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Datasheet for the decision of 30 July 2008

Case Number:	T 0287/06 - 3.3.05
Application Number:	96939738.0
Publication Number:	0866770
IPC:	C01F 11/18
Language of the proceedings:	EN

Title of invention:
Process for the preparation of discrete particles of calcium carbonate

Patentee:
MINERALS TECHNOLOGIES INC.

Opponent:
OMYA AG

Headword:
Calcium carbonate/MINERALS TECHNOLOGIES

Relevant legal provisions:
EPC Art. 54(1) (2), 83, 56

Relevant legal provisions (EPC 1973):
-

Keyword:
"Sufficiency of disclosure (yes)"
"Novelty (yes)"
"Inventive step (yes) - improvement"

Decisions cited:
T 0606/06, T 0077/87

Catchword:
-

Case Number: T 0287/06 - 3.3.05

DECISION
of the Technical Board of Appeal 3.3.05
of 30 July 2008

Appellant:
(Opponent)

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(Patent Proprietor)

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Decision under appeal:

Decision of the Opposition Division of the European Patent Office posted rejecting the opposition filed against European patent No. 0866770 pursuant to Article 102(2) EPC 1973.

Composition of the Board:

Chairman: G. Raths
Members: J.-M. Schwaller
H. Preglau

Summary of Facts and Submissions

I. This appeal was lodged by the opponent against the decision of the opposition division rejecting the opposition against the European patent No. 0 866 770, independent claim 1 of which reads as follows:

"1. A process for the preparation of discrete particles of prismatic calcium carbonate comprising preparing a first calcium hydroxide slurry from calcium oxide and water, subsequently adding to said first slurry from about 0.1 weight percent to 5.0 weight percent of a saccharide or polysaccharide and from about 0.1 weight percent to about 5.0 weight percent of aluminum sulphate to form a second calcium hydroxide slurry and while rapidly agitating, carbonating the thus prepared second calcium hydroxide slurry until the carbonation is substantially complete so as to obtain said discrete particles of prismatic calcium carbonate, said carbonation being started at a temperature of from about 8°C to about 64°C."

II. During the opposition procedure, the parties relied *inter alia* upon the documents:

D3: US 3443 890

D4: US 2 188 663

D5: GB 1 540 328

D6: PL 139391 (abstract)

D7: JP 54040830 (abstract)

D9: Tr. Nauchn.-Issled. lnst. Osnovnoi Khim. (1963), 15, 19-63 (abstract)

D11: Translation into English and abstract of RO 87978

D12: Translation into English and abstract of JP 60103025

D13: Soil Sci. Soc. Amer. Proc., vol. 33, 1969, 690-693

D14: US 4 892 590

III. The contested decision can be summarized as follows:

The invention is disclosed in a manner sufficiently clear and complete for it to be carried out by a person skilled in the art because the particle size and the specific surface area being product features, they are not essential technical features in defining a process for the preparation of calcium carbonate.

The subject-matter of claim 1 as granted is novel over each of D12 or D14 in the following respects:

- D12 discloses a list of crystal nucleating agents, the list comprising both aluminum salts and sucrose; however, D12 does not disclose the combination of sucrose and aluminum salts;
- D14 discloses the addition of starch after the preparation of the calcium carbonate; hence, starch is not present during the preparation of the calcium carbonate.

Concerning inventive step, (poly)saccharides and aluminum sulfate are both known as useful in the preparation of fine calcium carbonate particles, but as shown in Table 3 of the contested patent, the combination of sucrose and aluminum sulfate results in an improved specific surface area compared to sucrose alone. Although both sucrose and aluminum sulfate may be known from the prior art to affect the specific surface area, there is no teaching that the combination of these two substances would result in an additional increase in specific surface area.

IV. In its grounds of appeal dated 19 April 2006, the appellant objected to the contested patent under Article 100(a) and (b) EPC.

