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Datasheet for the decision of 23 September 2015

Case Number:	T 0190/13 - 3.3.06
Application Number:	07862141.4
Publication Number:	2104561
IPC:	B01J21/06, B01J23/66, B01J29/89, C07C67/055, C07D301/12
Language of the proceedings:	EN

Title of invention:
Inorganic oxide extrudates

Applicant:
LyondellBasell Acetyls, LLC

Headword:
Vinyl acetate preparation process / LYONDELL

Relevant legal provisions:
EPC Art. 52(1), 54(1), 54(2)

Keyword:
Novelty (no) -
preparation process not distinguishable from process of the prior art

Decisions cited:

Catchword:

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Case Number: T 0190/13 - 3.3.06

DECISION
of Technical Board of Appeal 3.3.06
of 23 September 2015

Appellant: LyondellBasell Acetyls, LLC
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Decision under appeal: **Decision of the Examining Division of the European Patent Office posted on 18 July 2012 refusing European patent application No. 07862141.4 pursuant to Article 97(2) EPC.**

Composition of the Board:

Chairman B. Czech
Members: L. Li Voti
C. Heath

Summary of Facts and Submissions

I. This appeal lies from the decision of the Examining Division to refuse European patent application no. 07 862 141.4.

II. Claims 1, 4 and 7 according to the sole claim request pending before the Examining Division (filed by letter of 20 September 2011) read as follows:

"1. An *extrudate comprising titania and a comb-branched polymer, wherein the titania constitutes at least 10 weight percent, and the comb-branched polymer comprises recurring units of ethylenic carboxylic monomer and a polyether macromonomer comprising an oxyethylene and oxypropylene copolymer and constitutes at least 1 weight percent of the extrudate.*"

"*4. A calcined extrudate of claim 1.*"

"*7. A vinyl acetate preparation process comprising reacting ethylene, acetic acid and oxygen in the presence of a palladium-gold catalyst prepared with a calcined extrudate of claim 4.*"

III. In its decision, the Examining Division found *inter alia* that the the subject-matter of this claim 7 lacked novelty over document D5 (US 6 821 922 B1) for the following reasons:

"...*D5 does not disclose the presence of any comb branched polymer in the raw extrudate.*
However, claim 7 refers to the calcined extrudate. As is stated in the present application, the polymer is removed during calcination (sentence bridging pages 7 and 8 of the application).

Hence, it has to be decided whether the presence of a comb-branched polymer inevitably results in distinguishable properties of the resulting catalyst, which no longer contains said polymer.
According to the applicant, the resulting catalyst did not have any distinguishable physicochemical properties, but exhibited a different catalytic behavior in the catalytic process...
it is acknowledged that the specific catalysts D and comparative G differ in their catalytic behaviour. However, considering the available experimental data as a whole, it is not possible to unambiguously attribute the difference in catalytic behaviour of catalyst D, as compared to comparative catalyst G, to the fact that the polymer used has a different, namely comb-branched, structure. On the contrary, it is clear from the examples as a whole that other parameters ... viz. the choice between polymer A, B or C (see table 2; all within the definition of claim 7) have a much greater effect on catalyst performance, than the structure of the polymer...
Hence, the available catalytic data does not establish that the structure of the polymer used, namely the use of a comb-branched polymer comprising the indicated monomer units, translates into any distinguishable properties of the resulting calcined extrudate and hence final catalyst..."

IV. With its statement of grounds of appeal dated 20 November 2012, the Appellant filed an amended set of claims, claim 1 thereof reading as follows:

"7. *A vinyl acetate preparation process comprising reacting ethylene, acetic acid and oxygen in the presence of a palladium-gold catalyst prepared with a calcined extrudate comprising titania and a comb* *branched polymer; said comb branched polymer comprising recurring units of ethylenic carboxylic monomer and a polyether macromonomer comprising an oxyethylene and oxypropylene copolymer; wherein the titania constitutes at least 10 weight percent of extrudate, and the comb branched polymer constitutes at least 1 weight percent of extrudate.*"

As an auxiliary measure, the Appellant requested oral proceedings.

It submitted *inter alia* that the subject-matter of claim 1 at issue was novel over document D5, arguing as follows:

"*According to the applicant, the resulting catalyst did not have any distinguishable physicochemical properties, but exhibited a different catalytic behaviour in the catalytic process. This could be due to the fact that the comb branched polymer is not inert but has some functionalities that can react with palladium and gold.*
In support of this argument, the applicant refers to examples 18 and 19 of the present application (catalyst D and comparative catalyst G, see table 4). These examples showed a difference in catalytic behaviour, which was evidence of a difference of the catalyst per se.
...
... In particular the Oxygen selectivity is defined as the amount of oxygen consumed in making vinyl acetate divided by the total oxygen consumed (see page 17, lines 17-18). Thus the closer is this variable to 1 the most efficient is the process with less by products...
...
[From the data reported] *it is clear that there are*

