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Zur Erklärung der Zweibuchstaben-Codes und der anderen Abkürzungen wird auf die Erklärungen (“Guidance Notes on Codes and Abbreviations”) am Anfang jeder regulären Ausgabe der PCT-Gazette verwiesen.

(54) Title: IRON OXIDE PIGMENTS

(54) Bezeichnung: EISENOSTPIGMENTE

(57) Abstract: The invention relates to iron oxide pigments comprising an average solid body diameter of 10 to 500 μm, and to the production and use thereof.

(57) Zusammenfassung: Die Erfindung betrifft Eisenoxipigmente mit einem mittleren Festkörperrdurchmesser von 10 bis 500 μm sowie ihre Herstellung und Verwendung.
**Iron oxide pigments**

The invention relates to red iron oxide pigments having a mean solids diameter of 10 to 500 μm and to their preparation and their use.

There are various processes for the preparation of red iron oxide pigments:

a) Laux process

The Laux process starts from nitrobenzene and Fe metal and leads initially to iron oxide black or iron oxide yellow and aniline. In order to prepare iron oxide red by this process, the iron oxide black obtained is calcined. The process is very complicated and is not easy to master since variable proportions of control chemicals have to be used in order to establish the desired particle size. Moreover, the required apparatus technology is demanding and correspondingly expensive. Furthermore, aniline forms as a second product under reaction and, owing to its properties, necessitates particular occupational hygiene measures.

A disadvantage of the iron oxide red prepared by the Laux process is that the iron oxide red has a tendency to flocculate in the finish and to agglomerate. Furthermore, the iron oxide red prepared by the Laux process produces dust and have a high DIN pH value (6).

b) Precipitation process

The preparation of iron oxide red by a direct precipitation process is described in US-5 421 878. The direct precipitation process is difficult in terms of process engineering since α-Fe₂O₃ is obtainable only in a narrow range and the reaction is not easy to master. The iron oxide red prepared by the precipitation process has the disadvantage of high salt loads which pollute the wastewater and are therefore ecologically unsafe.

Apart from high production costs, the iron oxide reds prepared by the precipitation process furthermore have the disadvantage that they produce dust and have a high DIN pH value (4.5 to 6).
c) Hydrothermal process

The hydrothermal process is described in DE-A-19917786. Iron oxide red pigments for high-quality applications, in particular for paints and finishes, can be prepared by the hydrothermal process. However, the high process costs due to the pressure technique have a disadvantageous effect here. This process is therefore not suitable for simple applications in which economical products are required.

Apart from high production costs, the iron oxide red pigments prepared by the hydrothermal process furthermore have the disadvantage that they produce dust.

d) Penniman-Zoph process

The hydrothermal process is described in DE-A-19958168. According to the Penniman-Zoph process, iron oxide red pigments are prepared by dissolving iron metal with addition of an iron oxide red nucleus and oxidizing it. Here, as a rule nitric acid is used for producing the nucleus, so that nitrate or ammonia is present in the wastewater and has to be removed by means of complicated process engineering. As in the hydrothermal process and in the precipitation process, this leads to high production costs which limit the application of such pigments to a few fields.

Apart from an expensive and environmentally polluting preparation process, the iron oxide reds prepared by the hydrothermal process furthermore have the disadvantage that they produce dust and have a high DIN pH (4.5 to 6).

e) Calcination of iron-containing materials

The calcination of iron-containing materials is described in EP-A-0 911 369. Iron oxide red can be prepared by calcining iron oxide yellow, iron oxide black or other iron-containing oxidic or metallic solids. Owing to the high temperatures required, the quality of the iron oxide red pigments prepared therefrom suffers. In order to prepare high-quality iron oxide red pigments, high-quality precursor compounds are therefore required and make the process more expensive.

The iron oxide red pigments prepared by calcining iron-containing materials furthermore have the disadvantage that they are relatively hard and have to be milled by a complicated procedure. Furthermore, the iron oxide reds prepared by calcining
iron-containing materials produce dust.

f) Decomposition of FeSO₄

The decomposition of iron(II) sulfate at high temperatures leads to iron oxide red and SO₂, which can be reacted to give sulfuric acid. Owing to the high temperatures required and the corrosiveness of the gases formed, this process requires high expenditures on apparatus technology.

