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(54) Titre : PROCEDE DE FABRICATION D'UN GERME D'OXYDE DE FER CONTENANT DE L'ALUMINIUM
(54) Title: METHOD FOR PRODUCING AN IRON OXIDE NUCLEUS CONTAINING ALUMINIUM

(57) Abrégé/Abstract:
The invention relates to a method for producing an iron oxide nucleus containing aluminium comprising a FeOOH crystal structure made of FeCl2. Said nucleus is suitable as a base material for producing iron oxide yellow and for use as a yellow coloured pigment.

Abstract

The invention relates to a method for producing an iron oxide nucleus containing aluminium comprising a FeOOH crystal structure made of FeCl2. Said nucleus is suitable as a base material for producing iron oxide yellow and for use as a yellow coloured pigment.

Method for producing an aluminum-containing iron oxide nucleus

The present invention relates to a method for producing an aluminum-containing iron oxide nucleus having an α-FeOOH crystal structure from $FeCl_2$. This nucleus is suitable as a starting material for the preparation of iron oxide yellow and for use as a yellow colored pigment.

Synthetic iron oxides are usually prepared by the Laux process, the Penniman process, the precipitation process, the neutralization process or the roasting process (Ullmann's Encyclopedia of Industrial Chemistry, 5th edition, 1992, Vol A20, page 297 et seq.). The iron oxides thus obtained are generally used as pigments.

For the preparation of finely divided α-FeOOH (needle width from 5 to 30 nm), there are in principle two known processes:

- the acidic process
- the alkaline process

In the acidic process, an iron(II) component, i.e. an iron salt dissolved in water, is initially introduced and an alkaline component, as a rule an alkali metal compound or an alkaline earth metal compound dissolved or suspended in water, or ammonia solution, is metered into said iron(II) component with thorough mixing. The amount of alkaline component metered in is as a rule from 15% to 70% of the stoichiometrically required amount. The pH after addition of the alkaline component is in the weakly acidic range.

After the end of the addition of the alkaline component, oxidation is effected with an oxidizing agent, as a rule atmospheric oxygen. The reaction is carried out at temperatures of from 20°C to 50°C. At substantially higher temperatures, there is the danger of formation of undesired magnetite. The end point of the reaction can be detected from a sharp drop in the pH and of the redox potential. After the end of the

reaction, the properties of the product obtained (generally referred to as nucleus) are determined and said product, if suitable, is immediately further processed to give the α-FeOOH pigment.

The alkaline process differs from the acidic process in the amount of the alkaline component metered in. In the alkaline process, this is at least 120% of the stoichiometrically required amount, but as a rule substantially more. The temperatures at which this reaction is carried out may be slightly above the temperatures used in the acidic process, since there is less danger of magnetite formation here.

In principle, relatively long-needle α-FeOOH crystallites having a length-to-width ratio of 10:1 to 30:1 are obtained in the alkaline process. Since, moreover, these crystallites have a very low dendrite content, this process is particularly suitable for the preparation of α-FeOOH as a starting material for magnetic tapes.

The nuclei produced by the alkaline process can be used directly only to a limited extent, if at all, for the preparation of α-FeOOH pigments for use in paints and finishes, since all color-imparting metals present in the Fe component are incorporated in this process. These metals (in particular Mn, Cr, Cu, Ni) have a substantial adverse effect on the color properties and thus permit the use of nuclei produced in this manner as colored pigments.

For the preparation of iron oxide yellow pigments, an α-FeOOH nucleus is preferably used, and this is then rendered coarser (built up) in the acid, with the result that the incorporation of the color-imparting metals is reduced. Furthermore, the synthesis may be effected only at a pH of less than about 4, since at higher pH the color-imparting metals are incorporated to an increasing extent. Furthermore, the particle shape of the α-FeOOH has a considerable influence on the color properties, the viscosity of the finish and the binder requirement.

In order to achieve a desired low viscosity in the finish and a low binder requirement, short-needle α-FeOOH particles are required. These can be prepared from long-needle α-FeOOH particles by thorough milling. A more economical alternative is the direct preparation of short-needle α-FeOOH particles.

