparatus for the preparation of urea allowing a more effective passivation of the stripper and the high-pressure synthesis reactor. The processes of claims 1 and 14 and the apparatus of claim 12 were not obvious in the light of document (1), also in combination with document (8).

IV. The appellant contested the conclusions of the opposition division with respect to the issue of inventive step and submitted that the claimed subject-matter of the patent as granted lacked an inventive step starting from document (1) or (2) as the closest prior art. The appellant further filed documents

(11) CN 1840523 and
(11a) English translation of document (11)

and submitted that the claimed subject-matter also lacked an inventive step starting from document (11) as the closest prior art.

V. The appellant (opponent) requests that the decision under appeal be set aside and the patent be revoked.

The respondent (patent proprietor) requests that the appeal be dismissed (main request), or subsidiarily, that the patent be maintained on the basis of one of auxiliary requests 1 to 8 filed with a letter dated 14 August 2018.

VI. At the end of the oral proceedings held on 9 February 2023 the decision of the Board was announced.

Reasons for the Decision

1. *Late filed document (11) - admissibility into the proceedings*

1.1 *Revised Rules of Procedure of the Boards of Appeal (RPBA 2020) - transitional provisions*

The present proceedings are governed by the revised version of the Rules of Procedure which came into force on 1 January 2020 (Articles 24 and 25(1) RPBA 2020), except for Article 12(4) to (6) RPBA 2020 instead of which Article 12(4) RPBA 2007 remains applicable (Article 25(2) RPBA 2020).

Article 12(4) RPBA 2007 gives the Board the power to hold inadmissible facts and evidence that could have been presented in the first instance proceedings.

1.2 Document (11) and its English translation (11a) were filed by the appellant with the statement setting out the grounds of appeal. The respondent requested not to admit document (11) at this stage, since appeal proceedings are primarily restricted to review the decision under appeal. Since document (11) was not part of the appealed decision it should not be considered by the board of appeal. Furthermore document (11) was not *prima facie* more relevant to documents (1) or (2) filed with the notice of opposition.

1.3 The appellant argued that document (11) is the closest state of the art to the invention and should be admitted in the appeal proceedings.

However, starting from the newly filed document (11) as the closest state of the art to the invention constitutes an important amendment to the case in the appeal proceedings, and would be contrary to the main aim of appeal proceedings which is to review the decisions of the first instance.

1.4 The board thus decides not to admit document (11) and and its translation into English (11a) into the appeal proceedings pursuant to Article 12(4) RPBA 2007.

Main request (patent as granted)

2. *Inventive step*

2.1 *Closest* prior art

It is agreed that, among the documents which are in the appeal/opposition proceedings, document (1) represents the closest prior art to the invention.Document (2) has similar teaching and is not closer to the invention than document (1).

Document (1) discloses a process for the preparation of urea from ammonia and carbon dioxide wherein ammonia and fresh carbon dioxide are fed to the bottom of a reactor 2 operating at a pressure of 226.10^2 KPa to form at the top of the reactor a liquid mixture containing urea, carbamate, ammonia and water produced in the carbamate dehydration to urea and a gas phase comprising CO_2, water and the inert gases which are contained in CO_2 reactant, i.e. ammonia saturated passivating air (see page 4, lines 15 to 18; figure 1).

The liquid solution is discharged from the upper part of the reactor, and sent to the top of a stripper 7 operating at a pressure of 130.10^2 kPa. The gases accumulated on the top of the reactor are sent to the bottom of the stripper so that passivating air contained in the gases carry out their anticorrosion action also within the stripper 7 (page 4, lines 19 to 24). The liquid solution is heated to 207°C and carbamate is decomposed (page 4, line 24-26).

The overhead gas of the stripper is mixed with preheated carbamate and fed to the pipes of condenser 10A (page 5, lines 2 to 6). The solution and the vapor which exit from the bottom of condenser 10B is sent to another condenser 11, enter separator 18 and is recycled to the reactor 2 (page 5, lines 18 to 20).

2.2 *Technical problem*

The respondent defined the technical problem to be solved as providing a more effective means of protecting the stripper from corrosion, while maintaining the efficiency of the urea synthesis process.

2.3 *Solution*

The proposed solution to the problem of providing a more effective means of protecting the stripper from corrosion is to implement a stand-alone separator between the reactor and the stripper, as reflected in the subject-matter of independent claims 1, 12 and 14.

2.4 *Success*

2.4.1 According to the respondent, the implementation of a stand-alone separator permits to better control and improve the gas-liquid separation and thus to increase the amount of passivation agent contained in the gas phase. The optimisation of the liquid/gas separation allows for better protection of the stripper due to a larger content of passivation agent present in the gas phase.

