BESCHWERDEKAMMERN DES EUROPÄISCHEN PATENTAMTS | BOARDS OF APPEAL OF THE EUROPEAN PATENT OFFICE | CHAMBRES DE RECOURS DE L'OFFICE EUROPEEN DES BREVETS

Internal distribution code:
(A) [ ] Publication in OJ
(B) [ ] To Chairmen and Members
(C) [X] To Chairmen

D E C I S I O N
of 9 February 2000

Case Number: T 0567/97 - 3.3.5

Application Number: 89906611.2

Publication Number: 0416015

IPC: C01G 23/07

Language of the proceedings: EN

Title of invention:
Process for preparing titanium dioxide

Patentee:
KERR-MCGEE CHEMICAL LLC

Opponent:
Tioxide Group Limited

Headword:
Titanium dioxide/KERR-MCGEE

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

Keyword:
"Novelty - yes"
"Inventive step - yes, non-obvious alternative"

Decisions cited:
-

Catchword:
-

Europäisches Patentamt | European Patent Office | Office européen des brevets

Beschwerdekammern | Boards of Appeal | Chambres de recours

Case Number: T 0567/97 - 3.3.5

D E C I S I O N
of the Technical Board of Appeal 3.3.5
of 9 February 2000

Appellant: (Opponent)
Tioxide Group Limited
Lincoln House, 137-143 Hammersmith Road
London W14 0QL (GB)

Representative:
Jackson, John Derek
Tioxide Group Limited
Patent Department
Haverton Hill Road
Billingham, Cleveland TS23 1PS (GB)

Respondent: (Proprietor of the patent)
KERR-MCGEE CHEMICAL LLC
123 Robert S. Kerr Avenue
Oklahoma City
Oklahoma 73102 (US)

Representative:
Woods, Geoffrey Corlett
J.A. KEMP & CO.
14 South Square
Gray's Inn
London WC1R 5LX (GB)

Decision under appeal: **Decision of the Opposition Division of the European Patent Office posted 10 March 1997 rejecting the opposition filed against European patent No. 0 416 015 pursuant to Article 102(2) EPC.**

Composition of the Board:

Chairman: R. K. Spangenberg
Members: G. J. Wassenaar
M. B. Günzel

Summary of Facts and Submissions

I. The appeal is from the decision of the Opposition Division to reject the opposition against European patent No. 0 416 015 comprising claims 1 to 7. Claim 1 thereof reads as follows:

"A method for preparing a rutile titanium dioxide pigment by reaction of a mixture of a titanium halide and an oxidizing gas in a vapour phase in a reaction zone of a vapour phase oxidation reactor at a temperature of at least 800°C in the presence of an added metal ion containing compound wherein the metal is selected from the group consisting of Groups IA, IIA and the Lanthanide Series of metals of the Periodic Table of Elements, said metal ion containing compound being present in a total amount of from about 100 to about 1000 parts per million, based upon the weight of the titanium dioxide pigment being produced, characterised by the steps of:

introducing said metal ion containing compound into said reaction mixture of titanium halide and oxidizing gas in said reaction zone in separate and discrete increments said increments comprising a first increment and at least one additional increment, wherein said first increment is introduced into said reaction zone at a point therein prior to onset of reaction between said titanium halide and oxidizing gas within said reaction zone and wherein said at least one additional increment is introduced into said reaction zone at a point therein subsequent to the reaction of at least 20 weight percent of the titanium halide with the oxidizing gas; and

recovering the titanium dioxide pigment substantially as produced."

II. In the decision, *inter alia*, the following prior art documents were considered:

D2: GB-A-1 153 637

D5: GB-A-1 161 514

D6: US-A-3 640 745

D8: GB-A-1 116 142

The Opposition Division held that the subject-matter of the claims as granted was new and involved an inventive step in view of the available prior art documents. In the grounds of the decision it was indicated that the process according to the patent in suit improved the selectivity of the formation of the rutile modification and the filterability of the formed titanium dioxide. It was held, that none of the available prior art documents mentioned the problem of improving the filterability and that there was no suggestion for the measures taught in the patent in suit to solve this problem.

III. In the statement of the grounds of appeal, the appellant(opponent) maintained that the subject matter of the granted claims lacked novelty and inventive step. Apart from the citations already on file before the Opposition Division, further reference was made to a new citation:

D10: GB-A-1 184 448.

During oral proceedings, which were held on 9 February 2000, novelty was only attacked on the basis of D5 in which reference is made to D8. Earlier further

submissions with respect to novelty were no longer maintained. Lack of inventive step was only argued on the basis of the prior art discussed in the patent in suit in combination with D2 and D10. The arguments of the appellant may be summarised as follows:

D5 disclosed in a process for the production of titanium dioxide by vapour phase oxidation of titanium chloride the addition of at least one source of potassium metal. One or more sources thereof might be added in conjunction with an inert gas, or one or more of the reactants, or both. The source of potassium might also be added directly to the reaction zone by a baffle as disclosed in D8. According to D8, the baffle is positioned in the reactor at a point wherein at least 20 wt.% of the titanium chloride has been reacted. The process of present claim 1 was, therefore, already envisaged in D5 as a suitable embodiment.

