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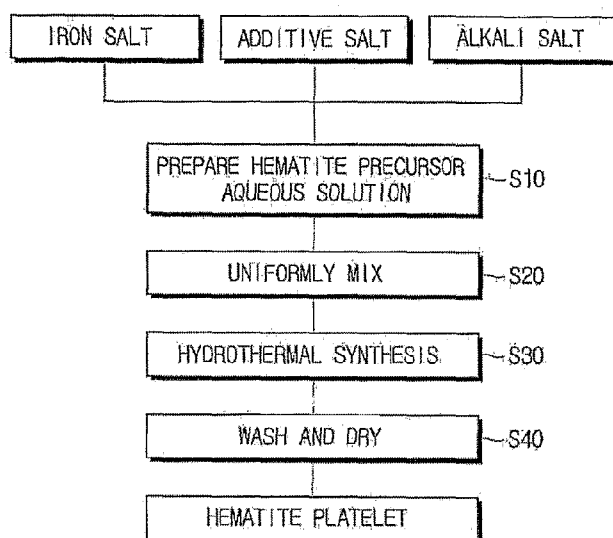
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(54) Title: METHOD OF PREPARING HEMATITE PLATELET, HEMATITE PLATELET PREPARED USING THE SAME, AND HEMATITE PLATELET PIGMENT INCLUDING THE HEMATITE PLATELET

[Fig 1]



(57) Abstract: Provided are a method of preparing a hematite platelet, a hematite platelet prepared using the method, and a hematite platelet pigment including the hematite platelet. The method of preparing a hematite platelet includes: preparing a hematite precursor aqueous solution comprising iron(III) salt; an aqueous alkali solution; and an additive comprising alkali-based metal salts; uniformly mixing the hematite precursor aqueous solution to obtain an intermediate product comprising Fe +3 and at least one of K<sup>+</sup>, Na<sup>+</sup>, Li<sup>+</sup>, and Ca<sup>+</sup>; and hydrothermally treating the hematite precursor aqueous solution. In addition, the hematite platelet pigment is prepared by using the hematite platelet prepared using the preparation method as a base and coating a metal oxide on the base. Moreover, a hematite platelet having a particle size of 1~100 $\mu$ m, a particle thickness of 0.3~4 $\mu$ m, and an aspect ratio of 20 or greater can be prepared.



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## Description

# METHOD OF PREPARING HEMATITE PLATELET, HEMATITE PLATELET PREPARED USING THE SAME, AND HEMATITE PLATELET PIGMENT INCLUDING THE HEMATITE PLATELET

### Technical Field

- [1] The present invention relates to a method of preparing a hematite platelet, a hematite platelet prepared using the same, and a hematite platelet pigment including the hematite platelet, and more particularly, to a method of preparing a hematite platelet that is suitable for use as a metallic pigment and has an aspect ratio of 20 or greater, a hematite platelet prepared using the same, and a hematite platelet pigment including the hematite platelet.

[2]

### Background Art

- [3] Hematite is the most common type of naturally occurring iron oxide mineral, and has been used as a colorant for centuries. Examples of hematites include hematites, pyrites, and magnetites, which are respectively red-colored, yellow-colored, and black-colored. Wall paintings painted with hematites remain in caves all over the world, and retain their original color tones. Currently, these hematites are mostly prepared as synthetic products, and thus are used in various fields as pigments having clear color tones and excellent durability, being inexpensive and having low toxicity and high stability. In this regard, hematites are applied to a variety of products, such as paints, inks, rubbers, plastics, paper, glass, cement, and the like. Further, high purity hematites are not harmful to humans, and thus can be used in foods, medicines, and cosmetics.
- [4] In particular, well-known synthetic hematite pigments include red or red brown-colored hematite particle powder ( $\alpha$ -Fe<sub>2</sub>O<sub>3</sub> or micaceous iron oxide (MIO)), yellow or deep brown-colored maghemite ( $\gamma$ -Fe<sub>2</sub>O<sub>3</sub>) particle powder, and black-colored magnetite (FeO $\cdot$ xFe<sub>2</sub>O<sub>3</sub> where 0<x $\leq$ 1).
- [5] Among these hematite pigments, a hematite pigment (hereinafter, referred to as "hematite platelet") in the form of a platelet, in particular, hematite is formed as a thin plate having a thickness of several  $\mu$ m to 10  $\mu$ m due to the shape of hematite, and thus can be arranged in the form of layers during coating. Thus, hematite platelet has excellent coating properties (protection from the external environment). Therefore,

hematite platelet can be used as a rust inhibiting pigment for steel structures, paint for protecting woods, and a print ink for plywood. Also, hematite that has a small particle size and is transparent is used as a coloring pigment for metallic paints, for example, vehicles, by using its metallic gloss properties. In addition, hematite has ultraviolet-ray absorbing properties and covering properties with respect to skin (skin covering effect), and thus is useful as a material for forming films for packaging foods, colorants for medicine containers, and an extender for cosmetics.

- [6] In particular, metallic pigments such as extenders for cosmetics may have sliding properties and adhesiveness, thereby facilitating good skin filling, and inhibiting excessive gloss, and thus prevents occurrence of unwanted gloss by reflective light, and have appropriate covering effects based on semi-transparency and natural color tones with respect to the skin. Metallic pigments, in particular, should have low gloss by adjusting gloss properties, and also have color tones by interference. Thus, to have these properties, metallic pigments should have a particle size of at least 1  $\mu\text{m}$  or greater, and an aspect ratio, which is defined as a ratio of particle size to thickness of pigment particles, of at least 10 or greater.
- [7] A conventional hematite platelet is prepared using a dry method or a wet method. Examples of the wet method include a melting method and a hydrothermal synthesis method. The dry method generates large amounts of sulfur dioxide gases and sulfuric acid gases, thus requiring expensive antipollution measures. Therefore, the dry method is rarely used. In the melting method, which is one of the wet methods, an iron sulfate or iron chloride solution reacts with alkali, thereby producing magnetite or goethite, and the resultant is sintered to obtain hematite platelets.
- [8] As an example of the melting method, Korean Patent Publication No. 1992-11922 discloses a method of  $\omega$ -precipitating a barium ion and an iron ion, and then heat treating the resultant. In this method, the barium ion and the iron ion are  $\omega$ -precipitated in the form of hydroxide or carbonate, and heat treated at a temperature of 900°C or greater to prepare hexagonal plate-shaped barium ferrite particulates having a size of 0.2~0.5  $\mu\text{m}$ .
- [9] In addition, US 4,867,795 discloses a method of preparing hematite in the form of which Mn or Al is employed by synthesizing  $\text{FeSO}_4$  as a raw material and a precursor comprising  $\text{Al}(\text{OH})_3$ ,  $\text{KMnO}_4$ , and  $\text{NaAlO}_2$  in an autoclave. The hematite prepared using this method has a good aspect ratio, i.e., 20 or greater. However, the synthesis temperature is over 800°C.
- [10] As an example of the hydrothermal synthesis method, Japanese Patent Laid-open No.

