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Bezeichnung der Erfindung: Process for preparing acicular iron oxide
Title of invention: particles
Titre de l'invention :

Klassifikation / Classification / Classement : C 01 G 49/02

ENTSCHEIDUNG / DECISION

vom / of / du 11 September 1987

Anmelder / Applicant / Demandeur : Eastman Kodak Company

Patentinhaber / Proprietor of the patent /
Titulaire du brevet :

Einsprechender / Opponent / Opposant :

Stichwort / Headword / Référence : Iron oxide particles/Eastman Kodak

EPO/EPC/CBE Article 56

Kennwort / Keyword / Mot clé : "Inventive step"

Leitsatz / Headnote / Sommaire

Europäisches
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European Patent
Office

Boards of Appeal

Office européen
des brevets

Chambres de recours



Case Number : T 77/86

D E C I S I O N
of the Technical Board of Appeal 3.3.1
of 11 September 1987

Appellant : Eastman Kodak Company
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Decision under appeal : Decision of Examining Division 025
of the European Patent Office
dated 13.09.1984 refusing European
patent application No. 81 401 165.6
pursuant to Article 97(1) EPC

Composition of the Board :

Chairman : K. Jahn
Members : R. Andrews
R. Schulte

Summary of Facts and Submissions

I. European patent application No. 81 401 165.6, filed on 22 July 1981 and published on 3 February 1982 (publication No. 0 045 246), was refused by a decision of the Examining Division 025 of the European Patent Office dated 13 September 1984. The decision was based on original Claims 1 to 23. The only independent Claim reads as follows:

"In a process for preparing acicular magnetic iron oxide particles comprising (a) adding an aqueous solution of ferrous salt to an aqueous solution of a stoichiometric excess of alkaline hydroxide while substantially avoiding local excesses of said ferrous salt, to form an aqueous dispersion of ferrous hydroxide particles, (b) oxidizing said particles to form non-magnetic alpha ferric oxide hydrate particles, and (c) converting said non-magnetic particles to magnetic iron oxide particles by dehydration and reduction or dehydration, reduction and oxidation, the improvement wherein said ferrous salt solution is subjected to reducing conditions prior to or during said addition to reduce contaminating ferric ions present therein to ferrous ions".

II. The stated ground for the refusal was that the subject-matter of the application did not involve an inventive step in the light of the disclosure in GB-A-1 328 115 (1). This document discloses a process for the preparation of acicular alpha ferric oxide hydrate (goethite) comprising the present steps (a) and (b) wherein step (a) is carried out under substantially non-oxidising conditions. In the opinion of the Examining Division the technical problem to be solved in both document (1) and the application-in-suit was to keep the ferrous salt solution free of contaminating ferric ions during the precipitation step. Since it is

common general knowledge that ferrous salts are sensitive to oxidation and that contamination of ferrous salt solutions by ferric ions is generally avoided by adding reducing agents, it was concluded that the subject-matter of Claim 1 and the dependent Claims 2 to 23 did not involve an inventive step.

- III. An appeal was lodged against this decision on 15 November 1984 with payment of the appropriate fee. The Statement setting out the grounds of appeal was filed on 4 January 1985.

The Appellant argued that the invention lay in recognising that it was critical in step (a) of the process not only to prevent the formation of additional ferric ions but also to remove the ferric ions present in the initial ferrous salt solutions. Furthermore the Appellant claimed that the use of reducing conditions during step (a) of the process resulted in a reduction in the oxidation times needed to convert the ferrous hydroxide to alpha ferric oxide hydrate and in the stoichiometric excess of alkaline hydroxide required in step (a). It was also possible to prepare suspensions in which the concentration of alpha ferric oxide hydrate particles was substantially in excess of the maximum of 15g/l disclosed in document (1). Moreover due to the use of low excesses of alkaline hydroxide acicular magnetic oxide particles of very short lengths and low acicularity ratios could be produced.

- IV. The Appellant requests that the decision under appeal be set aside and a patent be granted on the basis of the original Claims 1 to 23.

Reasons for the Decision

1. The appeal complies with Articles 106 to 108 and Rule 64 EPC and is, therefore, admissible.
2. The application relates essentially to a process for preparing acicular magnetic iron oxide particles comprising
 - (a) forming an aqueous dispersion of ferrous hydroxide particles,
 - (b) oxidising the said particles to non-magnetic alpha ferric oxide hydrate particles and
 - (c) converting the product of step (b) to magnetic iron oxide particles by dehydration and reduction or dehydration, reduction and oxidation.

Such a process is known from US-A-3 843 773 (2). However, this prior art process was unsatisfactory in that it was necessary to use an excess of approximately 200 % of alkaline hydroxide with respect to the stoichiometric quantity necessary to precipitate the ferrous hydroxide particles in step (a) (cf. Col. 2, lines 42 to 49) and it was only possible to obtain suspensions in step (b) in which the concentration of non-magnetic alpha ferric oxide hydrate was less than 15g/l (cf. Claim 1 and Col. 3, lines 13 to 16). This prior art process produces alpha ferric oxide hydrate particles whose lack of homogeneity in size and shape give rise to inferior magnetic recording and reproducing properties in the magnetic iron oxide produced from such particles. Furthermore, cubic crystals of alpha ferric oxide hydrate are formed if stoichiometric excess of

alkaline hydroxide used in step (a) of this prior art process is 300% or less (cf. Examples 8A and 8B and Figures 1/2 and 2/2 of the present application). Document (1) contains the identical teaching with regards to steps (a) and (b) (cf. Claim 1 and page 1, lines 80 to 89).

