**EUROPEAN PATENT SPECIFICATION**

**Date of publication of patent specification:** 19.12.84  
**Application number:** 81304177.9  
**Date of filing:** 11.09.81  
**Int. Cl.²:** C 01 G 49/00, C 08 K 3/18

**Yellow pigments stable at high temperatures.**

**Date of publication of application:** 23.03.83 Bulletin 83/12  
**Publication of the grant of the patent:** 19.12.84 Bulletin 84/51  
**Designated Contracting States:** DE FR GB IT NL SE

**References cited:**  
US-A-3 822 210  
US-A-3 832 455

**CHEMICAL ABSTRACTS,** vol. 85, no. 20, 15th November 1976, page 103, column 2, no. 144878v, Columbus Ohio (USA);

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Description

The present invention relates to inorganic pigments and to a method for making them. More particularly, the present invention concerns yellow pigments derived from inorganic materials. Even more particularly, the present invention concerns yellow pigments stable at high temperatures.

As is known to those skilled in the art to which the present invention pertains, yellow inorganic pigments, except nickel titanate, are not suitable for processing into plastics because of their thermal instability at temperatures above 175°C. Thus, yellow pigments such as iron oxide, lead chromate and zinc chromate are not well chosen for the yellow coloring of plastics such as polyethylene, polypropylene, polystyrene, polycarbonates, polyamide and the like. Furthermore, organic pigments show color degradation at temperatures of about 175°C, thereby precluding their use in the processing of plastics. Thus, the need for yellow pigments stable at high temperatures is readily apparent.

As will subsequently be detailed, the present invention provides such stable yellow pigments.

Statement of relevant patents

To the best of applicant’s knowledge, the following patents are the ones most relevant to a determination of patentability:

U.S. Patents Nos.
2,904,395 4,097,392 3,822,210
3,887,479 4,075,029 3,832,455

Perhaps the most pertinent of the patents mentioned above is Iwase et al. U.S. Patent No. 3,822,210. Although this patent teaches the making of zinc ferrite (spinel) pigments, it does not teach or suggest the present invention because it uses a method which is different from that of our invention, and its different method produces a different product. The products made by Iwase et al. are isotropic ferrites. They are made under conditions of temperature and mole ratio of alkali to metal salts different from those taught in accordance with the present invention. Iwase et al. teach that their product is formed only if the conditions of temperature and mole ratio of alkali to metal salts falls above the dashed curve in Figures 2A to 2C of the patent. Moreover, their method involves heating the precipitate while oxidation is occurring.

The products made by the present invention are acicular (needle-like) ferrites. The product is formed under conditions of temperature and mole ratio of alkali to metal salts which are different from those disclosed in Iwase et al. Moreover, our method provides that heating of the precipitate takes place after oxidation has occurred.

Summary of the invention

In accordance with the present invention, yellow pigments are provided by a spinel of iron and a metal selected from the group consisting of Mg, Zn, and Sn, as well as mixtures thereof, said spinel corresponding to the formula XFe₂O₄ wherein X is said metal, and being in the form of acicular particles having a length of 0.01 to 0.13 μm.

The spinels hereof may be produced by reacting a ferrous sulfate and a metal nitrate with a basic solution. The reaction usually proceeds at 5 to 50°C, preferably room temperature. The precipitate is then aerated and reheated to obtain the spinels hereof.

The spinels are temperature-stable up to about 900°C.

For a more complete understanding of the present invention, reference is made to the following detailed description and accompanying examples.

Description of the drawings

An understanding of the invention will be aided by consideration of the appended drawings, in which:

Figure 1 is a photomicrograph at a magnification of 160,000 dimensions of zinc ferrite pigment particles made in accordance with the present invention starting from zinc nitrate.

Figure 2 is a photomicrograph at the same magnification of zinc ferrite pigment particles made in accordance with the invention, starting from zinc sulfate, and

Figure 3 is a photomicrograph at the same magnification of particles as shown in Figure 1, after they have been calcined at 800°C for 20 minutes.