Sho 55-154319 discloses a method of economically preparing  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub> by hydrothermally treating iron oxide such as iron ore as a raw material with an oxidant in an aqueous alkali solution via magnetite.

[11] In addition, EPA0180881 discloses a method of preparing  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub> by using Fe(NO<sub>3</sub>)<sub>3</sub>·9H<sub>2</sub>O as a raw material by hydrothermal synthesis.

[12] In addition, Japanese Patent Laid-open No. Sho 58-69730 discloses a method of efficiently preparing particles in the form of platelet by hydrothermally treating  $\gamma$ -Fe<sub>2</sub>O<sub>3</sub> alone in an aqueous alkali solution.

[13] In addition, Japanese Patent Laid-open No. Hei 6-166520 discloses a method of preparing hematite platelet particulates in which aluminum is employed, the method including: mixing an aqueous iron(II) salt solution, an aqueous aluminum salt solution and an aqueous carbonate salt solution to obtain hydrotalcite-type particulates in the form of platelet, comprising Fe<sup>2+</sup> and Al<sup>3+</sup>, and hydrothermally treating the particulates.

[14]

## **Disclosure of Invention**

### **Technical Problem**

[15] According to the melting method described above, hematite particles having a desired aspect ratio can be obtained, and can be mass-produced. However, dispersibility of the hematite particles is not good in a solution, and thus it is difficult to adjust the size of hematite particles, it is difficult to obtain particles in the form of platelet, and the sintering process is performed at a temperature of at least 800°C or greater. Thus, particles are sintered with each other, and this method is non-economical. In particular, barium ferrite platelets have magnetic properties, and thus are not suitable as a pearl pigment.

[16] According to the hydrothermal synthesis method, hematite platelets can be prepared at a relatively low temperature, particles are not sintered with each other, and it is easy to adjust the size of particles. The hydrothermal synthesis method is mainly suitable for preparing nano-sized hematite particles, but is not suitable for preparing metallic pigments such as pigments for cosmetics, which should have micro-sized particles or more.

[17] In particular, the hematite platelet prepared using this method has a small aspect ratio, i.e., in a range of 2~10, and thus cannot satisfy requirements for a metallic pigment or a pearl pigment, in terms of, for example, optical properties and adhesiveness.

[18]

### **Technical Solution**

[19] The present invention provides a method of preparing a hematite platelet using a hydrothermal synthesis method, whereby the hematite platelet has an aspect ratio of 20 or greater in order to have low gloss and color tone by interference, which are properties suitable as a metallic pigment, and a hematite platelet prepared using the method.

[20] The present invention also provides a hematite platelet pigment including the hematite platelet.

[21]

### **Advantageous Effects**

[22] According to the present invention, a hematite platelet having a particle size of 1~100 $\mu\text{m}$ , a particle thickness of 0.3~4 $\mu\text{m}$ , and an aspect ratio of 20 or greater can be prepared.

[23]

### **Brief Description of the Drawings**

[24] The above and other features and advantages of the present invention will become more apparent by describing in detail exemplary embodiments thereof with reference to the attached drawings in which:

[25] FIG. 1 is a flowchart sequentially illustrating a method of preparing a hematite platelet, according to an embodiment of the present invention;

[26] FIG. 2 is a scanning electron microscopic (SEM) image of a side surface of hematite platelet particles prepared in Experimental Example 1-4 ;

[27] FIG. 3A is a SEM image of a top surface of the hematite platelet particles of FIG. 2;

[28] FIG. 3B is an optical microscopic image of a top surface of the hematite platelet particles of FIG. 2;

[29] FIG. 4 illustrates particle size distribution of the hematite platelet of FIG. 2;

[30] FIG. 5 illustrates X-ray diffraction (XRD) patterns of the hematite platelet powder of FIG. 2;

[31] FIG. 6 is a SEM image of a side surface of hematite platelet particles prepared in Experimental Example 2-1;

[32] FIG. 7 is a SEM image of a side surface of hematite platelet particles prepared in Experimental Example 3-4;

[33] FIG. 8 is a transmission electron microscopic (TEM) image of particle size and shape of a nanoparticle hematite seed;

[34] FIG. 9 is a SEM image of a top surface of hematite platelet particles prepared using the hematite seed of FIG. 8;

[35] FIG. 10 is a SEM image of a side surface of hematite platelet particles of FIG. 2 on which  $\text{TiO}_2$  is coated.

[36]

### **Best Mode for Carrying Out the Invention**

[37] The present invention will now be described more fully with reference to the accompanying drawings, in which exemplary embodiments of the invention are shown.

[38] FIG. 1 is a flowchart sequentially illustrating a method of preparing a hematite platelet, according to an embodiment of the present invention. Referring to FIG. 1, the method of preparing a hematite platelet includes: preparing a hematite precursor aqueous solution comprising iron(III) salt; an aqueous alkali solution; and an additive comprising at least one selected from the group consisting of potassium-based salts including potassium phosphate, potassium sulfate, potassium chloride, potassium nitrate, potassium carbonate, potassium hydrogen carbonate, potassium bromide, and potassium fluoride, sodium-based salts having the same anion as that of the potassium-based salts, lithium-based salts, and calcium-based salts (operation S10); uniformly mixing the hematite precursor aqueous solution to obtain an intermediate product comprising  $\text{Fe}^{+3}$  and at least one of  $\text{K}^+$ ,  $\text{Na}^+$ ,  $\text{Li}^+$ , and  $\text{Ca}^+$  (operation S20); and hydrothermally treating the hematite precursor aqueous solution (operation S30).

[39] The iron(III) salt used herein is not particularly limited, and can be any salt containing  $\text{Fe}^{+3}$  ions. Preferably, the iron(III) salt may be iron (III) chloride ( $\text{FeCl}_3$ ), iron(III) sulfate ( $\text{Fe}_2(\text{SO}_4)_3$ ), or iron(III) nitrate ( $\text{Fe}(\text{NO}_3)_3$ ) and these salts can be used alone or in mixtures thereof.

[40] The aqueous alkali solution may be any one of potassium hydroxide, sodium hydroxide, calcium hydroxide, and lithium hydroxide, which are general aqueous alkali solutions, or a mixture of at least two of these materials. The aqueous alkali solution is generally used in a wet preparation method of hematite, and, in the present invention, the aqueous alkali solution adjusts pH and alkali-neutralizes the iron(III) salt.

[41] The hematite precursor aqueous solution may have a pH of 11 or greater, and preferably a pH of 13 or greater in order to obtain the intermediate product comprising  $\text{Fe}^{+3}$  and at least one of  $\text{K}^+$ ,  $\text{Na}^+$ ,  $\text{Li}^+$ , and  $\text{Ca}^+$  as an additive, which will be described later. If the pH of the hematite precursor aqueous solution is less than 11, it is difficult to produce the intermediate product.

- [42] In addition, in operations S10 and S20, the aqueous alkali solution may be mixed by appropriately changing the amount of aqueous alkali solution. The aqueous alkali solution added in operation S10 reacts with  $\text{Fe}^{+3}$  to produce an initial product of  $\text{Fe}(\text{OH})_3$ , whereas the aqueous alkali solution added in operation S20 reacts with a cation of the additive to produce an intermediate product of alkali-based hematite. Thus, the aqueous alkali solutions of operations S10 and S20 are distinguished from each other.
- [43] In operation S20, the amount of aqueous alkali solution added may be in a range of 80~300 parts by weight, and preferably in a range of 100~200 parts by weight, with respect to 100 parts by weight of the iron(III) salt, and in operation S10, the amount of aqueous alkali solution added may be in a range of 30~900 parts by weight, and preferably in a range of 50~600 parts by weight, with respect to 100 parts by weight of the iron(III) salt, in order to produce each of the initial product and the intermediate product.
- [44] In addition, the additive of the present invention is ionized in a mixed solution as in the form of at least one of  $\text{K}^+$ ,  $\text{Na}^+$ ,  $\text{Li}^+$ , and  $\text{Ca}^+$ . The additive is employed in the form of amorphous alkali-based hematite, constituting the intermediate product, for example,  $\text{K}_x\text{Fe}_y\text{O}_z$  where x and y are each an integer in the range of 1~2, and z is an integer in the range of 1~4, together with  $\text{Fe}^{+3}$  ions of the iron(III) salt, or employed in nano-sized  $\alpha\text{-Fe}_2\text{O}_3$ , constituting a final product. Thus, the additive acts as a seed in crystallization of hematite platelet particles. In addition, more particularly, the hematite mixture  $\text{K}_x\text{Fe}_y\text{O}_z$  reacts with the final product, nano-sized  $\alpha\text{-Fe}_2\text{O}_3$  to grow the hematite platelet particles. As an amount of an alkali-based metal salt comprising at least one ion of  $\text{K}^+$ ,  $\text{Na}^+$ ,  $\text{Li}^+$ , and  $\text{Ca}^+$  is increased, the aspect ratio of the hematite platelet particles (particle size/thickness) is constant and the size thereof is increased. In addition, according to a processing temperature and processing method, colors of hematite platelet pigment can be various. In addition, hematite platelet crystals having a variety of aspect ratios may be prepared according to the amount of the additive in the form of neutral alkali-based metal salts.
- [45] That is, after the additive is oxidized, the additive adheres to the crystal surface of the hematite platelet in the crystallization process, thereby facilitating the growth of the hematite platelet in a size direction and relatively inhibiting the growth of the hematite platelet in a thickness direction. Thus, compared with the case when hematite platelet is formed alone, the additive forms hematite platelet crystals having similar size and effectively decreased thickness.



- [46] The amount of additive in the form of neutral alkali-based metal salts may be in a range of 0.01~4 parts by weight, and preferably in a range of 0.05~4 parts by weight, with respect to 100 parts by weight of the iron (III) salt. If the amount of additive is less than 0.01 parts by weight with respect to 100 parts by weight of the iron (III) salt, it is difficult to obtain an aspect ratio of 10 or greater. On the other hand, if the amount of additive is greater than 4 parts by weight with respect to 100 parts by weight of the iron (III) salt, the size of the obtained hematite platelet is small.
- [47] In addition, the additive may be di-ethanol amine, tri-ethanol amine, a chelate organic compound, or the like, comprising  $K^+$ ,  $Na^+$ ,  $Li^+$ , or  $Ca^+$ , in addition to the potassium-based salt, the sodium-based salt, the lithium-based salt, and the calcium-based salt described above.
- [48] The hematite platelet may be prepared by adding uniform nanoparticle hematite seed having a size of 1~50 nm to the hematite precursor aqueous solution in operation S10. This method comprises growing hematite platelet crystals by adding nanometer-sized hematite particles as a seed of crystallization in an initial stage of the process.
- [49] The amount of hematite seed added may be in a range of 0.001~1 parts by weight, and preferably in a range of 0.005~0.2 parts by weight, with respect to 100 parts by weight of the iron(III) salt. When the amount of hematite seed added is less than 0.001 parts by weight with respect to 100 parts by weight of the iron(III) salt, effects on the aspect ratio and dispersion of hematite platelet are insignificant. On the other hand, when the amount of hematite seed added is greater than 1 part by weight with respect to 100 parts by weight of the iron(III) salt, hematite particles are too small to easily obtain hematite platelet having an effective aspect ratio.
- [50] Operation S20, comprising uniformly mixing the hematite precursor aqueous solution, may be performed by stirring the obtained hematite mixed solution at a rotating speed of 200 rpm or greater, or using one of a homogenizer, a ball miller, a static mixer, ultrasonic waves, and a microwave device, or using at least two of these devices in combination.
- [51] Operation S30 may be performed using an autoclave device at a temperature in a range of 150~250°C for 2~240 hours. In general, the greater the concentration of alkali, the lower the reaction temperature that produces hematite platelet particles. When the reaction temperature is less than 150°C, it is difficult to obtain hematite platelet particles. On the other hand, when the reaction temperature is greater than 250°C, the hematite platelet particles can be obtained, but in order for stable operation of a hydrothermal treatment device and economically preparing hematite platelet, an

appropriate upper limit temperature may be 250°C.

