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D E C I S I O N
of 7 April 2003

Case Number: T 0310/01 - 3.3.5

Application Number: 94402283.9

Publication Number: 0648528

IPC: B01J 2/04

Language of the proceedings: EN

Title of invention:
Porous prilled ammonium nitrate

Patentee:
SASOL CHEMICAL INDUSTRIES (PROPRIETARY) LIMITED

Opponent:
GRANDE-PAROISSE S.A.

Headword:
-

Relevant legal provisions:
EPC Art. 21, 83, 113(2)(3), 52(1), 54, 56

Keyword:
"Main and first auxiliary request: inventive step (no) - obvious modification"
"Second auxiliary request: amendments - allowable, sufficiency of disclosure (yes), inventive step (yes): non-obvious modification"

Decisions cited:
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Catchword:
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Case Number: T 0310/01 - 3.3.5

D E C I S I O N
of the Technical Board of Appeal 3.3.5
of 7 April 2003

Appellant: (Opponent)
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Decision under appeal: **Interlocutory decision of the Opposition Division of the European Patent Office posted 6 February 2001 concerning maintenance of European patent No. 0 648 528 in amended form.**

Composition of the Board:

Chairman: R. K. Spangenberg
Members: A. T. Liu
H. Preglau

Summary of Facts and Submissions

I. An opposition had been filed against European Patent No. 648 528 on the grounds of Articles 100(a), (b) and (c) EPC. The following documents, *inter alia*, were submitted during the opposition proceedings:

D1: AU-A-0 621 994,

D5: Expancel™ product specification (2 pages),

D9: US-A-4 547 234.

II. The present appeal was lodged by the opponent against the interlocutory decision of the Opposition Division to maintain the patent with a set of amended claims 1 to 30.

III. At the oral proceedings which took place on 7 April 2003, the respondent presented four new sets of claims as basis for a main request and auxiliary requests 1 to 3. The set of claims according to the main request and auxiliary request 1 consisted of claims 1 to 30 and claims 1 to 14, respectively. Claim 1 of both these requests read as follows:

> "A crystalline porous prilled ammonium nitrate product which includes hollow polymer microspheres expanded to a size between 2 and 150 micrometer being polymer balloons present in a concentration (mass/mass) of between 0.05 and 0.8%, incorporated in the crystalline structure."

The set of claims according to the auxiliary request 2 consisted of claims 1 to 13, with claim 1 directed to a porous prilled product and claims 2 to 11 dependent thereon and claim 12 directed to a blasting composition and claim 13 dependent thereon. Claim 1 read as follows:

> "A crystalline porous prilled ammonium nitrate product which includes hollow polymer microspheres being polymer balloons which had expanded during the prilling process to a size between 2 and 150 micrometer, incorporated in the crystalline structure."

IV. The appellant's arguments may be summarised as follows:

- The deletion of the word "encapsulated" from the independent claims 1 and the introduction of the expression "incorporated in the crystalline structure" into these claims, do not have a basis in the original documents as filed.

- There was confusion as to the microspheres used in the only example so that it was impossible for the skilled person to reproduce the invention as disclosed.

- Due to the use of the word "including" the subject-matter of claim 1 was anticipated by D1.

- The incorporation of polymer balloons into explosive compositions was known from D9; the subject-matter of claim 1 was therefore rendered obvious by a combination of D1 with D9.

V. The respondent's arguments were briefly as follows:

- Document D9 was late filed and should not be admitted into the proceedings.

- The polymer balloons were a preferred embodiment of "encapsulated microspheres" which was a synonym for "hollow microspheres".

- The skilled person would read the feature "incorporated in the crystalline structure" as meaning "incorporated in the ammonium nitrate having a crystalline structure".

- Polymer balloons were state of the art. The skilled person could therefore perform the invention.

- D1 did not disclose or suggest polymer balloons being present in a concentration between 0.05 and 0.8% in an ammonium nitrate product.