V. The respondent reacted to the grounds of appeal in a letter dated 26 October 2006.

VI. In response to the summons to oral proceedings, the appellant reiterated in essence its position submitted in the grounds of appeal in a memorandum dated 30 June 2008. In particular, it considered D12 as the starting point for assessing inventive step.

VII. At the oral proceedings, which took place on 30 July 2008, while maintaining its previous objections, the appellant amended its line of argumentation and considered document D4 as representing the closest state of art.

VIII. The appellant's arguments can be summarised as follows:

(a) Concerning the objection raised under Article 100(b) EPC:

- The particle size range (0.5 to 0.018 µm) described as essential in the description (paragraph [0004] being absent from claim 1, the skilled person was free to produce any particle size.

- A relationship between the specific surface area and the particle size existed only in the specific case in which the particles were uniform in size and not aggregated.

- The method for determining the particle size was absent from the description.

- The subject-matter of claim 1 was abusively broad as the process claimed allowed also the production of particles with sizes outside that described in paragraph [0004] of the contested patent.

- The meaning of the words "discrete" and "prismatic" in claim 1 being unclear, it was impossible to determine whether the skilled person works within the terms of the claims or not.

(b) Claim 1 lacked novelty over D12, which disclosed at page 5 (2nd paragraph) a list of crystal nucleating agents made of:

  - on the one hand:

(i) mineral acids such as HCl, sulfuric and nitric acid, and lower carboxylic acids such as formic, acetic and propionic acid

(ii) "and/or" an ammonium salt, an alkali metal salt, and a water soluble salt of a metal, such as aluminum, zinc and magnesium, thereof;

- on the other hand (see the semi-colon after "thereof"): polyhydric alcohols such as sorbitol and pentaerythrytol and sucrose.

This disclosure clearly included the combination of an aluminum salt of sulfuric acid and sucrose. When furthermore read in the light of the abstract - which emphasizes aluminum sulfate by bold printing - alum was thus clearly disclosed and emphasised in this document.

In line with G 6/88 (OJ EPO 1990, 114), the sucrose/ alum combination was thus made available to the public by D12, when reading the translation and the abstract. The other features of claim 1 were found inter alia in the abstract.

(c) Claim 1 lacked an inventive step, because when starting from document D4, the problem to be solved was to be seen in the provisions of getting smaller particles while avoiding a close control of the temperature. The solution to this problem, namely using aluminium sulphate in addition to sucrose, was obvious in view of the content of document D6 in combination with either D7 or D9. Alternatively, it argued that the above solution was also suggested by documents D3, D5, D11, D12 and/or D13.

IX. The appellant requested that the decision under appeal be set aside and that the patent be revoked and the respondent requested that the appeal be dismissed.

Reasons for the Decision

1. *Sufficiency of disclosure*

1.1 According to the case law of the boards of appeal, the burden of proof is upon the opponent to establish that the skilled reader of a disputed patent, using his common general knowledge, would be unable to carry out the invention therein claimed.

In the present case, the appellant did however not provide any evidence in support of its allegations. In particular it did neither prove that the process according to claim 1 could not be carried out, nor give any evidence that its subject-matter was so broad that it would allow also the production of particles having a size outside that described in paragraph [0004] of the contested patent, nor did it provide any evidence that the process as defined in claim 1 would not lead to "discrete particles of prismatic calcium carbonate".

1.2 The appellant's arguments (see item VIII. (a)) cannot be accepted for the following reasons:

- The arguments which specifically address the particle size or the specific surface area of calcium carbonate are irrelevant, since the process for the preparation of "***discrete particles of prismatic calcium carbonate***" as presently defined in claim 1 recites none of these features.

- Concerning the argument that the terms "discrete" and "prismatic" would be so unclear that the skilled person would not be able to determine whether it would work within the terms of the claims, the board observes that - according to the Oxford English Dictionary (http://dictionary.oed.com) - "discrete" means "***separate, detached from others, individually distinct***" and "prismatic" means "***elongated, straight, and angular (esp. hexagonal) in cross-section***". Furthermore, the contested patent (page 3, lines 50 to 52) describes that "prismatic calcium carbonate" means that the calcium carbonate particles have a generally prismatic shape and the aspect ratio (L/W) averages 2.0 or less.