- [52] In operation S30, the intermediate product produced in operation S20 acts as a seed of crystal, thereby producing hematite platelet ( $\alpha\text{-Fe}_2\text{O}_3$ ) crystal in which K, Na, Li, or Ca is employed by hydrothermal reaction.
- [53] In addition, after operation S30, the method of preparing a hematite platelet may further include filtering, washing and drying the hematite platelet particles (operation S40) in order to obtain hematite platelet particles having a desired size and thickness.
- [54] The size of hematite platelet particles prepared may be in a range of 1~100  $\mu\text{m}$ , and preferably in a range of 5~80  $\mu\text{m}$ . When the size of the particles is less than 1  $\mu\text{m}$ , reflectance is decreased. On the other hand, when the size of the particles is greater than 100  $\mu\text{m}$ , the hematite platelet particles are not suitable as a metallic pigment.
- [55] In addition, the hematite platelet may have an aspect ratio of 20 or greater, and preferably an aspect ratio of 25 or greater. When the aspect ratio of the hematite platelet is less than 20, it is difficult to produce interference colors as an optical property and adhesiveness of particles is decreased, resulting in poor use thereof. An upper limit of the aspect ratio is not limited, but when the aspect ratio of the hematite platelet is greater than 100, strong particle gloss may be realized.
- [56] A hematite platelet pigment with metallic gloss in which the synthesized hematite platelet is used as a base and metal oxide is coated on the base is manufactured to prepare, for example, a pearl pigment with strong metallic effect.
- [57] The hematite platelet pigment may be prepared, for example, by suspending the hematite platelet obtained using the preparation method as described above in deionized water, adding an acid such as hydrochloric acid or sulfuric acid to the resultant and stirring, simultaneously adding a metal chloride solution and an aqueous alkali solution to the aqueous solution and refluxing the resultant to prepare a hematite platelet particle aqueous solution coated with a metal oxide hydrate layer, and filtering, washing, drying and sintering the hematite platelet particle aqueous solution.
- [58] The metal oxide coated on the surface of the hematite platelet is a metal oxide typically coated on the surface of pigment and used to produce metallic gloss and interference colors in the field of preparing metallic pigments. The metal oxide is not particularly limited, and may be, in particular,  $\text{SnO}_2$ ,  $\text{TiO}_2$ ,  $\text{ZrO}_2$ ,  $\text{SiO}_2$ ,  $\text{Al}_2\text{O}_3$ ,  $\text{MgO}$ , or  $\text{MnO}_2$ , or at least two of these materials.
- [59] More preferably, it is desirable to form a multi-layer coating structure by using a variety of metal oxides. For example, such a multi-layer coating structure may include a coating layer formed of  $\text{SiO}_2$ , and a  $\text{TiO}_2$  coating layer formed on the surface of the

SiO<sub>2</sub> coating layer. The advantages of such a multi-layer coating structure are an increase in covering power and obtainment of high-quality interference colors.

[60]

### Mode for the Invention

[61] The present invention will now be described more fully with reference to the accompanying drawings, in which exemplary embodiments of the invention are shown.

[62] FIG. 1 is a flowchart sequentially illustrating a method of preparing a hematite platelet, according to an embodiment of the present invention. Referring to FIG. 1, the method of preparing a hematite platelet includes: preparing a hematite precursor aqueous solution comprising iron(III) salt; an aqueous alkali solution; and an additive comprising at least one selected from the group consisting of potassium-based salts including potassium phosphate, potassium sulfate, potassium chloride, potassium nitrate, potassium carbonate, potassium hydrogen carbonate, potassium bromide, and potassium fluoride, sodium-based salts having the same anion as that of the potassium-based salts, lithium-based salts, and calcium-based salts (operation S10); uniformly mixing the hematite precursor aqueous solution to obtain an intermediate product comprising Fe<sup>+3</sup> and at least one of K<sup>+</sup>, Na<sup>+</sup>, Li<sup>+</sup>, and Ca<sup>+</sup> (operation S20); and hydrothermally treating the hematite precursor aqueous solution (operation S30).

[63] The iron(III) salt used herein is not particularly limited, and can be any salt containing Fe<sup>+3</sup> ions. Preferably, the iron(III) salt may be iron(III) chloride (FeCl<sub>3</sub>), iron(III) sulfate (Fe<sub>2</sub>(SO<sub>4</sub>)<sub>3</sub>), or iron(III) nitrate (Fe(NO<sub>3</sub>)<sub>3</sub>) and these salts can be used alone or in mixtures thereof.

[64] The aqueous alkali solution may be any one of potassium hydroxide, sodium hydroxide, calcium hydroxide, and lithium hydroxide, which are general aqueous alkali solutions, or a mixture of at least two of these materials. The aqueous alkali solution is generally used in a wet preparation method of hematite, and, in the present invention, the aqueous alkali solution adjusts pH and alkali-neutralizes the iron(III) salt.

[65] The hematite precursor aqueous solution may have a pH of 11 or greater, and preferably a pH of 13 or greater in order to obtain the intermediate product comprising Fe<sup>+3</sup> and at least one of K<sup>+</sup>, Na<sup>+</sup>, Li<sup>+</sup>, and Ca<sup>+</sup> as an additive, which will be described later. If the pH of the hematite precursor aqueous solution is less than 11, it is difficult to produce the intermediate product.

[66] In addition, in operations S10 and S20, the aqueous alkali solution may be mixed by appropriately changing the amount of aqueous alkali solution. The aqueous alkali

solution added in operation S10 reacts with  $\text{Fe}^{+3}$  to produce an initial product of  $\text{Fe}(\text{OH})_3$ , whereas the aqueous alkali solution added in operation S20 reacts with a cation of the additive to produce an intermediate product of alkali-based hematite. Thus, the aqueous alkali solutions of operations S10 and S20 are distinguished from each other.

[67] In operation S20, the amount of aqueous alkali solution added may be in a range of 80~300 parts by weight, and preferably in a range of 100~200 parts by weight, with respect to 100 parts by weight of the iron(III) salt, and in operation S10, the amount of aqueous alkali solution added may be in a range of 30~900 parts by weight, and preferably in a range of 50~600 parts by weight, with respect to 100 parts by weight of the iron(III) salt, in order to produce each of the initial product and the intermediate product.

[68] In addition, the additive of the present invention is ionized in a mixed solution as in the form of at least one of  $\text{K}^+$ ,  $\text{Na}^+$ ,  $\text{Li}^+$ , and  $\text{Ca}^+$ . The additive is employed in the form of amorphous alkali-based hematite, constituting the intermediate product, for example,  $\text{K}_x\text{Fe}_y\text{O}_z$  where  $x$  and  $y$  are each an integer in the range of 1~2, and  $z$  is an integer in the range of 1~4, together with  $\text{Fe}^{+3}$  ions of the iron(III) salt, or employed in nano-sized  $\alpha\text{-Fe}_2\text{O}_3$ , constituting a final product. Thus, the additive acts as a seed in crystallization of hematite platelet particles. In addition, more particularly, the hematite mixture  $\text{K}_x\text{Fe}_y\text{O}_z$  reacts with the final product, nano-sized  $\alpha\text{-Fe}_2\text{O}_3$  to grow the hematite platelet particles. As an amount of an alkali-based metal salt comprising at least one ion of  $\text{K}^+$ ,  $\text{Na}^+$ ,  $\text{Li}^+$ , and  $\text{Ca}^+$  is increased, the aspect ratio of the hematite platelet particles (particle size/thickness) is constant and the size thereof is increased. In addition, according to a processing temperature and processing method, colors of hematite platelet pigment can be various. In addition, hematite platelet crystals having a variety of aspect ratios may be prepared according to the amount of the additive in the form of neutral alkali-based metal salts.