3. In the light of this closest prior art the technical problem underlying the application-in-suit can be seen in avoiding the above-mentioned disadvantages and providing an economically improved process for preparing acicular magnetic iron oxide particles without affecting their magnetic recording and reproducing properties.

According to the patent application this problem is solved by subjecting the ferrous salt solution used in step (a) to reducing conditions prior to or during its addition to the aqueous solution of alkaline hydroxide.

In view of the results in Examples 2, 4, 5, 6 and 10 of the application in which the excess of sodium hydroxide in step (a) is 90%, 85%, 62%, 20% and 100% and the concentration of alpha ferric oxide hydrate at the end of step (b) was 30 g/l, 20 g/l, 40 g/l, 25 g/l and 20 g/l respectively, Examples 8A and 8B and page 24, lines 3 to 23, the Board is satisfied that this technical problem is plausibly solved.

4. After examination of the cited documents the Board has concluded that this technical teaching is not disclosed in any of them and the claimed subject-matter is, therefore, novel.
5. It still remains to be examined whether the requirement of inventive step is met by the claimed subject-matter.

5.1 Document (2) discloses a process for preparing acicular crystals of gamma ferric oxide by reacting, under non-oxidising conditions, an aqueous solution of ferrous salt with an aqueous solution of a stoichiometric excess of an alkaline hydroxide, introducing oxygen into the dispersion of ferrous hydroxide particles thus obtained, boiling the resulting dispersion containing less than 15 g/l of alpha ferric oxide hydrate and subjecting the recovered crystals of alpha ferric oxide hydrate to dehydration, reduction and oxidation to convert them to gamma ferric oxide crystals.

From the teaching of this document the skilled person would conclude that it was considered necessary to maintain the solutions and ambient atmosphere free of oxidising substances to prevent the formation of ferric ions during the addition of ferrous salt solution to the aqueous solution of alkaline hydroxide. By employing these non-oxidising conditions the formation of heterogeneous crystals which later lead to crystals the magnetic characteristics of which differ from the desired optimum was avoided (cf. Col. 3, lines 22 to 27). However there was absolutely no recognition in this prior art of the fact that even the very low concentrations of ferric ions that are normally present in ferrous salt solutions obtained by dissolving commercial grades of ferrous salts in water would detrimentally effect the magnetic recording and reproducing properties of the acicular magnetic iron oxide particles produced later in the process.

In view of the fact that substantially non-oxidising conditions are not equivalent to reducing conditions, a skilled person would not have been led to replace the non-oxidising conditions of the prior art by reducing conditions in an attempt to solve the technical problem underlying the application-in-suit. If, in the light of his

common general knowledge of the sensitivity of ferrous salt solutions to oxidation, the skilled person were, nevertheless, to make this replacement, he would not have done so in the expectation of solving the technical problem as defined above (cf. T 2/83, OJ 1984, page 265, particularly point 7 on page 270).

Therefore, in the Board's view the teaching of this prior art document or common general knowledge would not give any indication to the skilled person that the solution to the technical problem as defined above lay in replacing the non-oxidising conditions in step (a) of the prior art process by the reducing conditions of the present process.

- 5.2 Document (1) discloses a process for preparing acicular alpha ferric oxide hydrate comprising the steps (a) and (b) disclosed in document (2) (cf. Claim 1). In this document it is disclosed that this process may be further improved by conducting the solution of ferrous salt to an aspiration zone situated within the alkali metal hydroxide solution, whereby the ferrous salt solution is drawn out as it enters the alkali metal hydroxide solution, into a thin layer and penetrates the alkali metal hydroxide solution in a sheet form (cf. Claim 8). However, this additional technical teaching with regard to the rapid dispersion of the ferrous salt solution and the avoidance of any localised excess of ferrous salt together with the use of a large stoichiometric excess of alkali metal hydroxide in step (a) and a reduction of the oxidation time necessary in step (b) (cf. page 3, lines 76 to 83 and Examples IV and V) would not be of any assistance to the skilled person in his search for a solution to the above defined technical problem.

- 5.3 Therefore, it is concluded that the subject-matter of Claim 1 involves an inventive step. Dependent Claims 2 to 23, which relate to preferred embodiments of Claim 1, derive their patentability from this claim.
6. However, the patent sought cannot be granted since the present Claim 1 is not properly drafted in the two-part form required by Rule 29(1) EPC. Therefore, the Board makes use of its powers under Article 111(1) EPC to remit the case to the Examining Division for further prosecution.

Order

For these reasons, it is decided that:

1. The decision under appeal is set aside.
2. The case is remitted to the Examining Division for further prosecution on the basis of Claims 1 to 23 as originally filed.

The Registrar:



The Chairman:

