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Datasheet for the decision of 15 January 2014

Case Number: T 0735/11 - 3.3.05

Application Number: 06116202.0

Publication Number: 1892219

IPC: C01G49/14, C01G23/053, B01J19/18

Language of the proceedings: EN

Title of invention:
Method of treating a precipitate comprising iron(II)sulphate monohydrate, a plant, granular material and its uses

Patent Proprietor:
Sachtleben Pigments Oy

Opponents:
KRONOS INTERNATIONAL, INC.
Ferro Duo GmbH

Headword:

Relevant legal provisions:
EPC Art. 56

Keyword:
inventive step (no) - reformulation of the problem (no)

Decisions cited:
T 0287/86

Catchword:

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Beschwerdekammern
Boards of Appeal
Chambres de recours

European Patent Office
D-80298 MUNICH
GERMANY
Tel. +49 (0) 89 2399-0
Fax +49 (0) 89 2399-4465

Case Number: T 0735/11 - 3.3.05

D E C I S I O N
of Technical Board of Appeal 3.3.05
of 15 January 2014

Appellant: (Patent Proprietor)	Sachtleben Pigments Oy Titaanitie 28840 Pori (FI)
Representative:	Nobbe, Matthias Demski & Nobbe Patentanwälte Reichspräsidentenstraße 21-25 45470 Mülheim a.d. Ruhr (DE)
Respondent 1: (Opponent 1)	KRONOS INTERNATIONAL, INC. Postfach 10 07 20 D-51307 Leverkusen (DE)
Respondent 2: (Opponent 2)	opposition withdrawn
Representative:	HOFFMANN EITLE Patent- und Rechtsanwälte Arabellastrasse 4 81925 München (DE)
Decision under appeal:	**Decision of the Opposition Division of the European Patent Office posted on 7 February 2011 revoking European patent No. 1892219 pursuant to Article 101(3)(b) EPC.**

Composition of the Board:

Chairman: G. Raths
Members: H. Engl
C. Vallet

Summary of Facts and Submissions

I. European patent EP-B-1 892 219 was granted with 23 claims (Bulletin 2008/11). The patent is concerned with a method of treating a precipitate with iron(II)sulphate monohydrate, with a product obtainable from said method, with the use of the said product in cement manufacture and in cleaning effluent sewage water, and with a plant for producing granular material from a precipitate comprising iron(II)sulphate monohydrate.

II. The European patent was opposed by two opponents on the grounds of insufficiency of disclosure, lack of novelty and lack of inventive step (Article 100(a) and (b) EPC).

III. The documents cited in the opposition proceedings included the following:

D1: WO-A-96/033 133
D5: Brochure from Fa. Lödige: "Systemlösungen für die Umwelttechnik" (2003)
D6: Lödige brochure "Technologien für den Umweltschutz" (dated 07/06)
D6a: *ditto* (dated 05/02)
D7: Pamphlet from Fa. Eirich "Evactherm"
D8a: Pamphlet from Fa. Lödige: "Kontinuierlicher Pflugscharmischer" (2005)
D9: Kronos pamphlet "Produkte und Anwendungen"
D11: EP-A-1 265 823
D16: WO-A-2005/009 917

IV. In its decision the opposition division held that:

- the claims of the main request did not fulfil the

requirements of Article 83 EPC because the patent did not disclose the essential information required to reliably perform the measurement of the dusting values;

- the subject-matter of claims 1 and 14 in accordance with the first auxiliary request were not novel in regard to documents D16 and D18, respectively;

- the second auxiliary request was not admissible because the requirements of Rule 116 EPC and Article 123(2) EPC were not met;

- the subject-matter of claim 1 of the third auxiliary request lacked inventive step in regard to document D16 and claim 13 lacked inventive step in regard to document D6a.

Consequently, all the patentee's requests on file were rejected and the patent was revoked.