[69] That is, after the additive is oxidized, the additive adheres to the crystal surface of the hematite platelet in the crystallization process, thereby facilitating the growth of the hematite platelet in a size direction and relatively inhibiting the growth of the hematite platelet in a thickness direction. Thus, compared with the case when hematite platelet is formed alone, the additive forms hematite platelet crystals having similar size and effectively decreased thickness.

[70] The amount of additive in the form of neutral alkali-based metal salts may be in a range of 0.01~4 parts by weight, and preferably in a range of 0.05~4 parts by weight,

with respect to 100 parts by weight of the iron (III) salt. If the amount of additive is less than 0.01 parts by weight with respect to 100 parts by weight of the iron (III) salt, it is difficult to obtain an aspect ratio of 10 or greater. On the other hand, if the amount of additive is greater than 4 parts by weight with respect to 100 parts by weight of the iron (III) salt, the size of the obtained hematite platelet is small.

- [71] In addition, the additive may be di-ethanol amine, tri-ethanol amine, a chelate organic compound, or the like, comprising  $K^+$ ,  $Na^+$ ,  $Li^+$ , or  $Ca^+$ , in addition to the potassium-based salt, the sodium-based salt, the lithium-based salt, and the calcium-based salt described above.
- [72] The hematite platelet may be prepared by adding uniform nanoparticle hematite seed having a size of 1~50 nm to the hematite precursor aqueous solution in operation S10. This method comprises growing hematite platelet crystals by adding nanometer-sized hematite particles as a seed of crystallization in an initial stage of the process.
- [73] The amount of hematite seed added may be in a range of 0.001~1 parts by weight, and preferably in a range of 0.005~0.2 parts by weight, with respect to 100 parts by weight of the iron(III) salt. When the amount of hematite seed added is less than 0.001 parts by weight with respect to 100 parts by weight of the iron(III) salt, effects on the aspect ratio and dispersion of hematite platelet are insignificant. On the other hand, when the amount of hematite seed added is greater than 1 part by weight with respect to 100 parts by weight of the iron(III) salt, hematite particles are too small to easily obtain hematite platelet having an effective aspect ratio.
- [74] Operation S20, comprising uniformly mixing the hematite precursor aqueous solution, may be performed by stirring the obtained hematite mixed solution at a rotating speed of 200 rpm or greater, or using one of a homogenizer, a ball miller, a static mixer, ultrasonic waves, and a microwave device, or using at least two of these devices in combination.
- [75] Operation S30 may be performed using an autoclave device at a temperature in a range of 150~250°C for 2~240 hours. In general, the greater the concentration of alkali, the lower the reaction temperature that produces hematite platelet particles. When the reaction temperature is less than 150°C, it is difficult to obtain hematite platelet particles. On the other hand, when the reaction temperature is greater than 250°C, the hematite platelet particles can be obtained, but in order for stable operation of a hydrothermal treatment device and economically preparing hematite platelet, an appropriate upper limit temperature may be 250°C.
- [76] In operation S30, the intermediate product produced in operation S20 acts as a seed

of crystal, thereby producing hematite platelet ( $\alpha$ -Fe<sub>2</sub>O<sub>3</sub>) crystal in which K, Na, Li, or Ca is employed by hydrothermal reaction.

- [77] In addition, after operation S30, the method of preparing a hematite platelet may further include filtering, washing and drying the hematite platelet particles (operation S40) in order to obtain hematite platelet particles having a desired size and thickness.
- [78] The size of hematite platelet particles prepared may be in a range of 1~100  $\mu\text{m}$ , and preferably in a range of 5~80  $\mu\text{m}$ . When the size of the particles is less than 1  $\mu\text{m}$ , reflectance is decreased. On the other hand, when the size of the particles is greater than 100  $\mu\text{m}$ , the hematite platelet particles are not suitable as a metallic pigment.
- [79] In addition, the hematite platelet may have an aspect ratio of 20 or greater, and preferably an aspect ratio of 25 or greater. When the aspect ratio of the hematite platelet is less than 20, it is difficult to produce interference colors as an optical property and adhesiveness of particles is decreased, resulting in poor use thereof. An upper limit of the aspect ratio is not limited, but when the aspect ratio of the hematite platelet is greater than 100, strong particle gloss may be realized.
- [80] A hematite platelet pigment with metallic gloss in which the synthesized hematite platelet is used as a base and metal oxide is coated on the base is manufactured to prepare, for example, a pearl pigment with strong metallic effect.
- [81] The hematite platelet pigment may be prepared, for example, by suspending the hematite platelet obtained using the preparation method as described above in deionized water, adding an acid such as hydrochloric acid or sulfuric acid to the resultant and stirring, simultaneously adding a metal chloride solution and an aqueous alkali solution to the aqueous solution and refluxing the resultant to prepare a hematite platelet particle aqueous solution coated with a metal oxide hydrate layer, and filtering, washing, drying and sintering the hematite platelet particle aqueous solution.
- [82] The metal oxide coated on the surface of the hematite platelet is a metal oxide typically coated on the surface of pigment and used to produce metallic gloss and interference colors in the field of preparing metallic pigments. The metal oxide is not particularly limited, and may be, in particular, SnO<sub>2</sub>, TiO<sub>2</sub>, ZrO<sub>2</sub>, SiO<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub>, MgO, or MnO<sub>2</sub>, or at least two of these materials. More preferably, it is desirable to form a multi-layer coating structure by using a variety of metal oxides. For example, such a multi-layer coating structure may include a coating layer formed of SiO<sub>2</sub>, and a TiO<sub>2</sub> coating layer formed on the surface of the SiO<sub>2</sub> coating layer. The advantages of such a multi-layer coating structure are an increase in covering power and obtainment of high-quality interference colors.

[83] Hereinafter, the present invention will be described in further detail with reference to the following examples. These examples are only for illustrative purposes and are not intended to limit the scope of the present invention.

[84] First, synthesis of hematite platelet will be described.

[85]

[86] Experimental Example 1

[87] Synthesis of hematite platelet according to a change in the amount of potassium chloride (KCl) as an additive salt added to a precursor mixed solution

[88] A mixed solution of 10 g of iron chloride ( $\text{FeCl}_3$ ), 12 g of potassium hydroxide (KOH) and 120 g of water was stirred to prepare a hematite platelet precursor mixed solution. Then, 0.1~0.4 g of potassium chloride and 30 g of potassium hydroxide were further added to the hematite platelet precursor mixed solution as an additive salt to change the composition ratio. In this regard, the mixed solution was prepared by uniformly mixing at 200 rpm or greater by using an agitator. Then the mixture was put in a hydrothermal reactor and heated at 170°C for 20 hours. After the hydrothermal reaction was terminated, the hydrothermal reactor was cooled down at room temperature. Then the reactor was washed using distilled water more than 3 times and dried at 60°C for 1 day to obtain a final product.