2.4.2 The appellant contests that a stand-alone separator provides better separation, all the more because the claims does not define the conditions at which this separator is operated. According to the appellant, the liquid-gas separation in a stand-alone separator does not provide any improvement with respect to a liquid-gas separation which takes place at the top of a reactor, as disclosed in document (1).

2.4.3 However, as argued by the respondent a stand-alone separator can provide a better liquid/gas separation than the upper part of a reactor which might not be designed for that purpose. As pointed out at point 20 of the reasons of the contested decision, the liquid reaction mixture and the gaseous phase are extracted from the reactor at different levels, without necessarily reaching the gas-liquid equilibrium.

Document (1) does not mention that a liquid-vapour equilibrium is achieved when the liquid and the gas are removed from the reactor. The liquid phase may still contain dissolved gas comprising the passivation agent when it is removed from the reactor. The passivation agent that remains in the liquid phase is not made available for corrosion protection. The less passivation agent remaining in the liquid phase, the better the corrosion protection.

With regard to the lack of precise technical characteristics and operating conditions of the separator in the claims, the board notes that it is within the general knowledge of the person skilled in the art to find the appropriate configuration of a liquid/gas separator depending of the particular circumstances. Furthermore, the proposed solution is not related to a specific configuration of the liquid/gas separator, but to the location of the separator between the reactor and the stripper.

2.4.4 Therefore, the Board is convinced that the installation of a stand-alone separator between the reactor and the stripper will improve the effectiveness of the stripper's protection against corrosion by increasing the amount of passivation agent present in the gas phase.

2.5 *Obviousness*

It remains to be decided whether the use of a stand-alone liquid/gas separator as a solution to the problem of providing a more effective means of protecting the stripper from corrosion is obvious in view of the cited prior art.

According to the appellant, the proposed solution is obvious, since it is common knowledge that the liquid/gas separation can be performed at the top of the reactor or in a stand-alone separator. This is illustrated in document (8). Accordingly the claimed subject-matter lacks an inventive step over the combination of document (1) with document (8).

The board concurs with the appellant that it is known that the liquid/gas separation of the reaction product of a continuous process can be performed either at the top of the reactor or else in a stand-alone separator. However, the technical problem underlying the contested patent is not the provision of an alternative way for the liquid/gas separation or an alternative way of sending the reactor overhead gas from the top of the reactor to the bottom of the stripper, but to provide a more effective means of protecting the stripper.

Document (8) relates to a transformation on small or medium urea plants in order to save energy and increase production -see document (8a) paragraphs [0001] to [0003]. The plant transformation taught by document (8) consists in placing upstream of the existing urea reactor 4 an assembly comprising a first reactor 1, a separator 2 and a stripper 4 (see paragraph [0004] and figure). The reaction product of a first reactor 1 is send to a stand-alone separator 2. The oxygen-rich phase is separated out in this separator and sent to the bottom of a stripper 3 to achieve passivation corrosion prevention -see paragraph [0011].

However, document (8) does not teach that the combination of a reactor with a stand-alone separator has been chosen over a reactor in which gases are extracted at its top for the purpose of improving gas separation, let alone for the purpose of improving corrosion protection of the stripper. Like document (1), document (8) does not recognise that some gas may remain dissolved in the extracted liquid, making the protection of the stripper less effective. Moreover, the oxygen-rich gas phase is removed from the top of the second urea reactor 4. This oxygen-rich gas is also fed to the bottom of the original decomposition system for corrosion prevention -see page 11, lines 11 to 15.

Consequently, starting from the urea plant of the closest prior art document (1), the person skilled in the art would not have turned to document (8) to provide a more effective means of protecting the stripper from corrosion. There is no hint in document (8) that the protection from corrosion of the stripper of the urea plant of document (1) would be more effective if the liquid/gas separation of the reaction products takes place in a stand alone separator.

Moreover, as the contested decision points out, even if the person skilled in the art had turned to this document and applied the plant transformation taught by document (8) consisting of placing upstream of the urea reactor 2 of the urea plant of document (1) an assembly consisting of a first reactor, a stand-alone separator and a stripper, he would not have arrived at the subject-matter of the claims of the patent as granted.

2.6 The Board therefore comes to the conclusion that the subject-matter of independent claims 1, 12 and 14 of the patent as granted, and by the same token that of dependent claims 2 to 11, 13 and 15, is not rendered obvious in the light of the cited prior art.

Order

For these reasons it is decided that:

The appeal is dismissed.

The Registrar: The Chairman:

Beschwerdekammern des Europäischen Patentamts
Boards of Appeal of the European Patent Office
Chambres de recours de l'Office européen des brevets

C. Rodríguez Rodríguez P. Gryczka

Decision electronically authenticated