Description of the preferred embodiments

The present invention provides yellow pigments which are temperature-stable up to about 900°C.

As is known to the skilled artisan, yellow iron oxide pigments are called “geothite”. These pigments are considered to be hydrated oxides with a crystalline composition of alpha-FeOOH. It is the transformation of yellow alpha-FeOOH to a red alpha-Fe₂O₃ which causes color instability. The transformation of alpha-FeOOH to alpha-Fe₂O₃ occurs over a wide temperature range, beginning at 175°C, and is dependent upon the nature of the pigment and the particle size thereof.

The transformation is a function of temperature and length of time of exposure to such temperature. Generally, those skilled in the art consider the transformation to involve the dehydration of alpha-FeOOH to alpha-Fe₂O₃. A study of this phase transformation using differential thermal analysis shows that the transformation is complete at 265°C to 277°C, although the transformation commences at much lower temperatures.
The present invention, as will subsequently be detailed, is based upon the fact that the phase transformation is related not solely to dehydration but also to crystalline structure. For example, all four hydrated iron oxides with a composition of FeOOH differ in color by virtue of their crystalline structure, only alpha-FeOOH being yellow.

The present invention provides spinels of iron with various metals to produce stable yellow pigments. The various metals which are amenable hereto are selected from the group consisting of Mg, Zn, Sn, as well as mixtures thereof.

Although the applicants do not wish to be bound by any theory, it appears that, by virtue of the distribution of iron and other atoms within the spinel structure, the mobility of the iron atom is greatly inhibited and restricted. This restriction is further fortified by the electrostatic interaction between the metals, iron, oxygen and hydroxyl groups in the pigment. This restricted mobility results in a higher requirement of thermal energy to bring about the yellow-to-red color transformation of the iron oxide.

The spinels hereof may be produced by the reaction of hydrated ferrous sulfate and a metal nitrate hydrate or its equivalent in a solution, initially acidic, which has been subsequently brought to an alkaline pH. The reaction preferably proceeds at room temperature although those skilled in the art will appreciate that other temperatures such as 5 to 50°C also may be used, if desired. The precipitate so obtained is then oxidized to promote the oxidation of the ferrous iron.

To state the present invention in its method aspect comprehensively, this invention may be viewed as comprising a method of making a pigment having good stability at high temperatures, said method comprising the steps of forming a first aqueous solution consisting essentially of water, a soluble ferrous salt, and at least one other salt, said salt being a soluble salt of a non-ferrous metal selected from the group consisting of magnesium, zinc, and tin, said ferrous salt and said non-ferrous salt being present in said solution in proportions such that the respective quantities of iron and other metal which are present will yield, when said solution is so adjusted in pH as to cause precipitation, the formation of a substantial quantity of a precipitate oxidizable to a spinel of the formula XFe₂O₄, in which X is a metal selected from the group consisting of magnesium, zinc, and tin; mixing with said first aqueous solution a second aqueous solution to form a reaction mixture, said second solution containing a substantial proportion of a soluble alkalinizing compound selected from the group consisting of the carbonates, bicarbonates, and hydroxides of the alkali metals, the said second solution being used in such quantity and the said first and second solutions being in such a state of dilu-
tion that there results upon said mixing the formation of a precipitate in the form of particles of sub-micron size, the liquid phase of said reaction mixture being as a result of such precipitation substantially completely depleted in its content of metal ions having a valence greater than one; oxidizing at 15 to 35°C the said precipitate to form a spinel in aqueous solution; then heating said aqueous solution having said spinel therein to a temperature of from about 75°C to 100°C; and recovering said spinel.

Although we have worked particularly with hydrated metal nitrates among which Mg(NO₃)₂·6H₂O and Zn(NO₃)₂·6H₂O may be specifically mentioned, those skilled in the art will appreciate that it may in certain instances be possible to achieve the desired results with other equivalent materials which will suggest themselves to those skilled in the art. The use of hydrated salts is in general to be preferred, because such salts usually can be dissolved in water somewhat more quickly, other things being equal, than their anhydrous counterparts. After an aqueous solution of a desired strength has been obtained, it makes no difference whether the salt was or was not originally in the hydrated form.