[89] The obtained final product was monocrystalline hematite platelet ( $\alpha\text{-Fe}_2\text{O}_3$ ). FIG. 2 is a scanning electron microscopic (SEM) image of a side surface of hematite platelet particle powders prepared in Experimental Example 1-4. Referring to FIG. 2, it can be seen that as a result of measurement of the side surface of the hematite platelet particle, a monocrystalline hematite platelet having an aspect ratio of 27 (ratio of particle size/thickness=  $17\mu\text{m}/0.63\mu\text{m}$ ) was prepared. FIG. 3A is a SEM image of the overall shape of the hematite platelet particles of FIG. 2, and FIG. 3B is an optical microscopic image of a top surface of the hematite platelet particles of Experimental Example 1-4. Referring to FIGS. 3A and 3B, the particles have hexahedron-like crystal shapes in the form of platelet and red purple-colored monocystals. FIG. 4 illustrates particle size distribution of the hematite platelet of FIG. 2 measured using a particle size analyzer (Malvern Co.). The particle sizes  $D_{10}$ ,  $D_{50}$ , and  $D_{90}$  were measured as  $9\mu\text{m}$ ,  $17\mu\text{m}$ , and  $25\mu\text{m}$ , respectively. FIG. 5 illustrates X-ray diffraction analysis results of the hematite platelet powder of FIG. 2. As a result of the estimation of crystallization, it was confirmed that the product was hematite ( $\alpha\text{-Fe}_2\text{O}_3$ , JCPDS No. 86-0550).

[90]

[91] Experimental Example 2

[92] Change in synthesis temperature of hematite platelet

[93] A mixed solution of 10 g of iron chloride ( $\text{FeCl}_3$ ), 12 g of potassium hydroxide (KOH) and 120 g of water was stirred to prepare a hematite platelet precursor mixed solution. Then, 0.3 g of potassium chloride (KCl) and 30 g of potassium hydroxide were further added to the hematite platelet precursor mixed solution as an additive salt to change the composition ratio. In this regard, the mixed solution was prepared by uniformly mixing at 200 rpm or greater by using an agitator. Then the mixture was put in a hydrothermal reactor and heated at 170~220°C for 20 hours. After the hydrothermal reaction was terminated, the hydrothermal reactor was cooled down at room temperature. Then the reactor was washed using distilled water more than 3 times and dried at 60°C for 1 day to obtain a final product.

[94] The obtained final product was monocrystalline hematite platelet ( $\alpha\text{-Fe}_2\text{O}_3$ ). FIG. 6 is a SEM image of a side surface of the hematite platelet power prepared in Experimental Example 2-1. Referring to FIG. 6, as a result of measurement of the side surface, it can be seen that a monocrystalline hematite platelet having an aspect ratio of 25 (ratio of particle size/thickness=  $20\mu\text{m}/0.8\mu\text{m}$ ) was prepared.

[95]

[96] Experimental Example 3

[97] Synthesis of hematite platelet according to a change in the amount of potassium hydroxide (KOH) added to a precursor mixed solution

[98] A mixed solution of 10 g of iron chloride ( $\text{FeCl}_3$ ), 12 g of potassium hydroxide (KOH) and 120 g of water was stirred to prepare a hematite platelet precursor mixed solution. Then, potassium hydroxide was further added to the hematite platelet precursor mixed solution by changing the amount thereof in a range of 6~60 g to change the composition ratio. In this regard, the mixed solution was prepared by uniformly mixing at 200 rpm or greater by using an agitator. Then the mixture was put in a hydrothermal reactor and heated at 170°C for 20 hours. After the hydrothermal reaction was terminated, the hydrothermal reactor was cooled down at room temperature. Then the reactor was washed using distilled water more than 3 times and dried at 60°C for 1 day to obtain a final product.

[99] The obtained final product was monocrystalline hematite platelet ( $\alpha\text{-Fe}_2\text{O}_3$ ). FIG. 7 is a SEM image of a side surface of the hematite platelet particles prepared in Experimental Example 3. Referring to FIG. 7, it can be seen that a monocrystalline hematite platelet having an aspect ratio of 13 (ratio of particle size/thickness=  $23\mu\text{m}/1.8\mu\text{m}$ ) was prepared.



[100] From Experimental Example 3, it can be confirmed that as the amount of KOH increases, the size of hematite platelet particles is proportionally increased, and the aspect ratio is somewhat increased. Moreover, it was confirmed that the aspect ratio of the hematite platelet particles obtained without adding an additive salt was less than 20.

[101]

[102] Experimental Example 4

[103] A method of preparing hematite platelet by adding nanoparticle hematite seed

[104] Hematite platelet was prepared in the same manner as in Experimental Example 2.

[105] FIG. 8 is a transmission electron microscopic (TEM) image of a nanoparticle hematite seed used in Experimental Example 4. FIG. 9 is a SEM image of hematite platelet particle powder prepared in the same manner as in Experimental Example 2, by adding the nanoparticle hematite seed of FIG. 8 in an amount of 0.01 parts by weight with respect to 100 parts by weight of iron(III) salt. Referring to FIG. 9, as a result of measurement of the side surface of the particles, it can be seen that a monocrystalline hematite platelet having an aspect ratio of 17 (a ratio of particle size/thickness =  $10\mu\text{m}/0.6\mu\text{m}$ ) was prepared.

[106]

[107]

[108]

[109]

[110]

[111]

[112]

[113]

[114]

[115]

[116] Table 1

[Table 1]

[Table ]

		Weight of precursor mixed solution (g)				Hydro-thermal synthesis condition	Particle Size/ Thickness( $\mu\text{m}/\mu\text{m}$ )	Aspect ratio	Particle color
		Fe(NO <sub>3</sub> ) <sub>3</sub>	KOH	KCl	H <sub>2</sub> O	Temperature/Ti me (°C/h)			
Ex. 1	1-1	10	42	0.1	120	170/20	19/0.95	20	purple
	1-2	10	42	0.2	120	170/20	19/0.90	21	purple
	1-3	10	42	0.3	120	170/20	19/0.80	24	Red-purple
	1-4	10	42	0.4	120	170/20	17/0.63	27	Red-purple
Ex. 2	2-1	10	42	0.3	120	180/20	20/0.80	25	Red-purple
	2-2	10	42	0.3	120	200/20	21/1.00	21	purple
	2-3	10	42	0.3	120	220/20	22/1.20	18	purple
Ex. 3	3-1	10	24	-	120	180/20	11/1.20	9	Black-purple
	3-2	10	30	-	120	180/20	15/1.40	9	Black
	3-3	10	42	-	120	180/20	19/1.70	11	Black
	3-4	10	48	-	120	180/20	23/1.80	13	Black

[117] \* The size and thickness of hematite platelet particles are calculated as an average value of sizes and thicknesses of 20 particles in an electron microscopic image.

