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D E C I S I O N
of 15 January 2003

Case Number: T 0424/99 - 3.3.5

Application Number: 92400171.2

Publication Number: 0498686

IPC: C09C 1/00

Language of the proceedings: EN

Title of invention:

Bismuth oxychloride nacreous pigment having improved weather fastness and the production thereof

Patentee:

ENGELHARD CORPORATION

Opponent:

Merck Patent GmbH

Headword:

-

Relevant legal provisions:

EPC Art. 52(1), 54, 56, 123(2), 123(3)

Keyword:

"Main and first subsidiary request: inventive step (no) - obvious alternative"

"Second subsidiary request: inventive step (yes) - solution not obvious"

Decisions cited:

-

Catchword:

-



Case Number: T 0424/99 - 3.3.5

D E C I S I O N
of the Technical Board of Appeal 3.3.5
of 15 January 2003

Appellant: ENGELHARD CORPORATION
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Respondent: Merck Patent GmbH
(Opponent) Postfach
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Representative: -

Decision under appeal: Decision of the Opposition Division of the
European Patent Office posted 10 February 1999
revoking European patent No. 0 498 686 pursuant
to Article 102(1) EPC.

Composition of the Board:

Chairman: R. K. Spangenberg
Members: A.-T. Liu
J. H. Van Moer

Summary of Facts and Submissions

I. European patent No. 0 498 686 was granted with a set of 10 claims, of which claim 1 was directed to a pearlescent pigment with claims 2 and 3 depending thereon and claim 4 directed to a process for preparing an improved weather fastness pearlescent pigment, with claims 5 to 10 depending thereon.

II. Claim 1 read as follows:

"Pearlescent pigment comprising bismuth oxychloride crystals, characterised in that said bismuth oxychloride crystals are coated with cerium hydroxide, to improve weather fastness of said pigment".

III. A notice of opposition was filed against the patent on the grounds of lack of novelty and lack of inventive step (Article 100(a) EPC) and essentially supported by the following document:

D1: DE-PS-1 003 377

IV. The present appeal was lodged against the decision of the opposition division to revoke the patent on the finding that, *inter alia*, the subject-matter of claim 1 was not novel with respect to the disclosure of D1.

V. With the statement of the grounds of appeal, the appellant filed two new sets of amended claims. Further arguments and results of comparative tests were submitted by letter of 14 February 2000.

VI. The first subsidiary set of claims consisted of 9 claims, of which claim 1 had the same wording as

claim 1 as granted. The second subsidiary set of claims consisted of claim 1, directed to a pearlescent pigment with claim 2 depending thereon, and independent claim 3 directed to a preparation process, with claims 4 to 9 depending thereon. The independent claims read as follows:

"1. Pearlescent pigment comprising bismuth oxychloride crystals, characterised in that said bismuth oxychloride crystals are coated with from 1 to 7.5 wt% of cerium hydroxide, based on total weight of said pigment, to improve weather fastness of said pigment.

3. Process for preparing an improved weather fastness pearlescent pigment comprising bismuth oxychloride crystals, characterised in that it comprises the steps of:

- providing a solution of a cerium salt,
- providing a suspension of bismuth oxychloride pigment crystals,
- combining the cerium salt solution and the bismuth oxychloride suspension, and
- precipitating and coating the bismuth oxychloride crystals with from 1 to 7.5 wt% of cerium hydroxide."

VII. A third subsidiary set of claims was filed at the oral proceedings on 15 January 2003.

VIII. The appellant's submissions were essentially the following:

- D1 did not disclose pigments "coated with cerium hydroxide".

- The technical problem addressed in the patent in suit was that of "weather fastness", which was different from that of D1.
- The results of comparative tests showed that the products according to D1 did not solve the present technical problem.

IX. The respondent's arguments may be summarised as follows:

- BiOCl pigments coated with cerium hydroxide were disclosed in D1.
- D1 concerned the technical problem of making BiOCl pigments resistant to light and temperature, which was the same problem as addressed by the patent in suit.
- The stipulated amount of 1 to 7.5 wt% of added cerium hydroxide was obvious in view of the general teaching of D1.