- Neither did D1 disclose polymer balloons which had expanded during the prilling process. The polymers used in D1 were in an expanded state before the prilling process.

- There was no hint in D1 to incorporate polymer balloons during the prilling process.

- The polymer balloons according to D9 would not be expandable during the prilling process.

VI. At the end of the oral proceedings, the requests were as follows:

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

The respondent (patentee) requested that the decision be set aside and that the patent be maintained on the basis of the main request or one of the auxiliary requests 1 to 3 as filed during the oral proceedings.

Reasons for the Decision

1. Admissibility of document D9

The respondent has made the request that document D9, which was filed by the appellant just one day prior to the oral proceedings before the opposition division, be considered late filed within the meaning of Article 114(2) EPC.

The Board notes that D9 was introduced into the proceedings in reply to the preliminary view given by the opposition division and the subsequent submission by the respondent of a new set of claims (see communication dated 13 June 2000, item 3; respondent's submissions of 15 December 2000). The document was to clarify the term "encapsulated" which was in the independent claim 1 as originally filed but deleted from these amended claims. The Board therefore holds that the filing of D9 is a legitimate reply by the appellant who wanted to present evidence in order to

support his objection that the deletion of the term "encapsulated" from the claims was an infringement of Article 123(2) EPC (see facsimile transmission of the appellant's letter of 16 January 2001).

Furthermore, the respondent not only has had time to study the content of D9 but has even made reference to that document in his submissions at the oral proceedings of 7 April 2003 (see points 3.1, 4 and 6.5 below). The Board therefore does not see any reason to refuse D9 as late filed and not to take it into consideration for the present decision.

Main and first auxiliary requests

2. *Inventive step*

2.1 Claim 1, which has the same wording for both requests, is essentially directed to a prilled ammonium nitrate product which includes 0.05 and 0.8% expanded hollow polymer balloons in a size between 2 and 150 micrometer.

2.2 The Board can accept the respondent's submission that D1 represents the closest prior art. It discloses particles comprising:

99% - 50% ammonium nitrate and

1% - 50% of a particulate low density bulking material,

whereby the particulate low density bulking material is substantially coated with ammonium nitrate (see claim 1). In Example 1, sawdust was selected as low density material for compounding.

2.3 The respondent has submitted that the technical problem to be solved with regard to D1 is the provision of a prilled product with improved properties, especially low density, high oil absorption capacity and high sensitivity when used in form of an explosive composition ANFO (Ammonium Nitrate mixed with Fuel Oil), see respondent's letter dated 15 December 2000 submitted at the opposition proceedings (page 3, point 9).

2.4 The respondent has argued that the solution to the above technical problem as proposed in claim 1 is the provision of:

(I) a porous product which includes

(ii) hollow polymer balloons

(iii) in the very low concentration of between 0.05 and 0.8%.

2.4.1 Re: Feature (I)

Porosity of the prilled products

The respondent has advanced the argument that the question of porosity is not even mentioned in D1. He has also asserted that this porosity essentially stems from the different method of preparation as compared to D1. At the oral proceedings before the Board, he

has, however, conceded that the porosity of the present products is enhanced by the addition of potassium carbonate. He has also confirmed that potassium carbonate is not an essential component of the composition as claimed.

The Board observes that the respondent has not presented test results which have been obtained with a prilled ammonium nitrate product not including potassium carbonate. It would thus be guesswork to estimate the porosity of the claimed composition which does not necessarily comprise this component. On the other hand, the opposition division has pointed out that the comparative examples filed by the respondent at the opposition proceedings show that the products according to D1 are also porous (see decision under appeal, page 6, second paragraph). The respondent has not provided any argument, let alone proof to refute this finding. The Board therefore does not accept that the feature of porosity can be used to distinguish the claimed product from the prior art.

2.4.2 Re: Feature (iii)

Concentration of hollow polymer balloons between 0.05 and 0.8%.