Various other soluble anions may sometimes be used in place of nitrate, such as chloride or sulfate. As has been mentioned elsewhere, divalent tin may be used as a cation.

Suitable bases or solutions thereof are alkali-metal bases such as the sodium, potassium, or lithium carbonates, hydroxides, bicarbonates and the like. A particularly preferred base is sodium carbonate. Generally, a stoichiometric equivalent of base is employed. To be more specific, this means the use of one mole of sodium carbonate (or its equivalent, such as two moles of sodium bicarbonate) for each mole of, for example, hydrated ferrous sulfate. Insofar as its effect upon, for example, ferrous sulfate is concerned, the sodium carbonate may be considered as taking part in the reaction

\[ \text{FeSO}_4 + \text{H}_2\text{O} + \text{Na}_2\text{CO}_3 \rightarrow \text{Fe(OH)}_2 + \text{Na}_2\text{SO}_4 + \text{CO}_2 \]

Insofar as its effect upon, for example, magnesium nitrate is concerned, the sodium carbonate may be considered as taking part in the reaction

\[ \text{Mg(NO}_3)_2 + \text{H}_2\text{O} + \text{Na}_2\text{CO}_3 \rightarrow \text{Mg(OH)}_2 + 2\text{NaNO}_3 + \text{CO}_2 \]

The use of a stoichiometric quantity of sodium carbonate or the like is usually to be preferred, but satisfactory results can be obtained in some cases, whether the quantities used are in a stoichiometric relation or not. By “satisfactory results” we mean obtaining by reaction in an aqueous medium an appreciable yield of a fine-particled spinel having a desired composition and good high-temperature
stability. If the departure from the quantities dictated by stoichiometry is not too great, an appreciable yield of the desired spinel can be obtained, even though the process is operated somewhat wastefully in respect to its use of carbonate or of metal salt. It is necessary to use at least enough of sodium carbonate to precipitate both the iron and a substantial proportion of the other metal, such as zinc.

In the practice of the present Invention, obtaining particles of the desired size is an important consideration. The particle size is in part dependent upon the degree of dilution of the reactant solutions employed; the use of solutions more dilute yields particles of finer size, other things being equal.

It will be understood that the oxidation may be done conveniently, in most cases, by aeration at about room temperature, i.e., approximately 15 to 35°C.

Oxidation can be performed in any suitable manner. One way is aeration (spraying the suspension-containing solution into air from a perforated pipe). Another is bubbling air or a mixture of oxygen and inert gas through the suspension-containing solution. Another is shaking the suspension-containing solution in a vessel along with air or a mixture of oxygen and an inert or unreactive gas. Still another possibility is the use of a suitable chemical oxidizing agent, such as hydrogen peroxide or any of a number of chemical oxidants which can provide oxygen but will not (as potassium permanganate or sodium dichromate would) tend to impart any colour to the solution. The oxidation action which is required is one that it is not practicable to obtain by allowing the solution to stand while exposed to air or an atmosphere of oxygen. Oxidation can be monitored by titration to determine when it has been completed.

The solution is then heated to a temperature of from about 75°C to about 100°C. It is desirable to maintain the high temperature for a time long enough to “set the crystal”, i.e., ensure the development of desired crystalline forms in the product. X-ray diffraction tests can be used to monitor the progress and ensure the completion of the development of the desired crystalline forms.

The spinel is then recovered by conventional filtration and washing techniques.

The spinel thus obtained has a very fine particle size ranging from 0.01 to 0.13 μm. The performance of pigments containing the spinel particles is dependent upon their having particles of the size indicated. In general, a fusion process for obtaining spinels results in particles substantially larger than those obtained with the present invention. Fine particles made according to the present invention give purer colors than larger particles, made by a fusion process, do.