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

The appellant (patentee) requested that the decision under appeal be set aside and that the patent be maintained as granted (main request) or, in the alternative, on the basis of any of the subsidiary requests 1 to 3.

The respondent (opponent) requested that the appeal be dismissed.

Reasons for the Decision

1. *Main request*

Claim 1 of this request is directed to a pearlescent pigment, essentially defined in that the pigment particles comprising bismuth oxychloride crystals are coated with cerium hydroxide.

1.1 Novelty

1.1.1 It is undisputed that D1 also concerns a pearlescent pigment comprising bismuth oxychloride crystals to which a cerium salt has been added (example 3b). In the contested decision, the opposition decision took the view that, since BiOCl is a base, the process disclosed in example 3b of D1 will result in the base catalysed hydrolysis of the dissolved cerium salt. This process will thus result in bismuthoxychloride particles encapsulated with cerium hydroxide (see point 5.1 of the decision).

1.1.2 An explanation for the reaction that would presumably take place in the process of D1 has been given by the respondent with reference to common general knowledge. Thus, it has been submitted that the cerium salt solution contains hydrogen peroxide which, being a strong oxidation agent, will convert the soluble trivalent cerium salt to its tetravalent state. Furthermore, the solution of cerium trichloride is only slightly acidic (with a pH of 6.5) while the bismuthoxychloride suspension is basic. Since tetravalent cerium salt is less soluble and cerium hydroxide is formed at a pH slightly above 7, the

cerium salt will be hydrolysed immediately to cerium hydroxide upon its mixing with the bismuthoxychloride suspension and cerium hydroxide precipitated onto the bismuthoxychloride particles.

- 1.1.3 The appellant has strongly contested the finding of the opposition division and the interpretation of D1 given by the respondent. In his view, one can only derive from D1 that the cerium salt is adsorbed as such onto the surface of the bismuthoxychloride particles, without its being converted into cerium hydroxide.

- 1.1.4 The Board notes that the respondent has not put forward a particular pH value for the bismuthoxychloride suspension, let alone provided any evidence therefor. On the other hand, the appellant's argument that the addition of hydrogen peroxide, if anything, would lower the pH of the cerium salt solution rather than raising it, remains unchallenged. Thus, there is no indication on file permitting the skilled person to presume that the pH value of the mixture will be above 7. According to the respondent's submissions at the oral proceedings, the precipitation of cerium hydroxide from cerium salts not only depends on the pH of the reaction medium but also on the temperature, the duration of the reaction and last but not least on the anion involved. The respondent, however, has not indicated which of these conditions is met in the process of D1 which would necessarily lead to a precipitation of the cerium hydroxide. On the other hand, in example 3b of D1, the reaction conditions are such that BiOCl is suspended in an (acidic) solution of the cerium salt and directly filtered without prior washing. It is thus doubtful whether such reaction conditions particularly aim at hydrolysing the cerium salt. In the absence of any

convincing argument, let alone conclusive evidence, the Board therefore decides in favour of the appellant that D1 does not clearly and unambiguously disclose a process in which cerium hydroxide is precipitated from a cerium salt solution.

1.1.3 None of the other documents on file discloses cerium hydroxide incorporated on bismuthoxychloride. As a consequence, the subject-matter of claim 1 is new.

1.2 Inventive step

1.2.1 It is common ground that the closest prior art is represented by D1.

1.2.2 With respect to D1, the problem that the patent in suit has set out to solve is the provision of a bismuth oxychloride pigment having improved weather fastness (see patent in suit, column 1, lines 32 to 35). The appellant has asserted that this problem is not the same as the objective to be achieved in D1, which is to improve the resistance of bismuth oxychloride pigment to light and temperature (see also Statement of the grounds of appeal).