Apart from the disadvantages of the preparation process which are described above, the iron oxide reds prepared by decomposing FeSO₄ furthermore have the disadvantage that they produce dust.

For many applications in the area of the coloring of concrete parts, emulsion paints and paper coloring, granules are now used since they produce little dust, and are flowable and readily dispersible. In the customary granulation processes, powders are used as starting materials and are mixed with a binder and then granulated. Customary granulation processes are spray granulation, press granulation and pan granulation.

g) Pyrohydrolysis of FeCl₂ or Fe(NO₃)₂

Pyrohydrolysis has been developed to industrial maturity since about 1960 and initially served mainly for recovering HCl from pickling solutions (FeCl₂). In the meantime, it has become an important process for obtaining oxide raw materials, in particular iron oxides. Advantages of this process are that it can be operated continuously, uses liquid raw materials, is economical and uses no byproducts and process chemicals and is therefore particularly environmentally friendly.

This process is widely used in the metallurgical industry. The main product here is the recovered hydrochloric acid (in some cases also hydrofluoric acid or nitric acid), which is reused for pickling steel. Iron oxide forms as a byproduct and is initially fed back to the blast furnace. By special purification steps for the pickle (FeCl₂ solution), it is possible to prepare pure iron oxides for the ferrite industry. In the case of this application, an exactly defined chemical composition which is as constant as possible and a low degree of impurities are required. Relatively hard-sintered oxides having as low a chloride content as possible are required. The specific surface area
(determined according to the BET method) is usually 3-5 m$^2$/g, depending on the reaction temperature of the decomposition. In individual cases, BET surfaces areas of 10 m$^2$/g can also be achieved. The process is described in detail in articles and patents (Kladnig, W. & Kamer, W.; cfi/Ber DKG 67 (1990), 80; EP-A-0850881).

Owing to the low specific surface area of such products, these are not suitable for high-quality pigment applications since they have an undesired blue tinge. Customary commercially available iron oxide red pigments prepared by the pyrohydrolysis process have BET surface areas between 2 and 5.5 m$^2$/g (company brochure Bailey-PVS Oxides L.L.C.; company brochure Thyssen Krupp Stahl 05/2000 [Thyssen Krupp Steel 05/2000]). Owing to their low specific surface area, these products, too, have a blue tinge and are therefore not suitable for high-quality pigment applications.

It was the object of the invention to prepare a low-dust, free-flowing red iron oxide pigment which has good color properties, i.e. no blue tinge, even without a binder.

The invention relates to iron oxide pigments having L*, a* and b* values, measured in the lightened tone according to CIELAB units, of

\[
\begin{align*}
    L^* & = 58 \text{ to } 62, \text{ in particular } 59 \text{ to } 60.5, \\
    a^* & = 22 \text{ to } 27, \text{ in particular } 23 \text{ to } 26, \\
    b^* & = 10 \text{ to } 24, \text{ in particular } 10 \text{ to } 15, \text{ and}
\end{align*}
\]

having an iron oxide content greater than 99% by weight, based on the pigment, and a mean solids diameter of 10 to 500 μm.

In the context of this Application, "solids" are understood as meaning spheres or hollow spheres, it being possible for the hollow spheres to contain one or more holes. The majority of the solids, i.e. more than 50%, consists of hollow spheres. A photograph of the solids according to the invention is shown in Fig. 1.

The solids of the iron oxide pigments preferably have a BET surface area of 6.0 to 12.0 m$^2$/g.

The solids of the iron oxide pigments consist of primary particles which preferably have a mean size of 0.05 to 0.5, preferably of 0.1 to 0.3, μm. The primary particles
are likewise part of the invention.

The iron oxide pigment preferably has a DIN pH of 2.5 to 4.0, preferably of 2.8 to 3.5.

It is also preferable if the chloride content is 0.1% by weight or less, based on the pigment.