1.3 For the above reasons, the board has no doubts that the process for preparing discrete particles of prismatic calcium carbonate as defined in present claim 1 is disclosed in a manner sufficiently clear and complete to be carried out by a person skilled in the art.

2. *Novelty*

2.1 The appellant contested the novelty of the subject-matter of claim 1 as granted in the light of D12.

2.2 As disclosed in the translation into English submitted by the patentee (now the respondent) on 14 November 2005, D12 relates to a process for the production of superfine calcium carbonate with a BET specific surface area of 60 m^2/g or higher, wherein, after adding a crystal nucleating agent to an aqueous suspension of calcium hydroxide, carbon dioxide gas of a concentration of 15% by volume or higher is blown into the suspension at a flow rate of 40 L/min/kg $Ca(OH)_2$ or higher, and carbonation is carried out by adding a chain particle formation accelerator before the carbonation index reaches 40% (claim 1).

According to page 5 (2nd and 3rd paragraphs) of the same document:

- examples of the crystal nucleating agent include mineral acids, lower carboxylic acids and/or water-soluble metal salts thereof such as ammonium salts, alkali metal salts, **aluminum salts**, zinc salts and magnesium salts, polyhydric alcohols such as sorbitol and pentaerythritol, and **sucrose** ***(emphasis added by the board)***.

- examples of the chain particle formation accelerator include chelating agents and anionic polymer compounds.

Among the four "Practical Examples" of D12, aluminium sulphate is disclosed in Example 1, and (poly)saccharide in Examples 3 (sorbitol) and 4 (sucrose).

2.3 The appellant's arguments summarized in item VIII (b) ***supra*** cannot be accepted for the following reasons:

2.3.1 According to established jurisprudence (T 606/06, point 2.10.3; T 77/87, point 4.1.4), an abstract and its original basic document cannot disclose two different subject-matters. This means that, even if - as in the present case with respect to D12 - aluminum sulphate is emphasized by "bold" marking in the abstract, the latter cannot be considered as disclosing more than the original document (i.e. JP 60103025) or its certified translation into English.

2.3.2 As to the question whether the combination of aluminum sulphate and (poly)saccharide is disclosed in D12, even if there were a semi-colon in the list of crystal nucleating agents disclosed at page 7 of the appellant's translation into English of D12, the skilled person would have to make at least three choices in order to arrive at the combination of a (poly)saccharide and aluminum sulphate as defined in claim 1 as granted, namely:

- in a first step, a <u>first choice</u> in a list of suitable mineral acids (HCl, sulphuric acid, nitric acid) and a <u>second choice</u> in a list of metals (aluminum, zinc, magnesium) suitable as water soluble salt, in order to arrive at aluminum sulphate, i.e. the aluminum salt of sulphuric acid;

- then, in a second step, a <u>third choice</u> among another list of compounds, namely those quoted as "polyhydric alcohols such as sorbitol and pentaerythrytol and sucrose".

2.3.3 G 6/88 cited by the appellant has no relation to this case, as this decision refers to the question whether a claim to the use of a chemical compound for a particular non-medical purpose is novel within the meaning of Article 54 EPC, having regard to prior art which discloses the use of that compound for a different non-medical purpose, if the two teachings are carried out by identical technical means and the only novel feature in the claim is the use itself.

In this case, document D12 relates to a process for the production of superfine calcium carbonate, and the question to be answered was not whether a known compound was used for a same or a different purpose but whether document D12 disclosed the combination of aluminum sulphate and (poly)saccharide. Therefore G 6/88 has no bearing at all on this case.

2.3.4 The board concludes that D12 does not directly and unambiguously disclose aluminium sulphate **in combination** with a saccharide or polysaccharide during the preparation of calcium carbonate.

2.4 As furthermore none of the other cited prior art documents discloses the subject-matter of claim 1 as granted, it is concluded that the latter is new within the meaning of Articles 52(1) and 54(1)(2) EPC.