According to the patent in suit, the term "weather fastness" is "meant to include weather resistance and light stability" (column 1, lines 26 to 28). On the other hand, it is clearly stated in D1 that the disclosed bismuth oxychloride pigments which are resistant to temperature and light find applications in paint formulations (column 1, lines 5 to 12). Since paint formulations are expected to be exposed to weather conditions, the Board holds that the temperature resistance is essentially meant with

respect to weather conditions and that both D1 and the patent in suit essentially address the same problem. Under these circumstances, the Board can see the technical problem to be solved with respect to D1 in the provision of a further BiOCl pigment with substantially the same properties.

- 1.2.3 To solve the technical problem as stated above, it is proposed in claim 1 that the pigment be coated with cerium hydroxide. The Board has no doubt that the stated problem is solved by the pigment as claimed.
- 1.2.4 The appellant has submitted that the presence of cerium hydroxide is not the only differentiating feature in claim 1. He has argued that, in addition, the stipulation that the pigment crystals be "coated" means that these crystals are entirely encapsulated by the cerium hydroxide which has been precipitated onto their surface. In contrast thereto, in the process of D1, the cerium salt is merely adsorbed and not precipitated onto the surface the pigment particles. Moreover, the small amount of cerium salt used in D1 would imply a doping of the pigments particles and not a coating of these crystals.

The Board cannot concur with the appellant and wishes to observe that claim 1 does not stipulate the amount of cerium hydroxide for coating. As was established at the oral proceedings, the interpretation tentatively given by the appellant to the term "coated with" as standing for "entirely encapsulated by" is neither explicitly given in the patent in suit nor derivable from its disclosure. In fact, the appellant has conceded that a BiOCl pigment particle, whose surface is entirely enrobed by cerium hydroxide (or any cerium

salt) would lose at least some of its pearlescent characteristics. Claim 1 can therefore only be interpreted as stipulating an undefined amount of cerium hydroxide for coating. As is further advanced by the respondent and not refuted by the appellant, if cerium hydroxide is to coat the pigment particles, it will also have to adhere to the surface of these particles, otherwise it will precipitate as a separate solid phase. The fact that D1 also discloses the added salt component to be "attached" onto the pigment crystals is not disputed by the appellant (column 1, lines 15 to 17 and letter of 9 June 1999, page 3, paragraphs 4 and 5). In the Board's judgment therefore, claim 1 has to be interpreted in its broadest sense as stipulating BiOCl crystals having cerium hydroxide incorporated (or "attached") onto their surface. Since the amount involved is undefined, it encompasses such proportions as are used in D1.

- 1.2.5 The Board therefore holds that the question to be posed here is whether the incorporation of cerium hydroxide is obvious in view of D1. The Board notes that it is explicitly indicated in that document that resistance to temperature and light is obtained for BiOCl pigments by the incorporation of metal salts in which the cations are capable of changing their oxidation state, with the higher oxidation state being at least trivalent (column 1, lines 13 to 19; claims 1 and 10). In the Board's judgment, the teaching one can deduce from that statement is that the desired stabilisation is imparted by the cations whilst the anions apparently do not contribute to this effect. Furthermore, the appellant has not contested the fact that "cerium hydroxide" is also a "cerium salt" and thus envisaged to be used as additive in D1. In terms of a further

stabilised BiOCl pigment, the Board thus holds that the alternative as proposed in claim 1 is the result of a trivial choice and therefore obvious in view of D1.

1.2.6 Taking the examples of the patent in suit into consideration, the Board, however, notes that two products comprising the claimed pigments are exposed to testing for a period of 8 weeks or 12 months (examples 2 and 1, respectively). Furthermore, the Board can derive from the comments with respect to the comparative tests submitted by letter of 14 February 2000 that the appellant only considers those test results obtained after a certain length of exposure as significant. In view of these submissions, the Board holds that the technical problem can also be seen in the impartation of an improved long-term weather fastness with regard to the bismuth oxychloride pigments of D1. The Board has therefore taken this technical problem into consideration.

1.2.7 The Board notes that the appellant has repeated the experimental conditions of example 3b of D1 and exposed the obtained powder to ultra-violet light. The test results show that, whilst the procedure of D1 did provide some improvement in light fastness with regard to a BiOCl not having any cerium added, that improvement is however lost after an hour of exposure (see letter dated 14 February 2000, page 3, item II and the annexed graph). The appellant has not argued, let alone provided evidence that a different result would be obtained if cerium hydroxide instead of a "cerium salt" were used as additive in D1.