The invention furthermore relates to an iron oxide pigment having L*, a* and b* values, measured in the lightened tone according to CIELAB units, of

\[ L^* = 58 \text{ to } 62, \text{ in particular } 59 \text{ to } 60.5, \]
\[ a^* = 22 \text{ to } 27, \text{ in particular } 23 \text{ to } 26, \]
\[ b^* = 10 \text{ to } 24, \text{ in particular } 10 \text{ to } 15, \text{ and} \]

the iron oxide pigment consisting of primary particles which have a mean size of 0.05 to 0.5, in particular 0.1 to 0.3, \( \mu \text{m} \).

The iron oxide pigment preferably has a DIN pH of 2.5 to 4.0, preferably of 2.8 to 3.5.

The abovementioned iron oxide pigment preferably has a chloride content of 0.1% by weight or less, based on the pigment.

The invention furthermore relates to a process for the preparation of the iron oxide pigments according to the invention, characterized in that drops of an aqueous iron chloride solution are dewatered in order to form solids, which are subsequently calcined.

In the context of this Application, "calcination" is understood as meaning the thermal decomposition of the iron chloride solution according to one of the following equation:

\[ 2\text{FeCl}_2 + 2\text{H}_2\text{O} + 1/2\text{O}_2 \rightarrow \text{Fe}_2\text{O}_3 + 4\text{HCl} \]
\[ 2\text{FeCl}_3 + 3\text{H}_2\text{O} \rightarrow \text{Fe}_2\text{O}_3 + 6\text{HCl} \]

The process according to the invention is preferably carried out in such a way that
the iron chloride solution, in which iron chloride of the iron chloride solution is present FeCl₂ and/or FeCl₃, is sprayed into a reactor through an airless or binary nozzle so that drops having a mean diameter of 50 to 1000 μm form.

5 The dewatering preferably takes place at a temperature of 300 to 900°C, preferably of 400 to 700°C. The dewatering can be produced, for example, in a reactor by combustion gases, electrical heating, microwave heating or electromagnetic waves. The combustion gases can be fed cocurrently or countercurrently. The gas is preferably separated from the iron oxide pigment inside or outside the reactor and worked up to give hydrochloric acid solution.

10 The calcination is carried out at temperatures of 200 to 800°C.

The calcination can take place as a result of a thermal treatment, by bringing stream into contact with the solid at temperatures of 200 to 400°C. The thermal treatment can take place in the same or in a separate reactor.

15 After the calcination, a further thermal treatment at temperatures of 200 to 800°C can be carried out. In this thermal treatment, the primary particles grow with the result that the tinctorial properties improve. This thermal treatment can take place in the same or in a separate reactor.

20 The total residence time of the drops/solids at temperatures of more than 300°C during the dewatering and calcination is preferably between 1 second and 90 minutes, preferably between 5 minutes and 70 minutes.

25 In addition, after the dewatering, either before or after the calcination, the solids can be cooled and then washed with water.

30 All preparation steps can be carried out either in the same reactor or in different reactors.

35 The following reaction parameters are preferably established: Spraying an aqueous FeCl₂ solution having a content of 100 to 400 g/l of FeCl₂ by means of a binary nozzle into the reactor so that drops having a mean diameter of 50 to 200 μm form. Adjusting the reaction temperature to 300 to 600°C in the reactor. Subsequent dewatering in the same reactor at temperatures of 600°C to 800°C. Removal of
chloride by passing steam through a bed of the product at 200° to 400°C. Subsequent thermal treatment at temperatures of 600 to 800°C.

The preparation process gives yellow-red iron oxide pigments which are suitable for a broad spectrum of use. These iron oxide pigments can furthermore be prepared in the absence of a binder in the context of the invention. Solids are obtained by the process according to the invention. The majority of the solids are obtained as hollow spheres.

For some applications, particularly in the paint and finish sector, finely milled powders are required. The solids can therefore subsequently be milled after the calcination until a mean size of 0.05 to 0.5, preferably 0.1 to 0.3, µm is reached. The milling apparatus used is preferably a jet mill, a pendulum roller mill or a mechanical classifier mill.

The invention furthermore relates to the use of the iron oxide pigments according to the invention in the construction sector, for paints and finishes, as raw material for the production of hard and soft ferrites, for the production of catalysts, for coloring paper and for use in colored substances in food and/or in the cosmetics sector.