Modifying additives are required for controlling the particle shape of the α-FeOOH nucleus and hence that of the pigment synthesized therefrom to obtain a low length-to-width ratio. US-A 4 620 879 discloses the use of B, Al, Ga, Si, Ge, Sn or Pb as nucleus modifiers. This patent describes an iron oxide yellow having a particularly low silking index, which is achieved by an appropriate procedure in the pigment synthesis and by the addition of the abovementioned modifiers.

Although the use of $FeCl_2$ is described in this specification, the exact conditions for producing an α-FeOOH nucleus from $FeCl_2$ are not stated. Since, however, $FeCl_2$ differs considerably from $FeSO_4$, particularly in the nucleus-formation phase, the conditions under which a good pigment is obtained using $FeSO_4$ are not applicable to $FeCl_2$.

It was the object of the present invention to provide a method for the simple and economical production of a short-needle α-FeOOH nucleus by the precipitation process. In a further step, an α-FeOOH pigment is synthesized from this α-FeOOH nucleus.

This object is achieved by the method according to the invention: this is a method for producing aluminum-containing iron oxide nuclei having an α-FeOOH crystal structure having an aspect ratio of 2 100 to 3 600 and a BET surface area of 50 to 150 m2/g. In the present context, the aspect ratio is the product of BET surface area and mean crystallite size, which was determined by X-ray diffraction from the 110 reflection of α-FeOOH. These very finely divided α-FeOOH nuclei can be prepared by the following method using $FeCl_2$:

a.) First, an Al component is added in amounts of 4 - 13 mol%, based on total Fe, while stirring, to an iron(II) chloride solution having a total Fe content of 20 - 100 g/l, preferably 40 - 65 g/l, and an Fe(III) content of 0.1 - 10 mol% of Fe(III) (based on total Fe),

b.) this mixture is heated to a precipitation temperature of from 30°C to 60°C, preferably 35 to 50 degrees,

c.) a precipitating agent having an active substance content of 2 - 10 equivalents per liter, preferably 4 - 8 equivalents per liter, is added to the mixture, and the molar ratio of Fe + Al to the precipitating agent is 20% - 80%, preferably 30% - 60%, of the stoichiometry,

d.) the precipitated suspension is then oxidized with an oxidizing agent at a rate such that the oxidation rate is 2 - 50 mol%/h, preferably 10 - 35 mol%/h, of the iron to be oxidized and

e.) the Al-containing α-FeOOH nucleus obtained after the oxidation are, if required, isolated.

If desired, the Al-containing α-FeOOH nucleus obtained after the oxidation can be used, without further isolation, after testing of the properties, for the preparation of iron oxide yellow pigments.

Preferably, the following procedure is adopted:

Starting chemicals:

$FeCl_2$ solution having an Fe content of 55g/l of Fe, including 1.5 mol% of Fe(III)

$AlCl_3$ solution

NaOH solution having an NaOH content of 300 g/l = 7.5 equivalents of NaOH / l

Al/Fe ratio: 12 - 13

Ratio of Fe + Al / precipitating agent: 35- 40 %

Reaction conditions:

Temperature: 44° C

Oxidation rate: 30 - 35 mol% of Fe(II)/h

$AlCl_3$ (as an aqueous solution) is preferably used as the Al component. The use of Si or Ti as a nucleus modifier, in the form of their chlorides, is likewise possible but requires a greater technical effort in the production.

Precipitating agents used may be NaOH, KOH, Na_2CO_3, K_2CO_3, $Mg(OH)_2$, MgO, $MgCO_3$, $Ca(OH)_2$, CaO, $CaCO_3$, NH_3 or secondary or tertiary aliphatic amines in aqueous solution or aqueous suspension.

Oxidizing agents used are atmospheric oxygen, oxygen, ozone, H_2O_2, chlorine, nitrates of the alkali metals or alkaline earth metals or NH_4NO_3.