The examples in the patent in suit were not suitable to demonstrate an improvement over the prior art. In view of D6 it was unlikely that higher amounts of potassium ions added to the reactor would cause filtration problems. There was no evidence, that the problem underlying the invention as indicated in the decision under appeal was actually solved. According to the acknowledgement of the prior art in the patent in suit it was known to add potassium ions to the oxidizing gas stream or, if desired, directly into the reactor, ahead of the actual flame of the reaction. According to D2 and D10 it was advantageous to add a metal source such as potassium chloride to the effluent stream, ie at a point where at least 20 wt.% of the titanium chloride had been reacted. It was obvious to combine this teaching with the known process of adding potassium ions ahead of the reaction zone, whereby the claimed process was obtained. The process of claim 1, therefore, lacked an inventive step.

IV. With the response to the grounds of the appeal the respondent refuted the arguments of the appellant and submitted new evidence showing the effect of the positioning of the potassium ion injection point on the rutile content of the product. With a letter dated 6 January 2000 a new set of claims 1 to 6 was submitted as an auxiliary request. The respondent's arguments may be summarized as follows:

As testified by comparative examples, the process according to the patent in suit solved the problem of producing titanium dioxide pigment having desirable properties such as a uniform size, a high rutile content and a suitable CBU value with a process in which the particles suspended in the reactor effluent could be easily filtrated. The solution of this problem provided by the patent in suit was not disclosed or suggested by any of the cited documents.

V. The appellant requested that the decision under appeal be set aside and that the patent be revoked.

The respondent requested that the appeal be dismissed and that the patent be maintained. As auxiliary request, the respondent requested to maintain the patent with claims 1 to 6 filed with the respondent's letter dated 6 January 2000.

Reasons for the Decision

1. The appeal is admissible.

2. *Main request*

2.1 Novelty

D5 discloses in a process for the production of titanium dioxide by vapour phase oxidation of titanium chloride the addition of at least one source of thorium and at least one source of at least one metal selected from zinc, potassium, rubidium, caesium, sodium and lithium (page 1, lines 35 to 50). The source of the various additives can be introduced to the vapour phase reaction together or separately. One or more sources of one or more of the additives may be added in conjunction with an inert gas, or one or more of the reactants, or both. The source of each selected additive may also be added directly to the reaction zone independently of the reactants or the inert gas, eg as an atomized spray. The additives may also be added by erosion of construction parts inside of the reactor such as linings and baffles. With respect to the baffles, reference is made to D8 (page 2, line 118 to page 3, line 51). In the only example, the above mentioned additives are exclusively added to the oxygen stream. Although almost any introduction means of the additives in almost any possible combination was contemplated in D5, there is no specific disclosure of introducing one additive at different points in the reactor let alone to introduce a selected metal ion at a position upstream of the reaction zone **and** at a position where at least 20 wt.% of the titanium halide has been reacted. It is therefore insignificant whether the baffles as disclosed in D8 are situated at a position where one may assume that more than 20 wt.% of

the titanium halide has been reacted. Even if such a baffle contained a source of any of the additives according to present claim 1, which neither D5 nor D8 explicitly discloses, there is no disclosure to add the additive at the same time upstream of the reaction zone. Thus D5 does not destroy the novelty of claim 1 as granted. Neither of the other citations discloses in combination all the features of claim 1. This is no longer in dispute. The subject-matter of claim 1 is therefore novel within the meaning of Article 54(1) EPC.

2.2 Inventive step

2.2.1 The parties agreed that the process disclosed in US-A-3 208 866 as described on page 2, lines 9 to 19 of the patent in suit represented the relevant starting point for discussing inventive step. The Board sees no reasons to deviate from this common point of view. The pre-characterising portion of claim 1 is in agreement with said description of the prior art. According to the respondent the claimed process solved the problem of producing titanium dioxide pigment having desirable properties such as a uniform size, a high rutile content and a suitable CBU value with a process in which the particles suspended in the reactor effluent could be easily filtrated. The only factual basis for this submission are the comparative examples in the patent in suit and those filed with the letter dated 27 March 1998. In these examples the process of the patent in suit was, however, not compared with processes according to the state of the art but with experimental work made by the respondent. The experiments were made with a very specific reactor with two consecutive reaction chambers and it is not credible that the results are significant for the whole ambit of claim 1, which does not specify the type of reactor. The results of these experiments are thus not

suitable for proving that the stated problem had been credibly solved. Under these circumstances the Board considers that the problem underlying the invention should rather be seen in the provision of a further process for preparing rutile titanium pigment of a reasonable quality. According to claim 1 a solution of this problem is provided by a process comprising the introduction of the metal ion compound to the reactor in at least two increments, wherein the first increment is introduced at a point prior to the onset of the reactor and the additional other increments at a point subsequent to the reaction of at least 20 wt.% of the titanium halide. It is credible and uncontested that the process according to claim 1 actually solves the said problem.

2.2.2 It remains to be decided whether the claimed solution of this problem was obvious to a person skilled in the art in view of the available prior art documents.

D2, relating to a process for treating titanium dioxide, discloses the addition of alkali or alkaline earth metal ions in an amount of 0.01 to 5000 ppm TiO_2 to the hot effluent stream of a titanium halide oxidation reactor in order to reduce the surface energy of the pigment (page 1, line 46 to page 2, line 6 and page 3, lines 49 to 55). It is stressed that the additive should be added to the effluent in a second zone separate from and sufficiently remote from the reaction zone so as to ensure that none of additive is recirculated back into the reaction zone (page 3, lines 35 to 44). In the only example KCl is introduced at a point remote from the reactor exit with an auxiliary gas having a temperature of 142°F (61°C) resulting in a mixed gas stream with a temperature of 900°F (462°C). In the Board's opinion D2 actually dissuades the skilled person from adding any alkaline or alkaline earth metal compound to the reactor zone.

The Board cannot accept the appellant's position that the said second zone in D2, wherein the additive is introduced, is a part of the reaction zone within the meaning of present claim 1 because the effluent treated therein still contains unreacted titanium halide and oxygen. The Board does not contest that the effluent treated in D2 contains unreacted titanium halide and oxygen but that does not mean that a reaction takes place. For a reaction to take place the temperature and the concentration of the reactants must be above certain limits. As is evident from the example in D2, both temperature and concentration of the reactants in the effluent stream (dilution by a cool auxiliary gas) are substantially lower than in the reactor. If at the exit of the reactor, at temperatures above 1500°F (816°C), the reaction mixture still contains unreacted titanium halide no further reaction can be expected to take place in the subsequent treating zone at much lower temperature and concentration.

2.2.3 D10 discloses a process for the preparation of titanium dioxide by vapour phase oxidation of a titanium halide whereby solid, inorganic, water soluble salt in an amount of 0.01 to 5 % by weight of TiO_2 is introduced into the hot gaseous suspension of TiO_2 particles before or during cooling such suspension in order to reduce the forming of scale on the cooling surfaces. The salt can be added at any convenient point in the cooling system but is most conveniently added to the product stream as it discharges from the reactor or at any suitable point thereafter. The solid salts can be added at a plurality of addition points. According to the examples, KCl or an other salt is added at the beginning of the transfer line between the reactor and the first heat exchanger (see page 2, line 112 to page 3, line 102 and the examples). There is no indication in D10 to introduce the salt into the reaction zone. The Board cannot agree with the

appellant's submission that adding the salt to the product stream as it discharges from the reactor, as indicated in D10, is analogous to the point of addition described in the patent in suit at page 6, lines 3 to 5 (see point 39 of the grounds of the appeal). The patent in suit mentions in the sentence on page 6, lines 3 to 5 that the first increment was introduced into the oxidation reactor and mixed with the preheated oxygen reactant upstream of the first $TiCl_4$ inlet assembly and first reaction zone. In the following sentence it is said that the second increment of KCl was introduced into the first reaction zone of the reactor at a point immediately adjacent to the upstream side of the second $TiCl_4$ inlet assembly. The Board is unable to see any analogy between the addition points of the increments mentioned in the patent in suit, which unambiguously imply the introduction of the salt into the reactor, and the addition of the salt to the product stream discharged from the reactor as required by D10. Thus D10 provides no incentive for the claimed solution of the above mentioned problem.

2.2.4 The finding of the decision under appeal that the other citations do not disclose or suggest the introduction of an additive comprising alkali, alkaline earth or rare earth metal ions to the reactor at a point where a titanium halide has already been partly reacted with the oxygen, was no longer disputed (see point III above). Having regard to the documents on file representing the state of the art, the Board, therefore, holds that the process according to claim 1, independent of any technical improvement over the prior art, was not obvious to a person skilled in the art and thus involves an inventive step within the meaning of Article 56 EPC.

2.2.5 Claims 2 to 7 are dependent upon claim 1. The inventive step of the processes according to claims 2 to 7 follows from this dependency.

3. Since the main request is allowable, there is no reason to consider the auxiliary request.

Order

For these reasons it is decided that:

The appeal is dismissed.

The Registrar: The Chairman:

S. Hue R. Spangenberg