*small variations of oxygen selectivity by using the catalyst according to the invention. On the contrary there is clear difference between the oxygen selectivity of catalyst G** (i.e. the comparative catalyst G) *and catalyst D at 135°C.*
Thus the use of a comb-branched polymer comprising the indicated monomer units, translates into a distinguishable property of the resulting calcined extrudate and hence final catalyst, i.e. the oxygen selectivity. Consequently claim 7 [sic] *is novel over D5."*

V. The Appellant was summoned to oral proceedings. In a communication dated 23 June 2015 issued in preparation for the oral proceedings (points 3.1 to 3.13), the Board indicated the reasons for which it considered that the subject-matter of claim 1 at issue appeared to lack novelty over D5.

VI. The Appellant replied to the Board's communication by letter dated 21 August 2015. It merely stated:
"*This is in replay of the* [sic] *to the communication of the Board of appeal dated 23.06.2015:*
We withdraw the request for oral proceedings and ask a decision of the Board of Appeal on the file as such."

VII. As acknowledged by the Board in its communication, the Appellant requested in writing (as main and sole claim request) that the decision under appeal be set aside and that a patent be granted on the basis of the set of claims 1 to 6 filed with the statement of grounds of appeal dated 20 November 2012.

Reasons for the Decision

Novelty - Claim 1

1. In its communication dated 23 June 2015 (points 3.1 to 3.13) the Board indicated in detail the reasons for which the claimed process appeared to lack novelty over D5. The Appellant did not contest the accuracy and correctness of the Board's considerations and chose not to submit any further arguments.

   The Board, after having reconsidered all relevant aspects of the case, has no reason to deviate from its preliminary opinion, as expressed in its communication, and, based thereupon, issues the following decision.

2. Claim 1 at issue

2.1 Process claim 1 at issue corresponds to process claim 7 found to lack novelty by the Examining Division (see points II and IV, *supra*).

2.2 Claim 1 requires that the palladium-gold catalyst used is "*prepared with a calcined extrudate comprising titania and a comb branched polymer*" of a specific type, "*wherein the titania constitutes at least 10 weight percent of extrudate, and the comb branched polymer constitutes at least 1 weight percent of extrudate*".

The indicated weight percentages undisputedly relate to the raw extrudate before calcination.

2.3 The catalyst to be used used in the process according to claim 1 is thus defined *inter alia* in product-by-process terms, rather than by further structural or compositional features, or in terms of measurable properties.

It is also not in dispute that the features relating to the preparation of the catalyst, including the definition of the raw extrudate composition, do not define steps of the claimed process.

3. Document D5

3.1 D5 discloses a vinyl acetate preparation process (see example 9) comprising the step of reacting ethylene, acetic acid and oxygen in the presence of a palladium-gold catalyst.

More particularly, the catalyst used in the process disclosed in example 9 is that of example 8, which is prepared in the same manner as that of example 7, "on the basis of the molding according to example 1". According to said example 1, an extrudate in the form of a tablet, containing initially more than 10% by weight of titania and more than 1% by weight of a polyethylene glycol, is calcined for 6 hours at 750°C. The support so obtained is impregnated with solutions of palladium and gold.

3.2 Therefore, the process disclosed in document D5 undisputedly differs from that of claim 1 at issue, if at all, only insofar as the catalyst used is obtained using an extrudate containing, before calcination, 8.6 % by weight of a polyethylene glycol instead of at least 1 % by weight of a "*comb branched polymer*" as prescribed by claim 1 at issue.

3.2.1 Said "*comb branched polymer*", like the polyethylene glycol used in example 1 of D5, is totally combusted during calcination of the extrudate and is not present as such in the final catalyst. This was not disputed and appears to be conceded in the statement of grounds of appeal (page 2, lines 4 and 5) where it is stated that "*the resulting catalyst did not have any distinguishable physicochemical properties*" (compared to the catalyst used in D5).

3.3 Effects attributable according to the Appellant to the use of comb branched polymers

3.3.1 The Appellant submitted, however, (page 2, lines 5 to 7 of the statement of grounds) that the catalyst of the invention "*exhibited a different catalytic behaviour in the catalytic process. This could be due to the fact that the comb branched polymer is not inert but has some functionalities that can react with palladium and gold*".

This different behaviour would be shown, for example, by examples 18 and 19 (table 4 of the application) comparing a catalyst D according to the invention with a "comparative catalyst G" (hereinafter catalyst G*), the latter being prepared from an extrudate containing, before calcination, a polyethylene oxide instead of a comb branched polymer.

Therefore, in the Appellant's view, the catalyst used in the process of claim 1 at issue would necessarily be different from that disclosed in document D5. Hence, the Examining Division erred in concluding that, in the light of the overall experimental evidence contained in the application, it was not possible to attribute the difference in catalytic behaviour to the presence of the comb branched polymer in the initial extrudate before calcination.