In further description of the temperature-stable spinel product made in accordance with the invention, it may be stated that a procedure of the kind detailed above gives particles which are of acicular (needle-like) shape and, as is revealed in the drawings, of a length which is one the order of 0.01 to 0.13 μm. Figure 1 corresponds to the product of Example III, below, and Figure 2 is a photomicrograph of a similar product, but one made by starting with zinc sulfate instead of zinc nitrate. Figure 3 shows the effect of calcination upon the product shown in Figure 1; the particles are no longer acicular.

The photomicrographs reveal that the process discussed above yields a product different from any encountered in the above-mentioned U.S. Patents. The particles of U.S. Patent No. 2,904,395 are said to be acicular, but with a length of 0.4 to 1.0 μm, as compared with about 0.01 to 0.13 μm for the particles shown in Figures 1 and 2. That the materials shown in Figures 1 and 2 are spinels has been confirmed by X-ray diffraction tests. The same materials exhibit an absence of the characteristic endotherm in differential thermal analysis at a temperature around 260 to 277°C. Such endotherm is characteristic of phase transformation of yellow alpha-FeOOH to red α-Fe₂O₃. This means that the materials in Figures 1 and 2 are spinels and are not alpha-FeOOH.

U.S. Patent No. 3,832,455 does not provide information concerning the size and shape of the particles produced by its teachings, but its method involves a necessary calcining step, and in view of Figure 3, it appears unlikely that it yields a product containing acicular particles. U.S. Patent No. 3,887,479 similarly involves use of high temperatures and does not contain information concerning the morphology of its product particles. The differences between the particles of U.S. Patent No. 3,822,210 and those of the present invention are apparent from a consideration of Figures 1 to 3 hereof and the photomicrographs in that patent, allowance being made for the difference in scales between them and Figures 1 to 3.

After they have been obtained as indicated above, the fine-particled spinels made according to the invention may be incorporated in plastics as a pigment to produce a desired yellow, by using proportions and practices well known to a person of ordinary skill in the art.

In practicing the present invention, the spinels are preferably derived from iron and Mg, Zn, or mixtures thereof. Such spinels show temperature stability up to about 900°C. Particularly good results have been obtained with the iron-zinc spinels, which are preferred.

Those skilled in the art will know how to incorporate the spinel pigments into plastics or the like. The necessary proportions, procedures, and equipment do not require explanation.

A comparison of weight loss versus temperature between a yellow iron oxide pigment of the
prior art and an iron-zinc spinel of the present invention establishes that the spinel has less water, present as hydroxyl, than the pure iron oxide pigment. This is shown in Table I, below.

<table>
<thead>
<tr>
<th>Temp., °C</th>
<th>Iron-zinc spinel</th>
<th>Iron oxide</th>
</tr>
</thead>
<tbody>
<tr>
<td>100</td>
<td>2.7</td>
<td>1.9</td>
</tr>
<tr>
<td>250</td>
<td>6.2</td>
<td>7.2</td>
</tr>
<tr>
<td>350</td>
<td>7.7</td>
<td>13.7</td>
</tr>
<tr>
<td>400</td>
<td>8.1</td>
<td>14.0</td>
</tr>
</tbody>
</table>

The chemical compositions of the spinels hereof generally correspond to the structure XFe$_2$O$_4$ where X is a metal as denoted above. As hereinafter noted, mixtures of metals can be utilized. However, electroneutrality in the crystal must be maintained.

Following are specific examples illustrating the principles of the present invention. These examples are to be construed as illustrating and not limiting the present invention.

Example I

Into a suitable reaction vessel equipped with titration means, cooling means, aeration means and agitation means, there was added a three liter solution of 27.8 grams per liter (gpl) of FeSO$_4$·7H$_2$O and 17 gpl Mg(NO$_3$)$_2$·6H$_2$O. With stirring, 200 milliliters of 231 gpl Na$_2$CO$_3$ solution was added thereto. The temperature in the vessel at the time of carbonate addition was 19°C.