If the iron(II) chloride solution used contains relatively large amounts of color-imparting metals which can be precipitated at a pH of less than 4, they can be precipitated by adding an alkaline component to the iron(II) chloride solution up to pH 4. The resulting solid can be separated from the supernatant clear purified solution by sedimentation, filtration or centrifuging. In addition to the undesired color-imparting metals, Fe(III), which has a considerable undesirable effect on the reaction to give the α-FeOOH nucleus, is also removed here (from addition of black magnetite).

The reaction is carried out in batchwise or continuous stirred vessels, in stirred vessel cascades, loop reactors or stirrer-free reactors having binary nozzles as mixing elements.

After preparation of the α-FeOOH nuclei according to the invention, these are converted into a pigment, which is effected by conversion of the nucleus particles into coarser particles in a manner known per se (pigment synthesis). However, since the α-FeOOH nuclei according to the invention are not used as such, it is necessary to describe the pigment synthesis to give an iron oxide yellow pigment.

The Al-containing nucleus produced by the method according to the invention is pumped into a solution of $FeCl_2$ or $FeSO_4$ or another Fe(II) salt. 7 - 15 mol of Fe(II) salt in the form of a solution having an Fe content of 30 - 100 g/l of Fe are added per mole of FeOOH in the nucleus. Although the addition of Fe(II) salt also leads to α-FeOOH yellow pigments, the brightness decreases with increasing amount of added Fe(II) salts, which as a rule is undesired. The suspension is then heated to the reaction temperature, which is from 50°C to 90°C. After the precipitation temperature has been reached, oxidation and precipitation are begun simultaneously. As a rule, atmospheric oxygen is added via a suitable gassing means and the pH of the suspension is regulated using an alkaline precipitating agent. The pH is regulated in a range from 2.4 to 4.8. The oxidation rate should be from 0.5 to 8 mol% of Fe(III)/h.

After the end of the reaction (i.e. when all Fe(II) has been oxidized), the solid formed is separated off by filtration. It is washed salt-free and can then be dried.

The yellow pigment prepared using this method is distinguished by high color purity, virtually isometric particle shape, low oil number and high chemical purity. All of its properties together make it particularly suitable for:

- Use in the area of paints and finishes

- Applications as raw materials for catalysts
- Applications in the area of food coloring
- Applications in the area of paper coloring
- Applications in the area of polymer coloring
- Applications as UV stabilizer
- Applications in the area of high-quality building materials (renders, etc.)
- Applications in the area of emulsion paints

Owing to the economical raw materials and the high production rate in the yellow pigment production, this method is particularly economical. Owing to the particular reaction procedure and the use of an exactly specified nucleus, it is possible reliably to prepare particularly high-quality yellow pigments which have advantageous performance characteristics compared with pigments prepared by other methods. Environmentally relevant chemicals are not used in the preparation, according to the invention, of the yellow pigments.

In the preferred embodiment (use of $FeCl_2$, $AlCl_3$, NaOH and air as starting materials), a virtually closed material circulation is possible through electrolysis of the NaCl obtained as a byproduct. The sodium hydroxide solution obtained here can be used directly again in the process. The products H_2 and Cl_2 formed in the chloroalkali electrolysis can be converted into HCl, which in turn then serves for pickling the steel sheets. This particularly environmentally friendly technology is not possible at present with $FeSO_4$ since the electrolysis of Na_2SO_4 does not operate satisfactorily.

Description of the methods of measurement used

1. Measurement of the BET surface area

The BET surface area is determined by the so-called 1-point method according to DIN 66131. The gas mixture used comprises 90% of He and 10% of N_2 and measurement is effected at 77.4 K. Before the measurement, the sample is heated at 140 °C for 60 minutes.

2. X-ray diffraction measurement of the crystallite size

The determination of the crystallite size is carried out on a Phillips powder diffractometer. The 110 reflection is used for determining the crystallite size.

Hydrated α-iron oxide (M(FeOOH) = 88.9 g/mol

2.1 Range of use

Determination of the crystallite size in goethite in the range from 5 to 100 nm.