V. The patentee's (henceforth: the appellant) notice of appeal was received by letter dated 30 March 2011. The statement of grounds of appeal, dated 13 April 2011, was accompanied by new sets of claims as a main and an auxiliary request. A further submission was received by latter dated 30 April 2012.

VI. Independent claims 1 and 13 of said requests are worded as follows:

Main request:

"1. Method of producing granular material having a high soluble iron(II) content, the method comprising following steps:

- obtaining an amount of a crude precipitate

comprising iron(II)sulphate monohydrate and sulphuric acid from titanium dioxide production,
- mixing the said precipitate and a neutralising agent in a mixing apparatus to obtain a reaction mixture, the temperature during mixing of the reaction mixture being allowed to increase at the most to a temperature of 120°C, characterised in
- adding water to the reaction mixture in amount of 5 - 15 weight-%,
- mixing water to the reaction mixture in the same mixing apparatus and
- keeping the amount of neutralising agent sufficient to give a pH value from 1.5 to 3 to the end product,
- whereby mixing and granulation are done by using the same apparatus."

"13. Plant for producing granular material having a high soluble iron(II) content and treating a precipitate comprising ferrous sulphate monohydrate originating from titanium dioxide production, comprising
- at least one high shear rate mixing apparatus capable of mixing liquid, paste-like and solid material, having
  - a first feeding connection for precipitated ferrous sulphate monohydrate,
  - a second feeding connection for neutralising agent,
  - a third feeding connection for water and
  - an output connection,
whereby the mixing apparatus also functions as granulation apparatus,
- means for transporting the material from the output connection of the mixing apparatus,
- an after-cooler apparatus."

Auxiliary request:

Claim 1 has the same wording as claim 1 of the main request.

"13. Plant for producing granular material having a high soluble iron(II) content and treating a precipitate comprising ferrous sulphate monohydrate originating from titanium dioxide production, comprising
- at least one high shear rate mixing apparatus capable of mixing liquid, paste-like and solid material, having
  - a first feeding connection for precipitated ferrous sulphate monohydrate,
  - a second feeding connection for neutralising agent,
  - a third feeding connection for water and
  - an output connection,
- means for dispatching the steam from the high shear mixing apparatus for removing the steam generated during the drying of the reaction mixture under mixing
- means for transporting the material from the output connection of the mixing apparatus,
- an after-cooler apparatus."

VII. Opponent 2 withdrew its opposition by letter dated 14 May 2013. It is therefore no longer a party to the proceedings.

VIII. Opponent 1 (henceforth: the respondent) filed its observations by letters dated 5 October 2011 and 4 October 2012.

IX. Oral proceedings before the board took place on 15 January 2014. The appellant filed a synoptical

presentation in the form of a table of various data concerning examples 1 to 8 of the patent in suit.

X. The appellant essentially argued as follows:

The invention as claimed in the amended claims was directed to a method for producing a granular material having a high soluble iron(II) content. The material was intended to be used as a reducing agent in cement manufacture or as a flocculation or a cleaning agent in effluent or sewage water treatment. It should be safe to use, to dose and to transport.

To achieve these aims the claimed invention proposed a method of making granules characterised by a combination of selected parameters:

- The temperature of the reaction mixture should remain below 120°C;
- Water should be added in an amount of 5 to 15 weight-%;
- The pH-value should be kept in the range of from 1.5 to 3.

The new claims were sufficiently based on the application as filed and corresponded to those of the third auxiliary request discussed at the oral proceedings before the opposition division, with the amendment and limitation that the mixing and granulation were done using the same apparatus. Thus the requirements of Article 123(2), 83, 84 and 54 EPC were fulfilled for the reasons already given in the contested decision.

As regards inventive step, documents D1, D16 and D6a appeared relevant. However, D6a was inadmissible

because it had not been shown that it had been made publicly available in the same form as it was now presented.