3. *Inventive step*

3.1 In accordance with the established "problem-solution approach", it is necessary to identify the closest state of the art, to determine in the light thereof the technical problem addressed by the alleged invention and that the latter successfully solves, and finally to examine the obviousness of the claimed solution to this problem in view of the state of the art.

3.2 In agreement with the appellant, document D4 is taken as the starting point for assessing inventive step, as it concerns the production of fine particles of calcium carbonate, by addition of one or more agents selected from monosaccharides, disaccharides and polyhydroxy alcohols containing 4, 5 or 6 hydroxyl groups to a suspension of calcium hydroxide in water, and treating the suspension with carbon dioxide gas. The process enables the production of fine particle size calcium carbonate over inter alia a wide range of temperatures and concentrations of calcium hydroxide (D4, page 1, left column, lines 1 to 24).

As indicated at page 4, left column, lines 11 to 22, in producing calcium carbonate of a given fineness, the addition of sucrose increases the reaction temperature range employed, irrespective of the purity of the calcium oxide used, the method of slaking it, the concentration of the calcium hydroxide in water suspension, the concentration of the carbon dioxide gas, the rate of addition of the carbon dioxide gas to the suspension or the degree of agitation of the suspension.

By reference to Figure 2, it can in particular be inferred that 5 grams sucrose per liter of suspension (i.e. a sucrose concentration of 0.5 wt. %) enable the use of any temperature between 12 and 75° C. In Table II, sucrose is employed in concentrations up to 50 g per liter (i.e. up to 5 wt. %) in a temperature range however narrowed to 35 to 60° C.

It is to be noted that the particle size of the calcium carbonate produced by the above method is not directly and unambiguously derivable from D4.

3.3 Starting from this state of the art, the problem to be solved by the subject-matter of the disputed claim 1 consists - as stated by the appellant - in the provision of a process to get smaller particles while avoiding a close control of the temperature.

3.4 As a solution to this problem, the patent in suit proposes a process according to claim 1 which is characterized by adding to the first calcium hydroxide slurry from about 0.1 wt. % to about 5.0 wt. % aluminum sulphate further to 0.1 to 5.0 wt. % of (poly)saccharide.

3.5 As to the question whether the technical problem was effectively solved, the appellant held that it was not solved, arguing that the specific surface area increase shown in Table 3 could not be directly correlated with the diminution of the particle size. It did however provide no evidence in favour of this statement.

The board moreover does not agree with the appellant because - as indicated in the patent in suit - the size of discrete particles of prismatic calcium carbonate being extremely small (0.018 to 0.5 μm), this parameter is more accurately determined and expressed by the specific surface area measurement (paragraph [0027]). As furthermore derivable from paragraph [0045] and evidenced by the specific surface area measurements summarized in Table 3 of the patent in suit, the addition of alum (aluminum sulfate) to sucrose prior to carbonation reduces the particle size of calcium carbonate in comparison to the addition of sucrose alone. This effect exists for the whole temperature range extending from 10 to 38 °C, as corroborated by Table 3.

Therefore the board is satisfied that the problem as defined under point 3.3 has been successfully solved.

3.6 The question which thus remains to be answered is whether the solution as proposed by the subject-matter claimed is obvious or not in view of the cited prior art.

3.7 The appellant argued that the disclosure of document D6 taken in combination with the teaching of either D7 or D9 would render obvious the subject-matter of claim 1 as granted.

3.7.1 D6 reads:

"$CaCO_3$ is produced by carbonation of $Ca(OH)_2$ in a sucrose (I) soln. (prepd. by contacting CaO with the aq. sucrose soln.) at 20-90°. The resulting product contg. 2-10 wt.% I is useful for manuf. of lactic acid. I prevents caking and dusting of $CaCO_3$. Thus, 6.6 g CO_2 was absorbed in 0.3 L aq. soln. contg. $Ca(OH)_2$ 3.3 and I 13.5 during 20 min at 20° and pH 8-8.2. The resulting ppt. was sepd. by filtration and dried 2h at 105° to give a product (particle size 0.5-0.8 μm) consisting of 14 g $CaCO_3$ and 0.6 g I."