2.2 Basis

The determination in goethite is carried out by means of X-ray diffractometry by detection of reflections. The evaluation is carried out using silicon as an external standard.

2.3 Reagent

Silicon standard for angle calibration (ICDD No. 27-1402),

Philips PW 1062/20

2.4 Apparatus

2.4.1	Diffractometer:	Philips PW 1800 goniometer Type: Theta - 2 theta
2.4.2	Sample feed:	21-fold sample changer
2.4.3	Detector:	Xe proportional counter
2.4.4	Evaluation of reflections:	X-Pert software Rev. 1.2 on HP Vectra VL
2.4.5	Agate mortar and pestle	
2.4.6	Sample holder:	Philips PW 1811/00 and PW 1811/27

2.5 X-ray diffraction conditions

2.5.1	X-ray tube:	Long fine focus, Cu anode, 60 kV, 2200 W
2.5.2	Radiation:	$CuK\alpha_1$, $\lambda = 0.154056$ nm
2.5.3	Generator:	40 kV, 40 mA
2.5.4	Scan parameters:	
2.5.4.1	Scan type:	Step scan
2.5.4.2	Step size:	0.020° 2 theta
2.5.4.3	Step measuring time:	2.00 s

2.5.5 Silicon standard:

2.5.5.1 Low angle side: 27.00° 2 theta

2.5.5.2 High angle side: 30.00° 2 theta

2.5.6 Sample:

2.5.6.1 Low angle side: 18.50° 2 theta

2.5.6.2 High angle side: 23.50° 2 theta

2.6 Procedure

2.6.1 External standard:

2.6.1.1 Introduce silicon standard (2.1) into the sample holder of the diffractometer and start the measuring program.

2.6.1.2 Determine the maximum and the FWHM of the silicon reflection with the Miller indices hkl = 111 in the 2 theta angle range 27.00° to 30.00°. Print out the peak parameters (Tab. 1) and, if required, the diffraction pattern.

2.6.2 Determination of the sample:

2.6.2.1 Triturate about 2 g of sample in the agate mortar (4.5).

2.6.2.2 Introduce about 1 g of sample into the sample holder (4.6) of the diffractometer and start the measuring program.

2.6.2.3 Determine the maximum and the integral width of the goethite reflection with the Miller indices hkl = 110 in the 2 theta angle range from 18.50° to 23.50°. Print out the peak parameters (Tab. 2) and, if required, the diffraction pattern.

2.7 Calculations

2.7.1 In the crystallite size determination table displayed by the computer (X'Pert software, Rev. 1.2, (Philips Analytical GmbH, Kassel, DE) Profile Widths), enter the integral width (width of broadened profile), the maximum (peak position/° 2 theta) of the goethite reflection and the reflection FWHM (width of standard profile/FWHM) of the silicon standard. Prepare and print out the evaluation protocol (Tab. 2).

2.7.2 The determination of the crystallite size in the X'Pert program is carried out using the Scherrer equation:

$$D_{(\text{Crystallite size})} \quad \frac{k \cdot \lambda}{W_{Size} \cdot \cos\theta}$$

$D_{(\text{Crystallite size})}$	Crystallite size in nm
k	Form factor of the crystallites = 0.9 (mean value from the literature)
λ	Wavelength in nm
W_{Size}	Integral width of the goethite reflection - FWHM of the silicon standard
$\cos\theta$	Maximum of the goethite reflection in ° 2 theta

Table 1

Peak parameters of the silicon reflection		
Parameters		
Position (° 2 theta)		28.45746
Net height (counts)		8588.32
Background height at position (counts)		66.56
Net area (° 2 theta · counts)		1182
Background area (° 2 theta · counts)		200
FWHM (° 2 theta)		0.0976
Integral breadth (° 2 theta)		0.1376
FWHM / Integrad breadth		0.7094
Asymmetry		0.99
Background		
Low angle side	(° 2 theta)	27.02000
	(counts)	61.58
High angle side	(°2 theta)	29.98000
	(counts)	71.75