The claimed invention differed from D1 by the formation of the granular product by mixing of water to the reaction mixture in the specified range and by granulating the mixture using the same apparatus.

The objective problem was how to improve the process parameters for the production of a product with improved properties whilst at the same time keeping the process simple.

D1 taught away from the claimed process by keeping the water content low in order to control the reaction rate and the heat evolution in the neutralising step. Therefore, the skilled person would not be encouraged to add water in an amount of 5 to 15 weight-%, as in the process of the contested patent.

Nor was it obvious to select, among the many types of apparatus for mixing and granulation, precisely a combined mixer/granulator, disclosed in D11, D5 or D8a. The Eirich apparatus for the conditioning of ashes from boilers and incinerators, as shown in D6a, page 7, right-hand side, produced only an agglomerated product, not granules, and was therefore not suitable for the claimed method.

XI. The respondent essentially argued as follows:

Main request

New claims 1 and 13 of the main request contravened Article 123(2) EPC, because the originally filed

application documents did not disclose the combination of features as now claimed, viz. "adding 5 to 15 weight-% of water" and "mixing and granulating using the same apparatus".

The claims were also not clear because in the second step of water addition no specific amount of water was defined.

Furthermore, the claimed method did not contain a granulating step. In the examples, granulation was not explicitly mentioned. Nevertheless, in examples 1 to 3 a granular end product was obtained. The final product in accordance with examples 4 to 6 had powdery consistency. It was unclear how "mixing and granulating" could be distinguished from "mixing" alone and why in examples 1 to 3 a granular product was obtained. The patent did not disclose whether in the examples different kinds of mixing apparatuses were used. None of the working examples fell under the scope of the claims as amended. Therefore, the claimed invention was not disclosed sufficiently clearly and completely to be carried out by the skilled person.

Novelty

The subject-matter of claim 1 of the main request was not novel in regard to document D16. Said document disclosed a process for neutralizing Fe(II)monohydrate from the production of TiO_2. In the process, the temperature was kept below 110 °C (see claim 19) and the pH of the final granular product was 2.2 to 2.6 (see examples 31, 3b). For granulation to take place, a defined amount of water (100 to 550 mole-%, corresponding to 7 to 39 weight-%) was added to the mixture (page 13, last paragraph; claim 30). The

granulating was carried out immediately after the addition of water; there was no hint that the mixture should be transferred to another apparatus. Therefore, D16 did not anticipate the claim feature of mixing and granulating in the same apparatus.

The subject-matter of claim 13 lacked novelty over D6a. Contrary to the appellant's arguments, D6a was printed and made publicly available from the date of publication indicated in the imprint, i.e. 05/02 (May 2002). The respondent referred in this respect to T 287/86. D6a revealed an apparatus for the treatment of dusts, comprising a mixer (6), into which dust (1) and water (2) were introduced separately. Optionally, other solid products (2,3) could be added. After mixing, the agglomerated product was transported from the mixer into a storage vessel via a transport belt. Said belt could also be used as a cooling bed for the (granular) agglomerate. It was obvious that the mixer was at the same time a granulating device. Thus D6a anticipated the subject-matter claimed in claim 13.

Inventive step

Moreover, the claimed subject-matter lacked inventive step with regard to D16 and D11 which disclosed a single-step mixing/granulating apparatus. The use of a "Granuliermischer" was also known from D6a and D7.

In the alternative, lack of inventive step could be demonstrated starting from D1 as the closest prior art. Said document disclosed a process of neutralising Fe(II) monohydrate which was a by-product from the production of TiO_2. Fe(II)monohydrate and a neutralising agent containing CaO were mixed in a cement mixing plant. Although the end product was not explicitly

characterised as granular or coarse, it was said to be flowable and non-dusting. Therefore, it must be supposed to consist of small grains, rather than of powdery particles.

