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Datasheet for the decision of 14 November 2012

Case Number: T 1152/09 - 3.3.03

Application Number: 99953103.1

Publication Number: 1141029

IPC: C08B 11/20, C08B 17/06

Language of the proceedings: EN

Title of invention:
Process and apparatus for making cellulose ethers

Patent Proprietor:
Dow Global Technologies LLC

Opponent:
SE Tylose GmbH & Co.KG

Headword:
-

Relevant legal provisions:
EPC Art. 56

Keyword:
"Inventive step - yes"

Decisions cited:
T 1126/05

Catchword:
-

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Case Number: T 1152/09 - 3.3.03

D E C I S I O N
of the Technical Board of Appeal 3.3.03
of 14 November 2012

Appellant I: (Patent Proprietor)
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Decision under appeal:
Interlocutory decision of the Opposition Division of the European Patent Office posted 31 March 2009 concerning maintenance of European patent No. 1141029 in amended form.

Composition of the Board:

Chairman: B. ter Laan
Members: M. C. Gordon
C.-P. Brandt

Summary of Facts and Submissions

I. The appeals lie from the interlocutory decision of the opposition division announced on 10 March 2009 and posted on 31 March 2009 according to which it was held that European patent number 1 141 029 (granted on European patent application number 99953103.1, derived from international application number PCT/US99/23475, published under the number WO-A-00/32637) could be maintained in amended form on the basis of the first auxiliary request (claims 1-9) filed during the oral proceedings before the opposition division.

II. The patent was granted with a set of 10 claims, whereby claims 1 read as follows:

"A process for making a cellulose ether, comprising:

a) alkalizing a cellulose pulp;

b) etherifying the alkalized cellulose pulp to form a cellulose ether;

c) washing the cellulose ether;

d) drying the cellulose ether to a reduced moisture content;

e) milling the cellulose ether to a particulate form;

f) adjusting the temperature of the particulate cellulose ether to 50°C to 130°C;

g) continuously tumbling the particulate cellulose ether in a reactor, the reactor being rotated axially or end over end in an oven or a controlled-temperature chamber or room while simultaneously contacting the cellulose ether with an acid to partially depolymerise the cellulose ether such that a two percent aqueous

solution of it has a viscosity of 200 cP or less at 20°C; and
h) partially or substantially neutralizing the acid by contacting it with a basic compound."

Claims 2-7 were dependent on claim 1. Claim 8 was directed to an apparatus for making cellulose ethers, claims 9 and 10 being dependent thereon.

III. Notices of opposition against the grant of the patent were filed on 15 January 2004 by Opponent O1 and on 21 February 2004 by Opponent O2.

Both opponents invoked the grounds of opposition pursuant to Art. 100(a) EPC. Specifically, it was submitted that the subject-matter of independent claims 1 and 8 of the patent in suit was neither novel (Art. 54 EPC) nor founded on an inventive step (Art. 56 EPC).

The following documents, *inter alia*, were cited in support of the oppositions:

D1: US-A-3 391 135
D3': EP-B-210 917
D4: DE-OS-27 26 780

IV. By a decision announced orally on 5 July 2005 and issued in writing on 28 July 2005 the opposition division revoked the patent on the ground of lack of novelty in respect of the disclosure of D3'.

The patent proprietor filed an appeal against this decision.

Opponent O1 withdrew its opposition by letter of 21 June 2007.

In decision T 1126/05, dated 7 November 2007 the Board of Appeal 3.3.03 held that the subject-matter of the claims of the patent as granted was novel and remitted the case to the opposition division for further prosecution.

V. Following remittal to the opposition division, in the decision of 10 March 2009, the subject of the present appeal, it was held that the patent could be maintained on the basis of the auxiliary request, the wording of which is not relevant for the present decision.

The opposition division held that the main request, i.e. the claims of the patent as granted did not meet the requirements of Art. 56 EPC in view of the teachings of D3 (EP-**A**-210 917). The experimental evidence did not show that the use of an oven or a controlled temperature chamber or room resulted in any technical effect and consequently was an obvious solution to the problem of providing a further process for providing low molecular weight cellulose ethers with a low amount of carbonaceous tars.

The claims of the auxiliary request were however held to meet the requirements of Art. 56 EPC.

VI. A notice of appeal against the decision was filed by the patent proprietor on 25 May 2009 with simultaneous payment of the prescribed fee. The statement of grounds of appeal was filed on 17 July 2009. The patent

proprietor made further written submissions with letters dated 2 December 2009, 14 April 2010 and 3 October 2012.

A notice of appeal against the decision of the opposition division was also filed by the opponent on 2 June 2009 with simultaneous payment of the prescribed fee. The statement of grounds of appeal was filed on 10 August 2009. The opponent made further written submissions with letters dated 22 December 2009 and 9 October 2012.

VII. On 26 July 2012 the Board issued a summons to attend oral proceedings. In a communication dated 2 August 2012 the Board set out its preliminary assessment of the case.

VIII. Oral proceedings were held before the Board on 14 November 2012.

IX. The arguments of the patent proprietor can be summarised as follows:

The evidence of the examples of the patent and those submitted during the examination and opposition proceedings demonstrated that carrying out the reaction by tumbling in an oven resulted in higher quality product, in particular a lower occurrence of tar particulates than when carrying out the procedure according to the teaching of D3', i.e. in a water bath.

The claim required that contacting with acid and tumbling be simultaneous but did not specify or restrict the order in which these two steps were

initiated. Thus it was in accordance with the claim to introduce the acid and then initiate tumbling, or to commence tumbling and then introduce acid. The apparatus described in the patent was designed in particular to permit this second alternative and the examples had to be read and interpreted in the light of the description of this apparatus.

Furthermore, in the examples of the patent the heating was carried out in the sequence specified in claim 1, in particular steps (f) and (g) thereof, i.e. heating was performed prior to the commencement of tumbling. The required sequence was made explicit in the evidence submitted during the course of the examination and opposition proceedings.

X. The arguments of the opponent can be summarised as follows:

None of the examples of the patent was according to the claims since the acid was introduced before tumbling was initiated whereas the claims required that these steps be simultaneous. Furthermore in the examples of the patent the adjustment of temperature was carried out only after introduction of the acid, i.e. the sequence of steps (f) and (g) of the claim was not respected.

The other examples submitted during the course of the prosecution of the case, namely:

By the patent proprietor:

- The examples and comparative examples A and B submitted with a letter of 21 February 2002 (during the examination proceedings)
- Comparative example C submitted with letter of 1 October 2004 (at the start of the opposition proceedings)
- Examples E1-E5 and D1-D8 submitted with letter dated 22 November 2005 (together with the Statement of Grounds of Appeal in the first appeal)

By other parties:

- The examples of former opponent O1 submitted with letter of 2 June 2006 (during the first appeal proceedings) and resubmitted by the patent proprietor with a letter of 3 October 2007

all had one or more deficiencies. In particular there were multiple modifications between the examples that were stated to demonstrate the invention and the comparative examples meaning that no set of examples was able to show an effect arising from the distinguishing feature over D3' which was the closest prior art.

With regard to the examples of 22 November 2005, the sequence of heating and charging the reactor was not in accordance with the claims. Different starting materials had been used for the "inventive" and the "comparative" examples, whereby the materials differed in terms of the water content and the content of coloured particles. Even within the comparative examples two different materials - differing in water content - had been employed. The extent of charging

(filling) of the reactor was not reported. Further the times employed for the reaction and the amount of hydrogen chloride introduced differed between the "inventive" and "comparative" examples.