3.3.2 For the Board, however, the Appellant's statement "*This could be due to the fact that the comb branched polymer is not inert but has some functionalities that can react with palladium and gold*" is not convincing since according to description of the application, palladium and gold are deposited onto the extrudate after calcination, i.e. after the combustion of the branched polymer. Since the comb branched polymer is no longer present as such in the calcined extrudate, it cannot interact with the deposited metals.

As regards to comparative data invoked by the Appellant, the Board observes the following:

i) As apparent from table 4 of the application, catalyst D indeed performs better in the preparation of vinyl acetate than comparative catalyst G*. However, it must be considered that catalyst D (see table 1) is prepared using a calcined extrudate having voidage (67.9%) and bulk density (1.03 g/ml) values similar to those of the calcined extrudate used in the preparation of catalyst G* (67.9% voidage; 1.1 g/ml bulk density) **but a greater pore volume** (30.1 mL/g vs. 26.8 mL/g).

It is, however, far from clear whether this greater pore volume is indeed due to the use of comb-branched polymer, since the calcined extrudates used in the preparation of the other catalysts according to the invention, although all containing a comb polymer before calcination, have very different physical properties, as indicated hereinbelow.

ii) Catalysts E and F according to the invention (examples 9-11 of the application,; page 17, first paragraph) are prepared in a similar way as catalyst D (i.e. using the procedure of Example 8) but with the calcined extrudates of examples 5 and 6, which contained, before calcination, the same amount of comb-branched polymer as the extrudate used in the preparation of catalyst D. According to table 1 the calcined extrudates of examples 5 and 6 have, however, lower bulk densities (0.88 and 0.96 g/ml) and greater voidage values (71.4% and 70.0%) than the calcined extrudate used for preparing catalyst D (bulk density 1.03 g/ml and voidage 67.9%). Moreover, their **pore volume can be either greater or much lower** (compare E=32.5 mL/g, F=19.9 mL/g, with D=30.1 mL/g). The pore volume of the calcined extrudate used in preparing catalyst F (according to the invention) is thus even lower than that of the extrudate used for catalyst G* (26.8 mL/g).

3.3.3 These differences in the physical parameters of the calcined extrudates appear to have a significant effect on the performance of the catalysts prepared therefrom. In fact, the performances of the catalysts D, E and F according to the invention as tested in examples 12-14 differ significantly (see table 2). In particular, the oxygen selectivity, which the Appellant appears to consider as the most important performance parameter (see bottom of page 2 of the statement of grounds and point IV above), is lower for catalysts E and F (76.6% and 74.6%) than for catalyst D (77.4%). Therefore, it cannot in any way be assumed that the performance of catalysts E and F according to the invention would necessarily be better than that of catalyst G*, which shows a poorer performance than catalyst D in the catalyst testing reported in comparative example 19 (see table 4), the latter differing, moreover, from that reported in examples 12-14 of the application in terms of the higher sand bath temperature set (135°C vs. 130°C; see page 18, the two paragraphs below table 3).

Moreover, in view of the fact that the pore volume value of the calcined extrudate used in the preparation of catalyst G* (26.8 mL/g) lies between those of other extrudates used in preparing catalysts according to the invention (see point 3.3.2.ii above), the Board does consider it to be established that using a comb-branched polymer as defined in claim 1 as a component of the raw extrudate will not necessarily lead to a catalyst with characteristics different from those of a catalyst obtained using other polymers, also completely combusted during calcination, as component of the raw extrudate.

4. Hence, it cannot be gathered from the application that the better performance shown in table 4 can actually be attributed to the use of a comb-branched polymer as defined in claim 1 as a component of a raw extrudate from which the "*calcined extrudate*" carrier of the catalyst used in the process of claim 1 is prepared, as alleged by the Appellant, rather than to the different physical properties of the calcined extrudates used, i.e. irrespective of the type of the polymer component of the raw extrudate, as found in the decision under appeal (see point III above).

Therefore, in the Board's judgement, the Appellant did not convincingly show that the catalysts to be used in the claimed vinyl acetate preparation process can necessarily be distinguished from those used in the process disclosed in document D5. In other words, the Appellant did not discharge the burden of proof resting with it as regards the allegation that it was a feature of the preparation of the catalyst used in the claimed vinyl acetate preparation process (use of a specific comb-branched polymer component) that necessarily made the catalysts so obtained, and hence also the vinyl acetate preparation process involving them, distinguishable from the catalyst and vinyl acetate preparation process according to D5.

5. The Board concludes that the claimed subject-matter is not novel over the disclosure of document D5 (Articles 52(1) and 54(1)(2) EPC). Hence, the Appellant's sole request is not allowable.

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

D. Magliano B. Czech

Decision electronically authenticated