Starting from D1, the object was to obtain a granular product. In the light of document D16 it was obvious that granulation could be carried out in a single apparatus and by adding water in the amount specified in the opposed patent. A similar combination of the teachings of D1 and D11 led to the same conclusion of non-inventiveness.

Auxiliary request

The subject-matter of claim 13 differed from the main request only in that the apparatus comprised "means for removal of steam from the mixing device". This kind of "Brüdenabzug" was known from D7 and D11. Its combination with the apparatus of D6a was obvious.

XII. Requests

The appellant requested that the contested decision be set aside and that the patent be maintained on the basis of the claims of the main request or, in the alternative, on the basis of the claims of the auxiliary request, both filed with the statement of grounds of appeal.

The respondent requested that the appeal be dismissed.

Reasons for the Decision

1. Amendments (Article 123 (2), (3) EPC)

1.1 Main request

Claim 1 is based on a combination of features from claims 1 and 3 and the description, paragraph [0045], first sentence, as originally filed.

The board sees no violation of Article 123(2) EPC in combining said features into a new independent method claim, for the following reasons.

1.2 Firstly, claim 3 as originally filed was dependent on claim 1, so that its features, in particular the feature relating to the addition of water in an amount of 5 to 15 weight-%, are disclosed in combination with those of claim 1.

Secondly, the description, paragraph [0045], clearly refers to the invention as such ("According to preferred embodiment of the invention..."). The subsequent preferred feature, relating to mixing and granulation using the same apparatus, is thus understood to be combinable not only with the embodiment defined in claim 1, but also with further preferred embodiments containing features of the claims which depend on claim 1.

Claim 13 is fairly based on original claims 14 and the description, paragraph [0045], first sentence, as originally filed. For the allowability of the combination of features similar arguments as above apply.

1.3 Auxiliary request

Claim 13: The new feature relating to the "means for dispatching the steam from the high shear mixing apparatus" is based on the original disclosure of paragraph [46] of the description.

1.4 The requirements of Article 123(2) and (3) EPC are thus met.

2. Clarity of the claims

The method of claim 1 requires "adding water to the reaction mixture in amount of 5 - 15 weight-%". This feature was already present in granted claim 3 which depended on claim 1. Therefore, any alleged lack of clarity in connection with this particular feature must have affected the claims as granted. Adding still another claim feature, relating to the use of the same apparatus for mixing and granulation, apparently does not influence the alleged clarity problem concerning the amount of water added.

However, objections under Article 84 EPC do not fall under the grounds of opposition (Article 100 EPC) and thus cannot be dealt with in opposition or subsequent appeal proceedings.

The respondent also argued that the process step of granulation had no antecedent in the pre-characterising clause of claim 1, giving rise to a lack of clarity.

However, claim 1 literally relates to a a method of producing a granular material which clearly implies a granulation step.

3. Sufficiency of disclosure (Article 83 EPC)

3.1 The respondent's objection concerns the granulating step which was mentioned neither in claim 1 nor in the examples. Nevertheless, in examples 1 to 3 a granular end product was obtained, whereas the final product in accordance with examples 4 to 6 had a powdery consistency. The patent did not disclose whether in the examples different types of mixing apparatus were used. Therefore, the claimed invention was not disclosed sufficiently clearly and completely to be carried out by the skilled person.

Furthermore, according to the respondent, as none of the examples 1 to 6 comprised a step of water addition, none of them fell under the scope of claims 1 or 13. In the respondent's view, the invention was not supported by working examples and could not be re-worked by the skilled person.

3.2 Generally, for an objection of insufficiency of disclosure to succeed, it is necessary to identify a gap in information and/or lack of guidance which cannot be compensated for by general knowledge.

3.3 In the present case, the board is convinced that granulation of the material can be carried out by the skilled person in spite of the lack of detailed information in the opposed patent. Granulation of a (moist) material is a process common in the art and suitable apparatuses are well known to the skilled person. This implies on the other hand that this particular claim feature cannot be considered as involving an inventive step.