Table 2

Peak parameters of the goethite sample: crystallite size 46.5 nm		
Position (° 2 theta)		21.25219
Net height (counts)		2120.73
Background height at peak position (counts)		44.92
Net area (° 2 theta · counts)		589
Background area (° 2 theta · counts)		237
FWHM (° 2 theta)		0.2061
Integral breadth (° 2 theta)		0.2779
FWHM / Integrad breadth		0.7415
Asymmetry		1.16
Background		
Low angle side	(° 2 theta)	18.54000
	(counts)	58.38
High angle side	(° 2 theta)	23.67000
	(counts)	33.05

Table 3

Crystallite size determination using X'Pert program; Scherrer equation

Menu option: Additional functions in the X'Pert program section: X'Pert Organiser

Anode material	Cu (copper)
Radiation type	Cu Kα
Wavelength (nm)	0.154184
K factor (form factor mean value)	0.9000
Intensity ratio Cu $K\alpha_1$ / Cu $K\alpha_2$	0.5000

Signal broadening (° 2 theta)	Signal width (° 2 theta)	Particle size broadening (° 2 theta)	Lattice broadening (° 2 theta)	Signal position (° 2 theta)	Crystal-lite size (nm)
0.1376	0.0976	0.0400	0.0970	28.45500	205.1
0.2779	0.0976	0.1803	0.2602	21.25400	44.9
0.2766	0.0976	0.1790	0.2588	21.25100	45.2
0.8814	0.0976	0.7838	0.8760	21.22800	10.3
0.9325	0.0976	0.8349	0.9274	21.24400	9.7
0.4287	0.0976	0.3311	0.4174	21.22090	24.4
0.4274	0.0976	0.3298	0.4161	21.21911	24.5

3. Measurement of the color values

The measurement of the color values is carried out as described in EP-A 0 911 370.

Examples

Example 1: Aluminum-containing nucleus of $FeCl_2$ and $AlCl_3$

14.095 l of $FeCl_2$ solution containing 55.07 g/l of Fe and having an Fe(III) content of 1.5 mol% (based on total Fe) were introduced into a batchwise stirred vessel having an effective volume of 30 liter and equipped with a three-speed crossbeam stirrer and a gassing means (ring having holes below the stirrer). 914 g of $AlCl_3$ solution containing 5.06% by weight of Al and 3.95% by weight of HCl were then added.

The amount of $FeCl_2$ corresponded to 13.9 mol of Fe (Fe(II) and Fe(III)), the amount of $AlCl_3$ to 1.71 mol and the amount of HCl to 0.989 mol. The Al/total Fe ratio accordingly corresponded to 12.3 mol%, based on total Fe. This solution was heated to 44°C while stirring and gassing with 300 l(S.T.P.)/h of nitrogen. After this temperature had been reached, 1 615 ml of sodium hydroxide solution containing 300 g of NaOH/l (corresponding to 7.5 equivalents per liter) were pumped in in 6 minutes by means of a gear pump. Accordingly, 33.8% of the metals Fe + Al were precipitated. Immediately after the end of the precipitation, the gassing with nitrogen was stopped and gassing at 97 l(S.T.P.)/h of air was effected for oxidation. 180 minutes after the beginning of the oxidation, the reaction was complete. The oxidation rate was accordingly 33.3 mol% of Fe(II)/h. (Here, only the 33.8% of the Fe which were precipitated by the NaOH was taken into account. The Fe(III) fraction was calculated as Fe(II). It was furthermore assumed that Fe and Al are precipitated uniformly.)

The nucleus obtained had the following properties:

BET surface area:	100 m^2/g
Crystallite size:	27.0 nm
AV:	2700 (AV = BET surface area multiplied by crystallite size)

Comparative Example 1: Aluminum-containing nucleus of $FeCl_2$ and $AlCl_3$

14.095 l of $FeCl_2$ solution containing 55.07 g/l of Fe and having an Fe(III) content of 1.5 mol% (based on total Fe) are introduced into a batchwise stirred vessel having an effective volume of 30 liter and equipped with a three-speed crossbeam stirrer and a gassing means (ring having holes below the stirrer). 914 g of $AlCl_3$ solution containing 5.06% by weight of Al and 3.95% by weight of HCl are then added.