The appellant has submitted that, with the use of the term "includes", the wording of claim 1 allows for the incorporation of other particulate low density materials into the ammonium nitrate product in addition to the hollow polymer microspheres as expressly stipulated. This interpretation has been contested by the respondent who argued that the specification does not contain any indication that the prilled product

should include any other material of the type of polymer balloons.

The Board notes that, whilst claim 1 indicates the concentration of the hollow polymer microspheres, it does not stipulate **the concentration range of the ammonium nitrate** (emphasis added). The claimed product thus could contain any other component in any amount, as long as the polymer balloons are present in the stipulated concentration. The Board therefore concurs with the appellant in that claim 1 indeed encompasses prilled products according to D1 in which part of the particulate low density bulking material is replaced by hollow polymer microspheres, in the amount between 0.05 to 0.08%, based on the final product.

2.4.3 As a consequence, the Board holds that the solution as proposed in claim 1 is only distinguished from the closest prior art D1 in that the presence of from 0.05% to 0.8% of hollow polymer balloons in the prilled product is essential.

2.5 By letter of 15 December 2000, the respondent has filed an Experimental Report comparing the properties of products according to the patent in suit and those of D1. As is correctly pointed out by the appellant, for comparative purposes, the tested products were made with glass microspheres and not with sawdust as in the examples of D1. Furthermore, the examples made according to the patent in suit contain potassium carbonate, which is not an essential feature of the product of claim 1 (see also point 2.4.1 above). Thus, the results obtained in the Experimental Report are not suitable for demonstrating any improvement of the claimed product over that of D1.

On the other hand, the respondent has not submitted any comparative data based on the sole differentiating feature as established above (point 2.4.3). The Board itself has doubt that any improvement could be obtained by replacing part of the low density material in the product according to D1 with 0.05 to 0.08% of hollow polymer microspheres. Given the lack of evidence or at least of a convincing argument, the Board holds that the technical problem as advanced by the respondent is not being solved by the products proposed in claim 1.

In the Board's judgment, however, the technical problem with respect to D1 can be seen in the provision of a further porous prilled ammonium nitrate product with comparable properties. The question is therefore whether the proposed solution is obvious in view of the available prior art.

2.6 According to the general teaching of D1, the low density material may comprise a combustible material selected from synthetic or natural carbon-containing materials. In addition to sawdust which is used in Example 1, D1 lists as suitable combustible materials "plastic materials including polystyrene (expanded or unexpanded), polyethylene, polypropylene, expanded polyvinyl chloride, expanded polyurethane or like materials, rubber, cotton waste, phenolic glass or other microspheres or the like" (page 7, lines 5 to 10). In the Board's judgment, the incorporation of polymer microspheres into prilled material is thus foreseen by D1.

Polymer microspheres particularly suitable for incorporation into explosive compositions are disclosed in D9. These comprise coated hollow microspheres having

an average particle size of 10-100 micrometer, which have been obtained by heating and foaming and expanding unfoamed microspheres and their coating with a layer of thermosetting resin. Examples of hollow microspheres mentioned in D9 for coating with a thermosetting resin layer include foamed "Expancel™" products (D9, column 3, lines 26 to 28 and lines 50 to 68). These microspheres thus correspond to the definition in claim 1 of "hollow polymer microspheres expanded to a size between 2 and 150 micrometer being polymer balloons". In the Board's judgment, it is obvious to the skilled person that these hollow microspheres are also suitable for a prilled product which is a precursor for explosive compositions. No inventive skill can therefore be seen in replacing a minor amount (0.05 to 0.08%) of the low density material in the prilled product according to D1 by the hollow polymer microspheres according to D9.

As a consequence, the subject-matter of claim 1 of both the main and first auxiliary requests lacks an inventive step with regard to D1 in combination with D9.