Further explanations in this regard are not necessary,

since the contested patent cannot be maintained for the reasons set out further below.

4. Novelty

4.1 D16 was cited as novelty-destroying for the subject matter of claim 1. The respondent's arguments were in particular that

- the amount of water added in accordance with the teaching of D16 (100 to 550 mole-%) overlapped with the amounts given in the patent in suit;

- granulation was carried out directly after the addition of water, without transfer into another vessel or apparatus; therefore, D16 could be considered to generally disclose mixing and granulation in the same apparatus.

4.2 The board cannot agree for the following reasons. Although D16 does not explicitly mention a step of transferring the reaction mixture from the mixing apparatus to a different apparatus for carrying out the granulation step, this fact cannot be construed as a positive disclosure of the claim feature according to which mixing and granulation shall take place in the same apparatus. On the contrary, D16 discloses on page 14, penultimate paragraph, and in claim 33 that granulation is preferably effected by processes such as mechanical shaping, spraying with air, spraying through a nozzle or by using a rotating disk or a cooling belt or drum. None of these apparatuses is apparently typically designed in such a way that a previous mixing step could be carried out in it.

Novelty of the subject-matter of claims 1 and 13 of the

main request and of claim 1 of the auxiliary request is therefore accepted.

The dependent claims 2 to 12 and 14 are also novel by virtue of their dependency.

The requirements of Article 54 EPC are met.

5. Inventive step

5.1 The opposed patent is concerned with a method of producing a granular material having a high soluble iron(II) content from a crude precipitate comprising iron(II)sulphate monohydrate, and with a plant for producing such a granular material. The claimed method involves mixing and partially neutralising the crude precipitate with water and a neutralising agent while maintaining specified conditions of temperature and pH, and granulating the reaction mixture using a mixing/granulating apparatus.

5.2 D1 (cited in paragraph [0007] of the patent in suit) is considered to represent the closest prior art.

D1 reveals a method of making ferrous sulphate hydrate starting from a moist side product of the titanium pigment (TiO_2) production comprising iron(II)sulphate monohydrate and sulfuric acid. The moisture content of the material is preferably approximately 10% (see page 2, lines 10 to 13, 19 to 21 and 29 to 34). This starting material is partially neutralised to a pH of 1.5 to 5 by mixing in cement as a CaO-containing material and optionally slag, fly ash or limestone (see claims 2 and 3). On a large scale, mixing may be carried out in a continuous concrete mixing plant (see example 1). Importantly, to avoid oxidation of Fe^{2+} and

to retain the hydrate water, the reaction temperature must not exceed 120°C (see page 2, line 29 to page 3, line 12; paragraph bridging pages 4 and 5).

The product so obtained is non-dusting, has excellent handling and flow characteristics (page 3, lines 9 to 12). Although the product presumably consists of particles coarser than dust, it is not unambiguously disclosed as being granular. The product is useful for the reduction of chromium (VI) in the manufacture of cement (see page 1, first paragraph; example 3).

5.3 According to the patent in suit, the problem underlying the patent in suit is to provide a simple method for obtaining, from a crude precipitate comprising iron(II) sulfate monohydrate and sulfuric acid derived from titanium dioxide production, a granular material having a high soluble iron(II) content, with which method in a minimum of time a relatively homogenous material in granulate form can be produced without extensive pre-treatment of the used raw materials (see patent in suit, paragraphs [0010],[0011] and [0012]).

5.4 As a solution to this problem, the patent proposes a method in accordance with claim 1 of the main and auxiliary requests, characterised in that water is added to the reaction mixture in an amount of 5 to 15 weight-% and that mixing and granulation is carried out in the same apparatus.

5.5 As to the success of the solution, it is plausible that by the claimed method a granular product having a high soluble iron(II) content can be obtained. The board also considers that the claimed process is simplified insofar as the mixing and granulation takes place in the same apparatus.