[118]

[119] Then, by using the hematite platelets prepared in Experimental Examples 1-4 as a base, hematite platelet pigments were obtained by the following examples and the effects thereof were evaluated.

[120]

[121] Example 1

[122] 100~150 g of the hematite platelet particles prepared in Experimental Example 1-1

was suspended in 2L of deionized water, and then the temperature of the mixture was raised to 70~80°C. Then, a 5% hydrochloric acid was added to the mixture to adjust the pH of the mixture to 2, and the mixture was stirred for 30 minutes. Then, a 40% solution of  $\text{TiOCl}_2$  and NaOH was added to the mixture to form a  $\text{TiO}(\text{OH})_2$  coating layer on the surface of the hematite platelet particles. As the amount of the solution of  $\text{TiOCl}_2$  and NaOH to be added was increased, interference colors of gold, red, purple, blue, and green were exhibited. When a desired interference color was obtained, the addition of the solution of  $\text{TiOCl}_2$  and NaOH was stopped, and then the resultant was stirred for 10 minutes or greater. Then the resultant was filtered, washed and dried, and then sintered at 800°C to form a titanium dioxide ( $\text{TiO}_2$ ) layer having an anatase structure on the surface of the hematite platelet particles.

[123]

[124] Example 2

[125] 100~150 g of the hematite platelet particles prepared in Experimental Example 1-1 was suspended in 2L of deionized water, and then the temperature of the mixture was raised to 70~80°C. Then, a 5% hydrochloric acid was added to the mixture to adjust the pH of the mixture to 2, and the mixture was stirred for 30 minutes. Then, 60 ml of a 5%  $\text{SnCl}_4 \cdot 5\text{H}_2\text{O}$  solution and a NaOH solution were simultaneously added to the mixture to form a  $\text{TiO}(\text{OH})_2$  coating layer on the surface of the hematite platelet particles. As the amount of the solution of  $\text{TiOCl}_2$  and NaOH to be added was increased, thereby increasing the thickness of the coating layer, interference colors of gold, red, purple, blue, and green were exhibited. When a desired interference color was obtained, the addition of the  $\text{TiOCl}_2$  and NaOH solution was stopped, and then the resultant was stirred for 10 minutes or greater. Then the resultant was filtered, washed and dried, and then sintered at 800°C to form a  $\text{TiO}_2$  layer having a rutile structure on the surface of the hematite platelet particles. FIG. 10 is a SEM image of the hematite platelet particles coated with the  $\text{TiO}_2$  layer.

[126]

[127] Example 3

[128] 100~150 g of the hematite platelet particles prepared in Experimental Example 1-2 was suspended in 2L of deionized water, and then the temperature of the mixture was raised to 70~80°C. Then, a 5% hydrochloric acid was added to the mixture to adjust the pH of the mixture to 2, and the mixture was stirred for 30 minutes. Then, a 40% solution of  $\text{TiOCl}_2$  and NaOH was added to the mixture to form a  $\text{TiO}(\text{OH})_2$  coating layer on the surface of the hematite platelet particles. In this regard, the solution of

TiOCl<sub>2</sub> and NaOH was added until gold color was exhibited. Then the mixture was stirred for 30 minutes or greater, and an aqueous NaOH solution was added to the mixture to adjust the pH of the mixture to 3. When a desired color, pearl gray, was exhibited, an iron(III) chloride solution was added to the mixture. Then the resultant was filtered, washed and dried, and then sintered at 900°C to form a layer formed of TiO<sub>2</sub> and Fe<sub>2</sub>O<sub>3</sub> on the surface of the hematite platelet particles.

[129]

[130]     Example 4

[131]     100~150 g of the hematite platelet particles prepared in Experimental Example 1-2 was suspended in 2L of deionized water, and then the temperature of the mixture was raised to 70~80°C. Then, a 5% hydrochloric acid was added to the mixture to adjust the pH of the mixture to 3, and the mixture was stirred for 30 minutes. An aqueous NaOH solution was added to the mixture to continuously maintain the pH level, and then an iron(III) chloride solution was added to the mixture until a desired color, pearl gray, was exhibited. Then the resultant was filtered, washed and dried, and then sintered at 900°C to form a Fe<sub>2</sub>O<sub>3</sub> layer on the surface of the hematite platelet particles.

[132]     According to the present invention, hematite platelets having a particle size of 1~100 $\mu$ m, a particle thickness of 0.3~4 $\mu$ m and an aspect ratio of 20 or greater can be prepared.

[133]

[134]     While the present invention has been particularly shown and described with reference to exemplary embodiments thereof, it will be understood by those of ordinary skill in the art that various changes in form and details may be made therein without departing from the spirit and scope of the present invention as defined by the following claims.

[135]

### **Industrial Applicability**

[136]     The present invention relates to a hematite platelet, and a hematite platelet pigment including the hematite platelet .,

[137]     Hematite platelet can be used as a rust inhibiting pigment for steel structures, paint for protecting woods, and a print ink for plywood. Also, hematite that has a small particle size and is transparent is used as a coloring pigment for metallic paints, for example, vehicles, by using its metallic gloss properties.

[138]     In addition, hematite has ultraviolet-ray absorbing properties and covering properties with respect to skin (skin covering effect), and thus is used as a material for forming

films for packaging foods, colorants for medicine containers, and an extender for cosmetics.