Thus the objective problem was to provide a further process based on the teaching of D3'. The use of an oven as an alternative to a water bath was obvious in particular in view of the teachings of D1 and D4. D1 taught that in the depolymerisation of cellulose with HCl it was important to maintain HCl in the gaseous state so that it did not condense. This could be achieved most simply by placing the reactor in an oven, chamber or room with controlled temperature. A teaching to operate in such manner was provided by D4, example 2 which disclosed a flask maintained in an oven at 50°C.

XI. The appellant (patent proprietor) requested that the decision under appeal be set aside and that the patent be maintained as granted (main request) or, alternatively, that the appeal of the opponent be dismissed and the decision of the opposition division be confirmed.

XII. The appellant (opponent) requested that the decision under appeal be set aside and that the European patent No. 1 141 029 be revoked.

Reasons for the Decision

1. The appeal is admissible.

2. As the previous appeal T 1126/05 found the subject-matter of the patent in suit according to the main request to be novel, the only matter to be decided in the present decision is inventive step.

3. The closest prior art.

3.1 The patent in suit is directed to a process and apparatus for making cellulose ethers. The aim is to obtain low molecular weight cellulose ethers with a substantially reduced incidence of tar formation (paragraph [0009]).

Tar formation occurs according to the patent in suit due to excessive degradation of the cellulose ether. The degradation has various causes, e.g. non-uniform moisture distribution within the cellulose ether, static cling between the cellulose ether and the internal surfaces of the depolymerisation reactor, non-uniform application or adsorption of the strong acid, catalysation of the depolymerisation reaction by contact with catalytically active surfaces or substances (paragraph [0004] of the patent in suit). Excessive degradation due to non-uniform moisture distribution arises when water absorbed in the cellulose ether vaporises and condenses on colder spots on the internal surfaces of the reactor. Dry cellulose powder absorbs the water, and sticks to the internal surfaces. The acid, attracted to the high moisture, leads to regions of increased acid content within which

localised accelerated depolymerisation occurs causing tar particulates to form (paragraph [0005]). Another cause of excessive degradation is non-uniform application of the acid, also leading to localised increased concentrations (paragraph [0007]).

3.2 By common consent the closest prior art is D3', which document is directed to a method for the preparation of a water-soluble cellulose ether having a decreased average degree of polymerisation. According to claim 1 of D3' the method comprises:

(a) contacting a starting cellulose ether in a powdery form with an aqueous solution of hydrogen chloride in such an amount that the amount of hydrogen chloride is in the range of 0.1 to 1.0 % by weight based on the starting cellulose ether and the amount of water is in the range from 3 to 8 % by weight based on the overall amount of the starting cellulose ether and the aqueous solution of hydrogen chloride at a temperature of 40 to 85°C; and

(b) removing the hydrogen chloride from the mixture of the powdery cellulose ether and the aqueous solution of hydrogen chloride.

According to the examples of D3' the treatment is carried out by spraying the aqueous acid onto the cellulose in a running Henschel mixer, and placing a portion of the cellulose in a glass vessel which is then tumbled in a water bath at 75°C until the viscosity of the cellulose ether drops.

As held in T 1126/05, D3' provides no details of the form of the water bath, e.g. whether this is open to

the air or in some way enclosed. Further there is no disclosure of the extent to which the flask is immersed in the water bath or the angle of immersion. Consequently the process of D3' does not have features corresponding to feature (g) of operative claim 1 (T 1126/05, section 2.2 and 2.3 of the reasons).

4. The problem to be solved.

4.1 According to the wording of the examples of the patent in suit (paragraph [0040]) the reactor was charged with methylcellulose (lines 16-20) and acid was added (lines 21-24). The reactor "was placed in an oven set at 90°C" and tumbled (lines 25-26).

4.1.1 The wording of the examples is to an extent ambiguous since the sequence of steps and the use of the wording "was placed" could on one interpretation indicate that the reactor was first charged with the ether and acid and then placed in the oven and tumbled. Equally the phrase "was placed" could be interpreted as indicating the situation or location of the reactor, i.e. that it was located (already) in the oven.

According to the discussion of the reactor at column 7 in paragraph [0031], lines 14-20 of the patent in suit it emerges that the reactor is in fact located in an oven or is equipped with a heating jacket. Accordingly the latter interpretation of "was placed" is consistent with the discussion of the reactor in the patent in suit i.e. indicating the situation of the reactor, but not the action of introducing the reactor to an oven.

4.1.2 Regarding the mode of addition of the acid the grammar and wording of the example is also is open to interpretation since the wording could be understood either as indicating that the acid was introduced in a first step and subsequently tumbling initiated or that acid was introduced during tumbling.

However according to the discussion in paragraph [0031] of the patent in suit, the reaction apparatus is equipped with a passageway (42) extending through the centre of shaft (30), which passageway is adapted to supply hydrogen chloride to the chamber (18). Thus the reactor is constructed so as to permit the introduction of the acid during tumbling. This leads to the conclusion that it is the second interpretation which is correct, i.e. that in the examples of the patent in suit the acid is introduced during tumbling.

4.1.3 The examples of the patent in the light of the further information in the description of the patent therefore do correspond to the claimed process.

4.2 The evidence of the examples of the patent in suit is that by carrying out the process according to claim 1 a product free of tar particulates was formed.

4.2.1 Evidence submitted during the course of the examination proceedings and opposition proceedings (in particular that of 22 November 2005 and 2 June 2006) provided comparisons with a reaction carried out in a rotary evaporator with the flask containing the cellulose ether immersed to a greater or lesser degree in the water. Such a construction was consistent with the teachings of D3', notwithstanding that, as held in

T 1126/05 D3' was silent on the precise details of the construction of the reaction system.

4.2.2 The above indicated examples demonstrate that when operating according to the teachings derivable from D3' it was not possible to obtain a product free of tar particulates even in the case that the flask was immersed almost completely (only the neck above the water line - data of 22 November 2005).

4.2.3 Although the criticism of the appellant/opponent that in the data of 22 November 2005 multiple factors were varied between the examples intended to represent the subject-matter of the patent in suit and those reflecting the prior art teachings is valid, this has to be balanced against the fact that the examples show, consistently and independently of any other variations, that when operating according to the process of the patent in suit, i.e. locating the reactor in an oven or controlled chamber or room, a product free of tar particulates was obtained. In contrast, and again consistently when operating according to a process according to the teachings of D3', i.e. employing a water bath, it was not possible to obtain a product free of tar particulates, independently of any other variations in the process.

4.2.4 Furthermore the appellant/opponent did not provide any technical arguments, let alone evidence that the differences between the examples and comparative examples, apart from those relating to the apparatus employed for the reaction, could or would have been responsible for the observed different outcomes in respect of tar particulate formation.

4.3 Consequently the Board is satisfied that the evidence advanced demonstrates that the problem with respect to D3' of providing a process resulting in a product free of tar particulates is effectively solved by the claimed measures, i.e. locating the reactor in an oven or controlled temperature chamber or room rather than in a water bath.

5. Obviousness

5.1 D3' itself gives no hint to the claimed solution since the teaching thereof is restricted to the use of a water bath of some kind.

5.2 Other documents also do not provide any teaching towards the claimed solution.

5.2.1 Thus D1 relates, according to claim 1, to a method of manufacturing low molecular weight cellulose derivatives by treating high molecular weight cellulose derivatives, *inter alia* cellulose ethers in powder form, with not more than 5% by weight of an anhydrous hydrogen halide at a temperature of about 30°C to 80°C and removing thereafter the hydrogen halide from the treated mixture. According to Example II of D1, methyl hydroxy propyl cellulose ether is treated with hydrogen chloride in anhydrous methanol under agitation in a glass lined vessel, at 50°C for 3 hours. The temperature is controlled by means of a water bath.