In the context of the present invention, applications in the construction sector are understood as meaning applications in renders, paving stones, mortar mixtures, etc. The iron oxide pigment formed in the reactor can furthermore be filled and used directly after cooling.

The iron oxide pigments according to the invention can be used as solids directly in the construction sector and/or for the production of catalysts.

For some applications, finely milled powders are required. The iron oxide pigments according to the invention can therefore be used as primary particles in the construction sector, for paints and finishes, as raw material for the production of hard and soft ferrites, for the production of catalysts, for coloring paper and for use in colored substances in food and/or in the cosmetics sector.

The invention is to be explained by the following examples:
Examples

Experimental arrangement

The measurement of the lightened tone (color strength) of the particles obtained is effected as stated in EP-A-911369, page 6, line 9 to page 7, line 26.

The particle size was determined from transmission electron micrographs (primary particles) or scanning electron micrographs (solids, inter alia as hollow spheres).

The determination of the metallic secondary components was effected by ICP-OES. ICP-OES is a method for determining elements which are present in low concentration in an aqueous sample. It is a spectroscopic method in which the element to be determined is excited and the emitted light of the transition to the ground state, which is characteristic of each element, is measured (OES = optical emission). The excitation is effected by means of a plasma burner (ICP = inductive cuppled plasma). The limit of detection of the method of determination is 5 µg/kg.

The determination of the chloride content was determined argentometrically with potentiometric endpoint determination. The limit of detection of the method of determination is 50 mg/kg.

The measurement of the pH of the powder is effected in a suspension in demineralized water according to DIN-EN-ISO 787-9.

Example 1

In a spray roast reactor, an aqueous iron(II) chloride solution having a concentration of 340 g/l of FeCl₂ and an HCl content of 10 g/l was sprayed into the reactor at a temperature of 600°C. The throughput was 14 l of solution per hour. The material separated off in the cyclone was recycled to the feed stream. The residence time in the reactor was 10 seconds. Solids formed inter alia in the form of hollow spheres having a mean solids diameter of 80 to 400 µm.

After cooling, the material separated off was washed with water until a chloride content of less than 0.1% by weight was reached.
After the washing, the material was calcined in a laboratory chamber furnace at 800°C for 60 minutes. The total residence time at temperatures of more than 300°C was therefore 60 min 10 sec.

The end product had the following properties:

- Mean diameter of the solids: 200 μm
- Mean diameter of the primary particles: 0.2 μm
- L*: 58.4 (lightened tone)
- a*: 24.4 (lightened tone)
- b*: 13.4 (lightened tone)
- Cl content: 0.1% by weight
- BET surface area: 6.1 m²/g
- DIN pH: 2.9

**Example 2**

In a spray roast reactor, an aqueous iron(II) chloride solution having a concentration of 340 g/l of FeCl₂ and an HCl content of 10 g/l was sprayed into the reactor at a temperature of 560°C. The throughput was 14 l of solution per hour. The material separated off in the cyclone was recycled to the feed stream. The residence time in the reactor was 10 seconds. Solids formed inter alia in the form of hollow spheres having a mean solids diameter of 100 to 500 μm.

After cooling, the material separated off was washed with water until a chloride content of less than 0.1% by weight was reached.

After the washing, the material was calcined in a laboratory chamber furnace at 750°C for 60 minutes. The cooling rate was 130°C per minute. The total residence time at temperatures of more than 300°C was therefore 60 min 10 sec.

The end product had the following properties:

- Mean diameter of the solids: 300 μm
- Mean diameter of the primary particles: 0.2 μm
- L*: 58.9 (lightened tone)
- a*: 24.3 (lightened tone)
- b*: 13.2 (lightened tone)
- Cl content: 0.07% by weight
- BET surface area: 9.6 m²/g
- DIN pH: 3.4

Example 3

In a spray roast reactor, an aqueous iron(II) chloride solution having a concentration of 340 g/l of FeCl₂ and an HCl content of 10 g/l was sprayed into the reactor at a temperature of 560°C. The throughput was 14 l of solution per hour. The material separated off in the cyclone was recycled to the feed stream. The residence time in the reactor was 10 seconds. Solids formed inter alia in the form of hollow spheres having a mean solids diameter of 100 to 500 μm.