With stirring, a precipitate is formed in the flask. After precipitation ceased, air was bubbled into the flask via the aeration means. Contemporaneous with aeration, the oxidation of the ferrous sulfate was monitored by titration with potassium permanganate.

After aeration was completed, the precipitated solution was heated to 90°C and maintained thereat for two hours, then cooled to room temperature. The precipitate was then recovered by filtration of the solution. The precipitate was then washed and dried. A temperature-stable yellow pigment was thus obtained. In other words, the pigment displayed, upon being subjected to differential thermal analysis an absence of the transformation from goethite to hematite of the kind discussed herein above. The pigment also has been tested by being subjected to elevated temperatures, up to about 900°C, showing no change of color.

Example II

Example I was repeated using an iron-metal solution of 27.8 gpl FeSO$_4$·7H$_2$O and 19.8 gpl Zn(NO$_3$)$_2$·6H$_2$O, to which was added 46 gms of Na$_2$CO$_3$ solution. A yellow pigment which was temperature-stable was obtained.

Example III

A solution containing 67 lbs. (30.4 kg) of ferrous sulfate heptahydrate and 47 lbs (21.3 kg) of Zn(NO$_3$)$_2$·6H$_2$O was prepared in 150 gallons (567.8 liters) of water maintained at 20°C. To this solution was added a solution of 37 lbs (16.8 kg) of Na$_2$CO$_3$ dissolved in 19.3 gallons (73 liters) of water. The reaction mixture was aerated at 3 cubic feet per minute (84.9 liters per minute) of air until maximum oxidation of the ferrous ion occurred, as determined by potassium permanganate titration. The reaction mixture was then heated to 90°C, filtered, washed and dried. A temperature-stable yellow pigment was thus obtained.

Example IV

Example III was repeated, except that 46 lbs (20.9 kg) of zinc sulphate heptahydrate were used in place of the indicated quantity of zinc nitrate hexahydrate. Once again, a temperature-stable yellow pigment was thus obtained.

Example V

Samples of each of the pigments of Examples I—IV hereof were heated for 1/2 hour at 280°C in an oven. Each sample remained yellow in color.

When heated to 800°C for 1/2 hour, the samples still remained yellow.

In the claims, the term “transparent pigment” is used to indicate a pigment which is capable of being incorporated in a vehicle to provide a coating which is substantially transparent to visible light. This implies that the particles of pigment have a size less than the wavelength of visible light.

Claims

1. A spinel in the form of acicular particles having a length of 0.01 to 0.13 µm and consisting essentially of oxygen, iron, and at least one other metal, said spinel corresponding to the formula

   XFe$_2$O$_4$

   wherein X is said other metal, said other metal being one selected from the group consisting of magnesium, zinc, tin, and mixtures thereof.

2. A spinel as defined in Claim 1, wherein said other metal is one selected from the group consisting of magnesium, zinc, and mixtures thereof.

3. A spinel according to Claim 1, wherein the other metal is magnesium.

4. A spinel according to Claim 1, wherein the other metal is zinc.

5. A method for making a plastic having a yellow hue comprising incorporating into said
plastic as a transparent pigment a spinel in accordance with Claim 4.

6. A method for making a plastic having a yellow hue comprising incorporating into said plastic as a pigment a spinel in accordance with Claim 1.

7. A method of making a transparent yellow pigment having good stability at high temperatures, said method consisting essentially of the steps of:

- forming a first aqueous solution consisting essentially of water, a soluble ferrous salt, and at least one other salt, said salt being a soluble salt of a non-ferrous metal selected from the group consisting of magnesium, zinc, and tin, said ferrous salt and said non-ferrous salt being present in said solution in proportions such that the respective quantities of iron and other metal which are present will yield, when said solution is so adjusted in pH as to cause precipitation, the formation of a substantial quantity of a precipitate oxidizable to a spinel of the formula \( XFe_2O_4 \), in which \( X \) is a metal selected from the group consisting of magnesium, zinc, and tin