The amount of $FeCl_2$ corresponds to 13.9 mol of Fe (Fe(II) and Fe(III)), the amount of $AlCl_3$ to 1.71 mol and the amount of HCl to 0.989 mol. The Al/total Fe ratio accordingly corresponds to 12.3 mol%, based on total Fe. This solution is heated to 34°C while stirring and gassing with 300 l(S.T.P.)/h of nitrogen. After this temperature has been reached, 1 615 ml of sodium hydroxide solution containing 300 g of NaOH per liter (corresponding to 7.5 equivalents per liter) are pumped in in 6 minutes by means of a gear pump. Accordingly, 33.8% of the metals Fe + Al are precipitated by the NaOH. Immediately after the end of the precipitation, the gassing with nitrogen is stopped and gassing at 97 l(S.T.P.)/h of air is effected. 180 minutes after the beginning of the oxidation, the reaction was complete. The oxidation rate was accordingly 33.3 mol% of Fe(II)/h. (Here, only the 33.8% of the Fe which were precipitated by the NaOH was taken into account. The Fe(III) fraction was calculated as Fe(II). It was furthermore assumed that Fe and Al are precipitated uniformly.)

The nucleus had the following properties:

BET surface area:	173 m^2/g
Crystallite size:	17.5 nm
AV:	3027

Comparative Example 2: Aluminum-containing nucleus of $FeCl_2$ and $AlCl_3$

11.58 l of $FeCl_2$ solution containing 55.09 g/l of Fe and having an Fe(III) content of 1.0 mol% (based on total Fe) are introduced into a batchwise stirred vessel having an effective volume of 30 liter and equipped with a three-speed crossbeam stirrer and a gassing means (ring having holes below the stirrer). 818 g of $AlCl_3$ solution containing 6.00% by weight of Al and 1.6% by weight of HCl are then added.

The amount of $FeCl_2$ corresponds to 9.5 mol of Fe (Fe(II) and Fe(III)), the amount of $AlCl_3$ to 1.82 mol and the amount of HCl to 0.36 mol. The Al/total Fe ratio accordingly corresponds to 19.2 mol%, based on total Fe. This solution is heated to 44°C while stirring and gassing with 300 l(S.T.P.)/h of nitrogen. After this temperature has been reached, 1 061 ml of sodium hydroxide solution containing 300 g of NaOH/l (corresponding to 7.5 equivalents per liter) are pumped in in 10 minutes by means of a gear pump. Accordingly, 32.1% of the metals Fe + Al are precipitated by the NaOH. Immediately after the end of the precipitation, the gassing with nitrogen is stopped and gassing at 67 l(S.T.P.)/h of air is effected. 135 minutes after the beginning of the oxidation, the reaction was complete. The oxidation rate was accordingly 44.5 mol% of Fe(II)/h. (Here, only the 32.1% of the Fe which were precipitated by the NaOH was taken into account. The Fe(III) fraction was calculated as Fe(II). It was furthermore assumed that Fe and Al are precipitated uniformly.)

The nucleus had the following properties:

BET surface area:	186 m^2/g
Crystallite size:	15.5 nm
AV:	2883

Example 4: Preparation of yellow pigment

2 mol of yellow nucleus suspension from example 1 (calculated as moles of α-FeOOH) and 20 mol of $FeCl_2$ having an Fe content of 95.7 g/l of Fe are introduced into a batchwise stirred vessel having a gassing ring, pH measurement, temperature control and a three-speed crossbeam stirrer. This suspension was heated to 60°C with constant stirring. After this temperature had been reached, a pH of 3.4 is maintained by constantly metering in sodium hydroxide solution with 300 g/l of NaOH. At the same time, gassing is effected with 76 l(S.T.P.)/h of air. After a gassing time of 1 618 minutes, all Fe(II) had been oxidized, corresponding to an oxidation rate of 3.7 mol% of Fe/h.