The board cannot follow the appellant's argument that the particle size of the calcium carbonate produced in D4 can be derived from D6 for the following reasons:

- D6 discloses that the production of a product (particle size: 0.5 to 0.8 μm) consisting of **14 g $CaCO_3$** and 0.6 g sucrose by absorption of **6.6 g CO_2** in **0.3 liter** of an aqueous solution containing $Ca(OH)_2$ **3.3** (*sic*) and sucrose **13.5** (*sic*). D6 does however not indicate the unit regarding the amounts of $Ca(OH)_2$ and sucrose used.

- The unit "gram" can be excluded, because it is impossible to produce 14 g $CaCO_3$ - as indicated in the abstract - with only 3.3 g $Ca(OH)_2$ and 6.6 g CO_2.

- The unit "percentage" (e.g. by weight) is more realistic, as it would render plausible the production of the 14 grams of $CaCO_3$. With this unit, the sucrose concentration (13.5 wt. %) would however be far away from that disclosed in D4 (namely 0.1 to 5 wt. %) or from that defined in claim 1 under dispute.

Accordingly, as it is not directly and unambiguously derivable from D6 that the amount of sucrose used therein is the same as in D4, it is impossible to draw any conclusion as to the particle size of the calcium carbonate produced in the latter document.

3.7.2 Concerning D7 and D9, the following is observed:

(a) D7 - also an abstract - discloses the preparation of highly transparent calcium carbonate by mixing aqueous "$Ca(OH)_2$ suspensions 100 (based on solid content)" with 2-10 parts of aqueous sulfates and reacting with CO_2 at 20-40° to give $CaCO_3$ having an average grain size of 50–200 Ǻ. Specifically, D7 discloses that 8 parts of aqueous $Al_2(SO_4)_3$ are mixed with an aqueous "$Ca(OH)_2$ suspension 100 (based on solid content)" and reacted with CO_2 at 25° to give 60 Ǻ $CaCO_3$.

(b) D9 - a further abstract - summarizes the content of a scientific article studying the production conditions of highly dispersed $CaCO_3$ calcium carbonate pigments. In particular, the effect of additives during the manufacture of calcium carbonate e.g. on its specific surface area or on its particle size has been studied. The additives tested included $FeCl_3$, triethanolamine oleate with $Al_2(SO_4)_3$, Na_2SiO_3, $MgCl_2$, household soap, sulfated soap, triethanolamine, stearic acid, urea, thiourea and formamide. The production conditions used to produce an acceptable $CaCO_3$ pigment with a surface area of 30 to 50 m^2/g included a $Ca(OH)_2$ concentration of 120 g/l, a carbonation gas containing 26% CO_2, a temperature of from 13 to 20°, a carbonation rate of from 400 to 600 l CO_2/hr.kg $Ca(OH)_2$ and a final pH of 7.1 to 6.4.

3.7.3 If, as argued by the appellant, the skilled person faced with the above problem (item 3.3) takes D7 or D9 into consideration, it would not find any incentive in these documents to add "about 0.1 to about 5.0 wt. % of aluminum sulphate" to a sucrose-containing calcium carbonate slurry - such as the one disclosed in D4 - because neither D7 nor D9 discloses or suggests that smaller particles - in comparison to those obtained with sucrose only - might be obtained with the addition of aluminum sulphate.

As the skilled person furthermore neither knows the particle size, nor the surface area of the $CaCO_3$ produced in D4, he would not be prompted to add a further additive, let alone aluminum sulphate, to the sucrose-containing slurry disclosed in D4 to decrease the $CaCO_3$ particle size.

3.8 Concerning the other documents cited by the appellant, the board comments as follows:

3.8.1 D3 (column 1, lines 14 to 32 and column 2, lines 6 to 9) teaches the use of a second additive in addition to e.g. a mono- or disaccharide in order to produce extremely fine calcium carbonate particles over a wide range of reaction temperatures and reactant concentrations, however the additive is SiO_2 or solubilised starch, not aluminum sulphate as requested in claim 1 under dispute.