The board is therefore satisfied that the problem as stated in the patent in suit has been successfully solved.

Hence, there is no need to reformulate the problem in the light of document D1.

5.6 The question to be answered now is whether the claimed solution was obvious in view of the prior art.

The board notes that the patent in suit does not place any particular restrictions on the type of apparatus used for the combined mixing/granulation. According to preferred embodiments, a high-shear mixer or a fluid-bed mixer (such as an R-type Eirich mixer or a Lödige mixer) is used; however, these are not mandatory in the claimed process. The board concludes that any suitable mixing/granulation apparatus known in the art may be successfully employed in the claimed method.

The respondent has pointed out that such combined apparatuses are known for instance from D11 (column 5, lines 28 to 32; Figure 1), D5, D7 and D8a. Particular attention was directed to D5, page 14, "Maschinen für Chargenbetrieb", disclosing Eirich batch mixers for simultaneous mixing, moistening, reacting, granulating and surface-drying. D8a (see page 2) discloses a continuous ploughshare mixer having optional mixer blades in the mixing drum for disintegration of agglomerates and the forming of a granulate.

Document D11 discloses in Figure 1 and column 6, lines 19 to 31 and 47 to 55, a combined mixer/reactor/granulator having inlets for sludge (9), inlets (7, 14) for solids such as CaO, and an inlet (15) for water, a

mixing/granulating chamber (2) with rotating blades (4), an outlet (10) for the granulated product (11) and a steam exhaust (13).

In the board's view, the skilled person would employ the combined mixing/granulation apparatus disclosed in D11 in the method otherwise known from D1 to provide a simple method of making a granular product.

As regards the step of adding water to the reaction mixture, the board considers that it is *a priori* obvious to make sure that the mixture contains enough water - depending on the moisture content of the raw material - for the formation of granules. Adding water to the mixture is an obvious measure to adjust the water content if the initial amount in the starting product and reactants is insufficient for that purpose. The exact total water content necessary for granulation must be determined experimentally, for the opposed patent confines itself to explaining what is in the board's opinion common knowledge, namely that raw materials with high water content yield soft and irregular granules, whereas medium water content in the reaction mixture leads to effective formation of dry and hard granules (see paragraph [0034].

The amount to be added as specified in the claims (5 to 15 weight-%) is, in the board's view, neither selected purposefully nor particularly critical, because the initial moisture content of the crude precipitate (up to 10% according to D1) is not taken into account and the amount of water lost as steam during neutralisation (where the temperature rises to a maximum of 120°C) is not known *a priori*. Therefore, for determining the actual amount of water which must be added to obtain granules, the patent's teaching offers of little

guidance.

According to an argument of the appellant, the fact that no water is added in the method of D1 would teach away from the method as now claimed. The board does not find this argument persuasive, because the aim in document D1 is to produce a powdery, non-dusting product with good flow characteristics (see page 3, lines 5 to 19). As no granulation takes place in D1, there is apparently no need to add water to the mixture. In contrast, in the claimed invention, it is obvious that the water content must be controlled and, if necessary, water must be added, for granulation to take place. On the importance of controlled water addition for the granulation process, see for instance D11, paragraphs [0021], [0022], [0035] and [0044].

The fact is that the basis for the amount of water (5 to 15%) to be added is not indicated in the claim. Therefore, it is not clear whether the claimed weight - percentages of water addition refer to the amount of initial material, to the mixture of materials or to the solids content of the mixture. This claim feature can only be taken as a hint to add water but it cannot be considered as involving an inventive step.

5.7 The subject-matter of claim 1 of the main request and of the identical claim 1 of the auxiliary request thus does not involve an inventive step (Article 56 EPC). Therefore, these requests must both fail.

Order

For these reasons it is decided that:

The appeal is dismissed.

The Registrar: The Chairman:

C. Vodz G. Raths

Decision electronically authenticated