After cooling, the material separated off was washed with water until a chloride content of less than 0.1% by weight was reached.

After the washing, the material was calcined in a laboratory chamber furnace at 800°C for 60 minutes. The cooling rate was 150°C per minute. The total residence time at temperatures of more than 300°C was therefore 60 min 10 sec.

The end product had the following properties:

- Mean diameter of the solids: 300 μm
- Mean diameter of the primary particles: 0.2 μm
- L*: 58.9 (lightened tone)
- a*: 23.8 (lightened tone)
- b*: 11.9 (lightened tone)
- Cl content: 0.06% by weight
- BET surface area: 7.2 m²/g
- DIN pH: 3.2

Comparative example for Examples 2 and 3

The material from Examples 2 and 3 was investigated after washing without subsequent calcination in the laboratory chamber furnace and had the following properties:
- Mean diameter of the solids: 300 µm
- Mean diameter of the primary particles: 0.2 µm
- L*: 60.8 (lightened tone)
- a*: 23.5 (lightened tone)
- b*: 23.1 (lightened tone)
- Cl content: 0.09% by weight
- BET surface area: 20 m²/g

10 **Comparative Example 2**

In a spray roat reactor, an aqueous iron(II) chloride solution having a concentration of 340 g/l of FeCl₂ and an HCl content of 10 g/l was sprayed into the reactor at a temperature of 660°C. The throughput was 14 l of solution per hour. The material separated off in the cyclone was recycled to the feed stream. The residence time in the reactor was 10 seconds. Solids formed inter alia in the form of hollow spheres having a mean solids diameter of 100 to 500 µm.

After cooling, the material separated off was washed with water until a chloride content of less than 0.1% by weight was reached.

The end product had the following properties:

- Mean diameter of the solids: 250 µm
- Mean diameter of the primary particles: 0.2 µm
- L*: 59.0 (lightened tone)
- a*: 20.9 (lightened tone)
- b*: 10.9 (lightened tone)
- Cl content: 0.09% by weight
- BET surface area: 7.2 m²/g
**Overview of the examples**

<table>
<thead>
<tr>
<th></th>
<th>Examples</th>
<th>Comparative examples</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>1</td>
<td>2</td>
</tr>
<tr>
<td>Dewatering in ºC</td>
<td>600</td>
<td>560</td>
</tr>
<tr>
<td>Dewatering time in sec</td>
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<td>10</td>
</tr>
<tr>
<td>Calcination in ºC</td>
<td>800</td>
<td>750</td>
</tr>
<tr>
<td>Calcination time in min</td>
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<td>Total residence time</td>
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<tr>
<td>Mean diameter of the solids in µm</td>
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<tr>
<td>Mean diameter of the primary particles in µm</td>
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<tr>
<td>L*</td>
<td>58.4</td>
<td>58.9</td>
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<tr>
<td>a*</td>
<td>24.4</td>
<td>24.3</td>
</tr>
<tr>
<td>b*</td>
<td>13.4</td>
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<tr>
<td>Cl content (% by weight)</td>
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<tr>
<td>BET (m²/g)</td>
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</tr>
<tr>
<td>DIN pH</td>
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<td>3.4</td>
</tr>
</tbody>
</table>
Patent claims

1. An iron oxide pigment having $L^*$, $a^*$ and $b^*$ values, measured in the lightened tone according to CIELAB units, of

$L^* = 58$ to $62$, in particular $59$ to $60.5$,
$a^* = 22$ to $27$, in particular $23$ to $26$,
$b^* = 10$ to $24$, in particular $10$ to $15$, and

having an iron oxide content greater than 99% by weight, based on the pigment, and a mean solids diameter of 10 to 500 $\mu$m.

2. The iron oxide pigment as claimed in claim 1, characterized in that the solids have a BET surface area of 6.0 to 12.0 $m^2/g$.

3. The iron oxide pigment as claimed in claim 1, characterized in that the primary particles of the solids have a mean size of 0.05 to 0.5, in particular of 0.1 to 0.3, $\mu$m.