- mixing with said first aqueous solution a second aqueous solution to form a reaction mixture, said second solution containing a substantial proportion of a soluble alkali-metal compound selected from the group consisting of the carbonates, bicarbonates, and hydroxides of the alkali metals, the said second solution being used in such quantity and the said first and second solutions being in such a state of dilution that there results upon said mixing the formation of a precipitate in the form of particles of sub-micron size, the liquid phase of said reaction mixture being as a result of such precipitation substantially completely depleted in its content of metal ions having a valence greater than one;

- oxidizing at 15 to 35°C the said precipitate to form a spinel in aqueous solution;

- then heating said aqueous solution having said spinel therein to a temperature of from about 75°C to 100°C, and recovering said spinel.

8. A method as defined in Claim 7, wherein said soluble ferrous salt is a hydrated salt.

9. A method as defined in Claim 7, wherein said other metal is zinc.

10. A method as defined in Claim 7 or 9, wherein said alkali-metal compound is sodium carbonate.

11. A method as defined in Claim 7, wherein the step of oxidizing is done by subjecting the precipitate to the action of aqueous elemental oxygen by aeration.

Patentansprüche

1. Ein Spinell in Form nadelförmiger Teilchen mit einer Länge von 0,01 bis 0,13 µm, der im wesentlichen aus Sauerstoff, Eisen und mindestens einem anderen Metall besteht und der Formel

\[ XFe_2O_4 \]

entspricht, in der X das weitere Metall bedeutet, wobei dieses Metall Magnesium, Zink, Zinn oder eine Mischung aus diesen Metallen ist.

2. Ein Spinell nach Anspruch 1, dadurch gekennzeichnet, daß das weitere Metall Magnesium, Zink oder eine Mischung aus diesen Metallen ist.

3. Ein Spinell nach Anspruch 1, dadurch gekennzeichnet, daß das weitere Metall Magnesium ist.

4. Ein Spinell nach Anspruch 1, dadurch gekennzeichnet, daß das weitere Metall Zink ist.

5. Verfahren zur Herstellung eines Kunststoffs mit gelbem Farbton, dadurch gekennzeichnet, daß man dem Kunststoff als transparentes Pigment einen Spinell nach Anspruch 4 zusetzt.


7. Verfahren zur Herstellung eines transparenten gelben Pigments mit guter Hochtemperaturbeständigkeit, das im wesentlichen aus folgenden Schritten besteht:

- Herstellung einer ersten wäßrigen Lösung, die im wesentlichen besteht aus Wasser, einem löslichen Eisen(II)-salz und mindestens einem weiteren Salz, das ein lösliches Salz eines Nichteisenmetalls aus der Gruppe Magnesium, Zink und Zinn ist, wobei das Eisen(II)-salz und das Nichteisensozal in solchen Mengen in der Lösung enthalten ist, daß die jeweiligen Mengen an Eisen und Nichteisenmetall, wenn das pH-Wert der Lösung so eingestellt wird, daß eine Fällung eintritt, die Bildung einer beträchtlichen Menge eines Niederschlags ergeben, der zu einem Spinell der Formel \( XFe_2O_4 \) oxidiert werden kann, in der X eines der Metalle Magnesium, Zink oder Zinn ist;

- Mischen der ersten wäßrigen Lösung mit einer zweiten wäßrigen Lösung zu einer Reaktionsmischung, wobei die zweite Lösung eine beträchtliche Menge einer löslichen alkalisierenden Verbindung aus der Gruppe der Carbonate, Bicarbonate und Hydroxide der Alkalimetalle enthält und wobei die zweite Lösung in einer solchen Menge verwendet wird und jede Lösung in solcher Verdünnung vorliegen, daß sich bei diesem Vermischen ein Niederschlag in Form von Teilchen mit einer Größe von weniger als 1 µm bildet und der Gehalt der flüssigen Phase der Reaktionsmischung an Metallen mit einer Wertigkeit von mehr als 1 infolge dieser Ausfällung im wesentlichen vollständig erschöpft ist;