3.8.2 D5 (claim 1) discloses a process for producing chain structured corpuscular calcium carbonate comprising carbonating calcium hydroxide suspended in water at a temperature of 0° C to 300° C in the presence of a chelating agent until the suspension becomes a viscous colloid, thereafter adding from 0.00001 to 0.5 mole per mole of calcium hydroxide of a water-soluble salt and continuing the carbonation. In claim 5, the chelating agent is disclosed as being selected from aliphatic carboxylic acids, oxy- or ketocarboxylic acids, thio-carboxylic acids, aromatic carboxylic acids or aldehydes, aromatic sulfonic acids, aminopolycarboxylic acids, amino acids or proteins, purine bases or nucleosides, antibiotics, metallo-chromic indicators, oximes and diketones, amines, and salts thereof.

In Example 16, aluminum sulphate is used as the water-soluble salt and 2Na.EDTA as the chelating agent.

D5 does however not suggest to use aluminum sulphate in combination with a saccharide or polysaccharide, or to add aluminum sulphate to a sucrose-containing slurry, such as that of D4, in order to decrease the particle size of the $CaCO_3$.

3.8.3 D11 discloses a process for making pure calcium carbonate in the form of calcite or aragonite from a purified solution of calcium hydroxide containing 5 to 15% mineral salts, phenols, polyalcohols, saccharides or polysaccharides (see the claim). The carbonation with concentrated carbon dioxide at a temperature of 75 to 95°C results in a precipitate of pure calcite having a particle size of less than 10 μm. Under conditions of carbonation where Mg^{2+} ions are present in a concentration of 1 to 2% in a solution of calcium hydroxide, pure aragonite with a particle size of less than 10 μm is precipitated.

In Example 2, D11 discloses the use of sucrose and magnesium nitrate, both being used at a concentration of 10%.

Even if, for the sake of argumentation, it could be admitted that the above example would suggest the use of sucrose in combination with another metallic salt, the concentrations used in the Example are much higher than those defined in claim 1 under dispute. Furthermore, D11 does not suggest the use of aluminum sulphate in combination with a saccharide or polysaccharide, let alone to add aluminum sulphate to a sucrose-containing slurry, such as that of D4, in order to decrease the particle size of $CaCO_3$.

3.8.4 D12 discloses - as already indicated in item 2.2 - a process for producing superfine calcium carbonate having a BET specific surface area $\geqslant 60\ m^2/g$, said process comprising the addition inter alia of either aluminum sulphate **or** sucrose to an aqueous suspension of calcium hydroxide.

In order to achieve the above particle fineness, D12 however **requires the further addition of a chain particle formation accelerator** such as a chelating agent or an anionic polymer compound (claims 1 and 3), and the examples show that in the absence of such a further additive, the particle fineness as claimed in D12 cannot be achieved.

D12 furthermore does neither disclose nor suggest to add both aluminum sulphate and sucrose to obtain the said fineness, let alone to add aluminum sulphate to a sucrose-containing calcium hydroxide slurry in order to further decrease the particle size of $CaCO_3$.

3.8.5 D13, which studies the solubility of calcium carbonate precipitated in aqueous solutions of magnesium and sulphate salts, neither discloses aluminum sulphate, nor any saccharide or polysaccharide, let alone their combined use in the preparation of calcium carbonate.

3.9 The remaining documents cited during the opposition proceedings do not contain further information which would point towards the claimed solution of the problem stated above.

3.10 Accordingly, for the reasons indicated above, the subject-matter of claim 1 and by the same token that of dependent claims 2 to 7, which include all the features of claim 1, involves an inventive step within the meaning of Articles 52(1) and 56 EPC.

Order

For these reasons it is decided that:

The appeal is dismissed

The Registrar:	The Chairman:
D. Magliano	G. Raths