4. The iron oxide pigment as claimed in claim 1, characterized in that the iron oxide pigment has a DIN pH of 2.5 to 4.0, in particular 2.8 to 3.5.

5. The iron oxide pigment as claimed in any of claims 1 to 4, characterized in that the iron oxide pigment has a chloride content of 0.1% by weight or less, based on the pigment.

6. An iron oxide pigment having $L^*$, $a^*$ and $b^*$ values, measured in the lightened tone according to CIELAB units, of

$L^* = 58$ to $62$, in particular $59$ to $60.5$,
$a^* = 22$ to $27$, in particular $23$ to $26$,
$b^* = 10$ to $24$, in particular $10$ to $15$, and

having an iron oxide content greater than 99% by weight, based on the pigment, the iron oxide pigment consisting of particles which have a mean size of 0.05 to 0.5, in particular 0.1 to 0.3, $\mu$m.
7. The iron oxide pigment as claimed in claim 6, characterized in that the iron oxide pigment has a DIN pH of 2.5 to 4.0, in particular of 2.8 to 3.5.

8. The iron oxide pigment as claimed in claim 6, characterized in that it has a chloride content of 0.1% by weight or less, based on the pigment.

9. A process for the preparation of iron oxide pigment as claimed in any of claims 1 to 8, characterized in that drops of an iron chloride solution are dewatered in order to form solids, which are subsequently calcined in order to reduce their chloride content.

10. The process for the preparation of iron oxide pigment as claimed in claim 9, characterized in that the iron chloride of the iron chloride solution is FeCl₂ or FeCl₃.

11. The process for the preparation of iron oxide pigment as claimed in claim 9, characterized in that the drops have a mean diameter of 50 to 1000 μm.

12. The process for the preparation of iron oxide pigment as claimed in claim 9, characterized in that the dewatering takes place at a temperature of 300 to 900°C, in particular of 400 to 700°C.

13. The process for the preparation of iron oxide pigment as claimed in claim 9, characterized in that the calcination is carried out at temperatures of 200 to 800°C.

14. The process for the preparation of iron oxide pigment as claimed in claim 9, characterized in that the dechlorination takes place by means of a thermal treatment, by bringing steam into contact with the solid at temperatures of 200 to 400°C.

15. The process for the preparation of iron oxide pigments as claimed in claim 9, characterized in that a second thermal treatment is carried out at temperatures of 200 to 800°C.

16. The process for the preparation of iron oxide pigment as claimed in claim 9, characterized in that the total residence time of the drops/solids at
temperatures of more than 300°C during the dewatering and calcination is between 1 s and 90 min, in particular between 5 min and 70 min.

17. The process for the preparation of iron oxide pigment as claimed in claim 9, characterized in that in addition, after the dewatering, either before or after the calcination, the solids are cooled and are then washed with water.

18. The process for the preparation of iron oxide pigment as claimed in claim 9, characterized in that all preparation steps are carried out in the same reactor or in different reactors.

19. The process for the preparation of iron oxide pigment as claimed in any of claims 9 to 17, characterized in that, after the calcination, the solids are subsequently milled, in particular using a jet mill, a pendulum roller mill or a mechanical classifier mill, until a mean size of 0.05 to 0.5, in particular of 0.1 to 0.3, μm is reached.

20. The use of iron oxide pigments as claimed in any of claims 1 to 8 or the use of iron oxide pigments prepared as claimed in claims 9 to 19 in the construction sector, for paints and finishes, as raw material for the production of hard and soft ferrites, for the production of catalysts, for coloring paper and for use in colored substances in food and/or in the cosmetics sector.

21. The use of iron oxide pigments as claimed in any of claims 1 to 5 or the use of iron oxide pigments prepared as claimed in claims 9 to 18 in the construction sector and/or for the production of catalysts.

22. The use of iron oxide pigments as claimed in any of claims 6 to 8 or the use of iron oxide pigments prepared as claimed in claim 19 in the construction sector, for paints and finishes, as raw materials for the production of hard and soft ferrites, for the production of catalysts, for coloring paper and for use in colored substances in food and/or in the cosmetics sector.
Application number numéro de demande: EP04/02178.

Figures: ____________________________

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