The yellow pigment prepared has the following properties:

BET surface area:	32.7 m^2/g
Crystallite size:	27 nm
Color strength (relative to Bayferrox® 915):	97%
da* :	0.2
db* :	-1.2
dL* (relative to Bayferrox® 915) :	-0.7
da* :	0.7
db* :	-1.2

Example 5: Preparation of yellow pigment

If instead of the nucleus from example 1, the nucleus from example 2 is used, and the amount of air is set at 76 l(S.T.P.)/h, under otherwise identical reaction conditions, a yellow pigment having the following properties is obtained after 1 618 minutes (oxidation rate 3.7 mol%/h):

BET surface area:	45.6 m^2/g
Crystallite size:	25 nm
Color strength (relative to Bayferrox® 915):	97 %
da* :	0.1
db* :	-2.6
dL* (relative to Bayferrox® 915) :	-3.4
da* :	1.1
db* :	-4.0

<u>Example 6:</u> Preparation of yellow pigment

If instead of the nucleus from example 1, the nucleus from example 3 is used, the following yellow pigment is obtained under the reaction conditions of example 5:

BET surface area:	32.7 m^2/g
Crystallite size:	32 nm
Color strength (relative to Bayferrox® 915):	101 %
da* :	-0.9
db* :	-2.1
dL* (relative to Bayferrox® 915) :	-2.4
da* :	-1.0
db* :	-4.0

Only the pigment prepared using a nucleus according to the invention has a color sufficiently similar to the comparative type Bayferrox® 915 to enable it to be used as a high-quality yellow pigment. The two yellow pigments prepared from the nuclei of the comparative examples are substantially too dark (in full shade) and not sufficiently yellow.

Patent claims:

1. A method for producing aluminum-containing iron oxide nuclei having an α-FeOOH crystal structure with an aspect ratio of 2 100 to 3 600 and a BET surface area of 50 to 150 m2/g using FeCl2, characterized in that

   a.) first, an Al component is added in amounts of 4 - 13 mol%, based on total Fe, while stirring, to an iron(II) chloride solution having a total Fe content of 20 - 100 g/l, preferably 40 - 65 g/l, and an Fe(III) content of 0.1 - 10 mol% of Fe(III) (based on total Fe),

   b.) this mixture is heated to a precipitation temperature of from 30°C to 60°C, preferably 35 to 50 degrees,

   c.) a precipitating agent having an active substance content of 2 - 10 equivalents per liter, preferably 4 - 8 equivalents per liter, is added to the mixture, and the molar ratio of Fe + Al to the precipitating agent is 20% - 80%, preferably 30% - 60%, of the stoichiometry,

   d.) the precipitated suspension is then oxidized with an oxidizing agent at a rate such that the oxidation rate is 2 - 50 mol%/h, preferably 10 - 35 mol%/h, of the iron to be oxidized.

2. The method as claimed in claim 1, characterized in that aluminum chloride is used as the Al component.

3. The method as claimed in claim 1 and 2, characterized in that NaOH, KOH, Na_2CO_3, K_2CO_3, $Mg(OH)_2$, MgO, $MgCO_3$, $Ca(OH)_2$, CaO, $CaCO_3$, NH_3 or

secondary or tertiary aliphatic amines in aqueous solution or aqueous suspension are used as the precipitating agent.

4. The method as claimed in any of claims 1 to 3, characterized in that atmospheric oxygen, oxygen, ozone, H_2O_2, chlorine, nitrates of the alkali metals or alkaline earth metals or NH_4NO_3 are used as the oxidizing agent.

5. The method as claimed in any of claims 1 to 4, characterized in that the reaction is carried out in batchwise or continuous stirred vessels, in stirred vessel cascades, loop reactors or stirrer-free reactors having binary nozzles as mixing elements.

6. The use of the α-FeOOH nuclei produced as claimed in any of claims 1-5 for the preparation of yellow pigment.

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