Second auxiliary request

3. *Amendments*

3.1 Interpretation of the term "encapsulated".

Claim 1 is directed to "a crystalline porous prilled ammonium nitrate product which includes hollow polymer microspheres being polymer balloons", whilst the

subject-matter of claim 1 as originally filed was "a porous prilled product which includes **encapsulated** (emphasis added) microspheres".

The appellant has submitted that the expression "polymer balloons" cannot be accepted as a synonym for "encapsulated microspheres" since both uncoated or coated ("encapsulated") polymer balloons (such as Expancel hollow microspheres) are well known in the art (see D9, column 3, lines 26 to column 4, line 34). Nor can the ammonium nitrate in the final product be interpreted as the coating or encapsulating medium for the microspheres. This interpretation would be inconsistent with the description of the process for preparing the prilled product where it is stated that the method includes "the step of adding encapsulated microspheres to the product during the prilling of the product" (see description as originally filed, page 7, lines 12 to 14). The deletion of the term "encapsulated" in relation with the microspheres is therefore an infringement of Article 123(2) EPC.

As is, however, observed by the respondent, the only example of "encapsulated microspheres" explicitly mentioned in the original application document are **Expancel**™ microballoons (page 4, line 6; page 15, line 14 and claim 22). Such microspheres are known in the art as hollow microspheres (D9, column 3, lines 50 to 68, in particular lines 62 and 67).

The Board concedes that, according to D9, the Expancel™ microspheres are further encapsulated with a layer of thermosetting resin before their incorporation into the explosive composition. However, this is a specific, proprietary use of the microballoons. On the other

hand, it is undisputed that the raw materials in D9 are commercial products which do not include that thermosetting resin layer. Furthermore, there is no mention of such an additional layer for the Expancel microspheres in the present application documents. In the Board's judgment, the term **"encapsulated microspheres"** in the context of the patent in suit is therefore not restricted to those with a two-layered structure as in D9 but is synonymous with **"hollow microspheres**". This interpretation would be consistent with the description of the preparation process and does not imply that the ammonium nitrate is to be regarded as the encapsulating agent.

3.2 Interpretation of the expression "incorporated in the crystalline structure"

The appellant has pointed out that the expression "incorporated in the crystalline structure" was totally absent from the application documents as filed and there is absolutely no support in the description as to how this feature is to be construed. In particular, it is queried as to how microspheres in a size up to 150 micrometer can be incorporated **into** (emphasis added) the crystalline structure of ammonium nitrate whose dimensions are unknown (see also letter of 27 April 2001, page 4, last paragraph of item 1.2).

As is noted by the respondent and not refuted by the appellant, ammonium nitrate is always crystalline, even if it exists in different crystalline forms (compare original description page 6, lines 6 to 13 and appellant's letter of 27 April 2001, item 1.2, pages 3 to 4). Whilst the respondent has conceded that the expression "crystalline structure", when used in the

crystallographic sense, normally means "crystal lattice", he has argued that the dimensions of such crystal lattices, independent of the crystalline form of the ammonium nitrate, are always in the nanometre range. Thus, it is clear to the skilled person that it is impossible to incorporate the polymer microspheres into the crystal lattice itself. Therefore, the only meaningful interpretation of the expression in question is that the microspheres are "embedded in the crystalline ammonium nitrate".

In the Board's judgment, the respondent's explanation above is plausible and supported by photograph No. 2 showing the microstructure of a prilled product and the corresponding reference thereto in the description (page 12, lines 9 to 13). The Board can therefore accept the respondent's submission that the expression "incorporated in the crystalline structure" should be interpreted as "incorporated in the ammonium nitrate having a crystalline structure".

3.3 In view of the above interpretation, the Board holds that present claim 1 is fairly based on claims 1, 2 and 4 as originally filed and as granted, in combination with the original description, page 6, lines 6 to 11; page 12, lines 9 to 13 and photograph No. 2. Claims 2 to 13 are essentially based on claims 3, 8 to 16, 36 and 37 as originally filed and as granted. The requirements of Article 123(2) and (3) are therefore met.

4. *Sufficiency of disclosure*

The appellant has submitted that, as can be seen from the listing in D5, Expancel™ stands for a number of commercial products but specifically Expancel™ 910 which is used in the examples of the patent in suit is not one of the products listed. There is thus confusion as to the type of microspheres actually used. As a consequence, the skilled person would not be in a position to carry out the invention as disclosed in the patent in suit.

As is correctly submitted by the respondent, the patent in suit gives detailed instructions for selecting suitable polymer microballoons, of which the Expancel™ 910 product is only a preferred embodiment (page 2, lines 29 to 43). Thus, the general disclosure of the patent in suit is not exclusively restricted to the use of a particular type of Expancel™. Furthermore, polymer balloons sold under the trade name Expancel are described in the sale brochure D5 and mentioned in D9 (column 3, lines 62 to 68). The Board therefore holds that suitable hollow polymer microspheres were available to the skilled person at the priority date of the patent in suit.

On the other hand, it is undisputed that clear instructions for preparing the prilled products are given in the description, irrespective of the selected polymer microballoons. In the Board's judgment, specific examples are not necessary in the present case for understanding the disclosure since the skilled person can carry out the claimed invention by choosing a suitable commercial product as polymer balloon and following the given procedure for preparing the prilled

product as described. The Board therefore holds that the conditions of Article 83 EPC are met.

5. *Novelty*

The novelty of the prilled product according to claim 1 is not in dispute. Indeed, the Board observes that none of the documents on file discloses a prilled ammonium nitrate product incorporating polymer balloons which had expanded during the prilling process.

6. *Inventive step*

6.1 As agreed by all parties, D1 is also considered here to comprise the closest prior art.

6.2 With regard to D1, the Board can see the technical problem again in the provision of a further porous prilled ammonium nitrate product with comparable properties.

6.3 The solution to the above technical problem as proposed in claim 1 is the provision of a product which includes polymer balloons which had expanded during the prilling process to a size between 2 and 150 micrometer.

6.4 It is undisputed that the solution as proposed in claim 1 indeed solves the technical problem as stated in point 6.2.

6.5 As was established at the oral proceedings, it is common ground that the wording of claim 1 implies that the balloons are of a thermoplastic material since thermosetting microspheres are not expandable under heating. The patent in suit discloses a process in

which the hollow microspheres are added to the product during the prilling of the product, at a point where the liquid product is divided into droplets. As is indicated in the patent in suit, this procedure serves to minimise the time that the microspheres are affected by the high temperatures (see patent specification, page 3, lines 21 to 28 and lines 41 to 58). In the Board's judgment, it is plausible that such a process allows for a regulation of contact time in order to obtain a desired size of the microballoons, without the risk of their bursting under heat.

In D1, the light-weight material is incorporated into molten ammonium nitrate before the admixture is formed into prills (page 8, lines 11 to 26). As is confirmed by the appellant, due to the temperature of the molten ammonium nitrate and the length of contact time according to the method of D1, the thermoplastic microspheres would burst in the moulding process.

D9 concerns thermoplastic resin hollow microspheres coated with a thermosetting resin; such microspheres are not encompassed by the wording of claim 1 since they would not be expandable during the prilling.

The modification offered in present claim 1 is thus not derivable from the teaching of D1, either by itself or in combination with any of the available prior art documents including D9.

6.6 Claims 2 to 11 are preferred embodiments of the product of claim 1; claims 12 and 13 are directed to blasting compositions including a product according to claim 1. By the same token, the subject-matter of these claims is also accepted as novel and involving an inventive

step. The patent can thus be maintained with the present claims, after the necessary adaptation of the description.

Order

For these reasons it is decided that:

1. The decision under appeal is set aside.

2. The case is remitted to the first instance with the order to maintain the patent according to claims 1 to 13 of the auxiliary request 2 and the description to be amended.

The Registrar: The Chairman:

U. Bultmann R. Spangenberg