The geometry of the reaction vessel is not disclosed and there is consequently neither an explicit nor an implicit disclosure that tumbling is carried out. On

the contrary the term "agitated" would indicate that tumbling was not carried out. Furthermore no details are provided about the water bath, in particular the extent to which the reaction vessel is immersed therein. Consequently D1 also does not disclose that the reactor is located in an environment which corresponds to "an oven or a controlled temperature chamber or room" i.e. an enclosed space bounded on all sides (see also T 1126/05 reasons 2.1.5).

5.2.2 D4 relates, according to claim 1, to a process for treating a cellulose ether or ester to provide a lower molecular weight compound by contacting the dry powder with a hydrogen halide at 15-80°C followed by treating the product with sulphur dioxide.

In example 1 of D4 hydroxybutyl methyl cellulose and hydrogen chloride are introduced to a flask and the flask rotated at ambient temperature ("Umgebungstemperatur") for 70 hours. Example 2 of D4 relates to a process carried out at elevated temperature, namely 50°C. Thus gaseous hydrogen chloride and methyl cellulose are introduced to the flask, the pressure equalised to atmospheric with nitrogen and the reaction carried out by placing the flask in an oven at 50°C for 72 hours. Following application of a vacuum, the flask is opened in air, the residual acid in the cellulose neutralised and the cellulose ether placed in a flask, evacuated, SO_2 introduced and the flask rotated at room temperature for four hours. Thus in the process of example 2 of D4 gaseous hydrogen chloride is added to the cellulose ether and only subsequently is the temperature adjusted to 50°C. There is however no explicit disclosure in D4

that the flask is rotated during the contacting with hydrogen chloride at 50°C. Consequently D4 does not teach the sequence of steps required by operative claim 1.

5.2.3 Neither D1 nor D4 addresses the problem of formation of tar particulates.

Whilst D1 does address at col. 3, line 24ff the aspect of condensation of hydrogen chloride, it is apparent that the thrust of this teaching diverges from the consideration of condensation in the patent in suit. Thus D1 teaches that it is necessary to maintain conditions such that the hydrogen chloride does not condense, i.e. that it is sufficiently gasified, but to avoid elevated temperatures (above 30-60°C) to prevent formation of a rubbery or sticky product. Consequently, according to D1, the reaction is operated under reduced pressure conditions to maintain the hydrogen chloride in a sufficiently gaseous state (col. 3, lines 24-39). D1 does not address the problem of condensation on "cold spots" of the reactor and does not discuss the risks arising from localised increased concentrations of acid within the portions of cellulose ether as addressed in the patent in suit.

D4 acknowledges that discolouration can occur during the degradation reaction, but rather than taking measures to minimise or avoid this *ab initio*, the remedy is to subject the cellulose ether products to a post treatment with SO_2.

Consequently neither D1 nor D4 identifies the employment of an even temperature over the entirety of

the reaction vessel as a route to solve the respective problems set out in D1 and D4.

5.3 The conclusion is that neither D1 or D4 discloses a process involving tumbling the reactor in an oven or a controlled-temperature chamber or room, for any reason, let alone in order to reduce the formation of tar particulates in the reaction of degradation of cellulose ethers.

Consequently neither D1 or D4 contains any teaching that would lead the skilled person to modify the process of D3' such as to carry out the tumbling as specified in claim 1 of the patent in suit.

5.4 The subject-matter of claim 1 is therefore not obvious.

5.5 As the process of claim 1 is not rendered obvious by the prior art, there is correspondingly no suggestion or teaching to provide an apparatus as defined in operative claim 8 in order to carry out said process. Consequently the subject-matter of claim 8 is also considered not to be obvious.

6. The subject-matter of the main request therefore meets the requirements of Art. 56 EPC.

Order

For these reasons it is decided that:

1. The decision under appeal is set aside.

2. The appeal of the opponent is dismissed.

3. The European patent No. 1 141 029 is maintained as granted.

The Registrar The Chairman

E. Görgmaier B. ter Laan