As is discussed above, one can derive from D1 that the stabilisation of the BiOCl pigments is imparted by the

cations (see point 2.1.5). In view of this teaching and due to lack of proof to the contrary, the Board deduces from the experimental results submitted by the appellant that the desired long-term effect will not be achieved with BiOCl coated with the same proportion of cerium hydroxide. The conclusion to be drawn is that the technical problem as stated in point 2.1.6 is not solved over the whole range of claim 1.

1.2.8 As corollary of the above, the implication of an inventive step in claim 1 must be denied.

2. *Auxiliary request 1*

The subject-matter of the present claim 1 is the same as that of claim 1 of the main request. The above result therefore applies mutatis mutandis to the present claim. Consequently, neither request is allowable due to lack of inventive step (Article 56 EPC).

3. *Auxiliary request 2*

3.1 Claims 1 and 3 of the present request are based on claim 2 and on claim 4 in combination with claim 2 as granted, respectively. The dependent claims 2 and 4 to 9 correspond to granted claims 3 and 5 to 10, respectively. The basis in the original documents for the claims as granted has never been queried. The Board is therefore satisfied that the requirements of Article 123(2) and (3) EPC are met.

3.2 The subject-matter of present claim 1 differs from that of the previous requests in that it stipulates that cerium hydroxide be incorporated in an amount from 1 to

7.5 wt%, based on the total weight of the pigment.

3.3 Examples in the patent in suit show that the technical problem as stated in point 2.1.6 above, namely the long-term effect, is obtained with a coating of cerium hydroxide in the stipulated amount. This is not in dispute. The only question is whether this solution is obvious in view of the available prior art.

3.4 It is irrefutable that the amount of cerium used in example 3b of D1 is 0.04 weight percent, based on the weight of the bismuth oxychloride crystals. It is true that D1 mentions that the light and temperature-fastness of the pigment may be further increased by repeatedly carrying out the step of adding the metal salt(s). The final amount of additives in the pigment is however indicated to be at a level of 0.1% (column 1, lines 35 to 36 and column 2, lines 36 to 43). Compared to this amount of added cerium salt, be it as expressedly used in example 3b or according to the general teaching of D1, the amount of 1 to 7.5 wt% of cerium hydroxide (based on the total weight) as stipulated in claim 1 is not only slightly higher but on a different scale. In the Board's judgment, the claimed range is therefore not envisaged by D1. On the other hand, D1 does not discuss the problem of long-term weather fastness. It thus cannot give the skilled person incentive for optimising the amount of additive with that aim in mind. In addition to that, one should bear in mind that the skilled person will always endeavour to incorporate as little additive as possible, otherwise it may be detrimental to the pearlescence of the pigment (see letter of the respondent dated 3 November 1999, page 3, paragraph 4). Using the teaching of D1, the skilled person will

therefore not arrive at the stipulated value merely by trial and error.

- 3.5 The respondent has not argued and the Board cannot find that the stipulated amount of cerium additive as proposed in claim 1 is suggested in any of the other prior art documents on file.
4. The process according to claim 3 leads to bismuth oxychloride crystals coated with the same amount of cerium hydroxide as stipulated in claim 1. The dependent claims 2 and 4 to 9 are directed to preferred embodiments of the pigment or of that process, respectively. The subject-matter of these claims is also new and involves an inventive step. The patent can thus be maintained with the claims of the present request.

As was established at the oral proceedings, the coating of BiOCl crystals with the cerium compound in an amount of 1 to 7.5 wt% is referred to in the description as part of the invention and not as a preferred embodiment (see patent in suit, column 3, lines 3 to 5). The Board therefore concurs with the parties that no further adaptation of the description is necessary in the present case.

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 with the following documents:

- claims 1 to 9 (second auxiliary request)
- description as granted.

The Registrar:

The Chairman:

U. Bultmann

R. Spangenberg