- Oxidieren des Niederschlags bei 15 bis 35°C zu einem in wäßriger Lösung vorliegenden Spinell,

- Erhitzen dieser wäßrigen, den Spinell enthaltenden Lösung auf eine Temperatur von etwa 75°C bis 100°C, und

- Isolieren des Spinells.
8. Verfahren nach Anspruch 7, dadurch gekennzeichnet, daß das lösliche Eisen(II)-salz ein hydratisiertes Salz ist.
10. Verfahren nach Anspruch 7 oder 9, dadurch gekennzeichnet, daß man als alkalisierte Verbindung Natriumcarbonat verwendet.

Revendications

1. Une spinelle sous forme de particules aciculaires d’une longueur de 0,01 à 0,13 μm et constituée essentiellement d’oxygène, de fer et d’au moins un autre métaux, cette spinelle correspondant à la formule

\[ XFe_2O_4 \]

où X est un métal choisi dans le groupe consistant en magnésium, zinc, étain et leurs mélanges.
2. Une spinelle telle que définie à la revendication 1, dans laquelle ledit autre métal est choisi dans le groupe constitué par le magnésium, le zinc et leurs mélanges.
3. Une spinelle selon la revendication 1, où l’autre métal est le magnésium.
4. Une spinelle selon la revendication 1, où l’autre métal est le zinc.
5. Un procédé de fabrication d’une matière plastique ayant une teinte jaune, selon lequel on incorpore dans cette matière plastique, en tant que pigment transparent, une spinelle selon la revendication 4.
6. Un procédé de fabrication d’une matière plastique ayant une teinte jaune, selon lequel on incorpore dans cette matière plastique, en tant que pigment une spinelle selon la revendication 1.
7. Un procédé de préparation d’un pigment jaune, transparent, ayant une bonne stabilité aux températures élevées. Ce procédé consistant essentiellement en les opérations suivantes:

formation d’une première solution aqueuse constituée essentiellement d’eau, d’un sel ferreux soluble, et d’au moins un autre sel, ce sel étant un sel soluble d’un métal non ferreux choisi dans le groupe constitué par le magnésium, le zinc et l’étain, ledit sel ferreux et ledit sel non ferreux étant présents dans ladite solution en proportions telles que les quantités respectives de fer et d’autre métal présent, conduisent, quand ladite solution est réglée en pH de telle sorte qu’il y ait précipitation, à la formation d’une quantité notable d’un précipité oxydable en une spinelle de formule \( XFe_2O_4 \), où X est un métal choisi dans le groupe constitué par le magnésium, le zinc et l’étain; mélange avec ladite première solution aqueuse d’une seconde solution aqueuse pour former un mélange de réaction, ladite seconde solution contenant une proportion notable d’un composé alcalinant soluble choisi dans le groupe constitué par les carbonates, les bicarbonates et les hydroxides des métaux alcalins, ladite seconde solution étant utilisée en quantité telle et lesdites première et seconde solutions étant dans un état de dilution tel qu’il en résulte, après ledit mélange, la formation d’un précipité sous forme de particules de la taille du micron, la phase liquide dudit mélange de réaction étant, par suite de cette précipitation, à peu près complètement privée de sa teneur en ions métal d’une valeur supérieure à un; oxydation entre 15 et 35°C dudit précipité pour former une spinelle en solution aqueuse; puis chauffage de ladite solution aqueuse contenant cette spinelle à une température d’environ 75° à 100°C; et récupération de cette spinelle.
8. Un procédé tel que défini dans la revendication 7, dans lequel ledit sel ferreux soluble est un sel hydraté.
9. Un procédé tel que défini dans la revendication 7, dans lequel ledit autre métal est le zinc.
10. Un procédé tel que défini dans la revendication 7 ou 9, dans lequel ledit composé alcalinant est du carbonate de sodium.
11. Un procédé tel que défini dans la revendication 7, dans lequel l’opération d’oxydation est effectuée en soumettant le précipité à l’action d’oxygène élémentaire aqueux par aération.