[139]

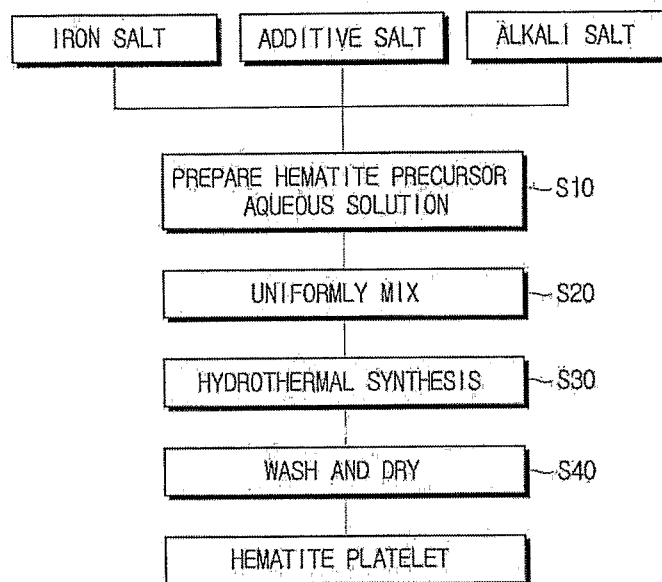
## Claims

- [1] A method of preparing a hematite platelet, comprising preparing a hematite precursor aqueous solution comprising iron(III) salt; an aqueous alkali solution; and an additive comprising at least one selected from the group consisting of potassium-based salts comprising potassium phosphate, potassium sulfate, potassium chloride, potassium nitrate, potassium carbonate, potassium hydrogen carbonate, potassium bromide, and potassium fluoride, sodium-based salts having the same anion as that of the potassium-based salts, lithium-based salts, and calcium-based salts; uniformly mixing the hematite precursor aqueous solution to obtain an intermediate product comprising  $\text{Fe}^{+3}$  and at least one of  $\text{K}^+$ ,  $\text{Na}^+$ ,  $\text{Li}^+$ , and  $\text{Ca}^+$ ; and hydrothermally treating the hematite precursor aqueous solution.
- [2] The method of claim 1, wherein the hydrothermally treating process is performed at a temperature in a range of 150~250°C for 2~240 hours.
- [3] The method of claim 1, wherein the iron(III) salt comprises at least one of  $\text{FeCl}_3$ ,  $\text{Fe}_2(\text{SO}_4)_3$ , and  $\text{Fe}(\text{NO}_3)_3$ .
- [4] The method of claim 1, wherein the aqueous alkali solution comprises at least one selected from the group consisting of potassium hydroxide, sodium hydroxide, calcium hydroxide, and lithium hydroxide.
- [5] The method of claim 1, wherein the hematite precursor aqueous solution has a pH of 11 or greater.
- [6] The method of claim 1, further comprising, after the hydrothermally treating process, filtering, washing and drying the hematite platelet particles.
- [7] The method of claim 1, wherein the hematite precursor aqueous solution further comprises a hematite seed, constituting hematite nanoparticles having a size of 1~50 nm, in an amount of 0.001~1 parts by weight with respect to 100 parts by weight of the iron(III) salt.
- [8] A hematite platelet prepared using the method of claim 1, wherein the hematite platelet comprises hematite ( $\alpha\text{-Fe}_2\text{O}_3$ ) particles in which K, Na, Li, or Ca is employed, and has a particle size of 1~100  $\mu\text{m}$ , a particle thickness of 0.3~4  $\mu\text{m}$ , and an aspect ratio of 20 or greater.
- [9] A hematite platelet prepared using the method of claim 2, wherein the hematite platelet comprises hematite ( $\alpha\text{-Fe}_2\text{O}_3$ ) particles in which K, Na, Li, or Ca is employed, and has a particle size of 1~100  $\mu\text{m}$ , a particle thickness of 0.3~4  $\mu\text{m}$ ,

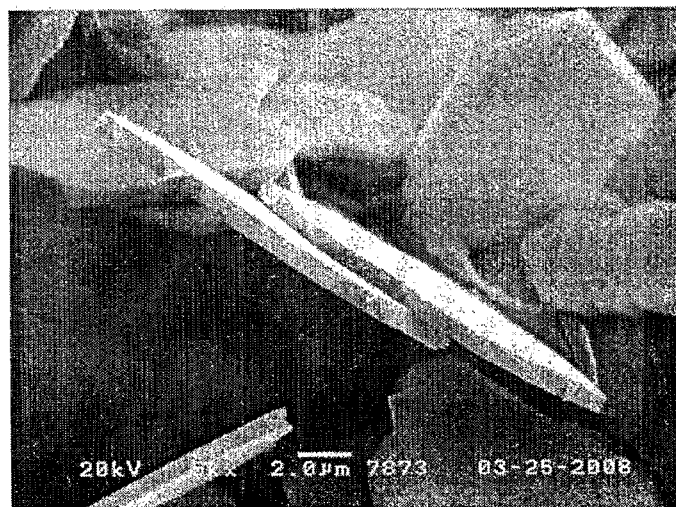
and an aspect ratio of 20 or greater.

- [10] A hematite platelet prepared using the method of claim 3, wherein the hematite platelet comprises hematite ( $\alpha$ -Fe<sub>2</sub>O<sub>3</sub>) particles in which K, Na, Li, or Ca is employed, and has a particle size of 1~100  $\mu$ m, a particle thickness of 0.3~4  $\mu$ m, and an aspect ratio of 20 or greater.
- [11] A hematite platelet prepared using the method of claim 4, wherein the hematite platelet comprises hematite ( $\alpha$ -Fe<sub>2</sub>O<sub>3</sub>) particles in which K, Na, Li, or Ca is employed, and has a particle size of 1~100  $\mu$ m, a particle thickness of 0.3~4  $\mu$ m, and an aspect ratio of 20 or greater.
- [12] A hematite platelet prepared using the method of claim 5, wherein the hematite platelet comprises hematite ( $\alpha$ -Fe<sub>2</sub>O<sub>3</sub>) particles in which K, Na, Li, or Ca is employed, and has a particle size of 1~100  $\mu$ m, a particle thickness of 0.3~4  $\mu$ m, and an aspect ratio of 20 or greater.
- [13] A hematite platelet prepared using the method of claim 6, wherein the hematite platelet comprises hematite ( $\alpha$ -Fe<sub>2</sub>O<sub>3</sub>) particles in which K, Na, Li, or Ca is employed, and has a particle size of 1~100  $\mu$ m, a particle thickness of 0.3~4  $\mu$ m, and an aspect ratio of 20 or greater.
- [14] A hematite platelet prepared using the method of claim 7, wherein the hematite platelet comprises hematite ( $\alpha$ -Fe<sub>2</sub>O<sub>3</sub>) particles in which K, Na, Li, or Ca is employed, and has a particle size of 1~100  $\mu$ m, a particle thickness of 0.3~4  $\mu$ m, and an aspect ratio of 20 or greater.
- [15] A hematite platelet pigment in which the hematite platelet of claim 8 is used as a base, and a metal oxide is coated on the base.
- [16] The hematite platelet pigment of claim 15, wherein the metal oxide comprises at least one selected from the group consisting of SnO<sub>2</sub>, TiO<sub>2</sub>, ZrO<sub>2</sub>, SiO<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub>, MgO, and MnO<sub>2</sub>.
- [17] The hematite platelet pigment of claim 15, wherein the metal oxide is formed as a multi-layer structure comprising at least two layers.
- [18] The hematite platelet pigment of claim 16, wherein the metal oxide is formed as a multi-layer structure comprising at least two layers.

[Fig 1]

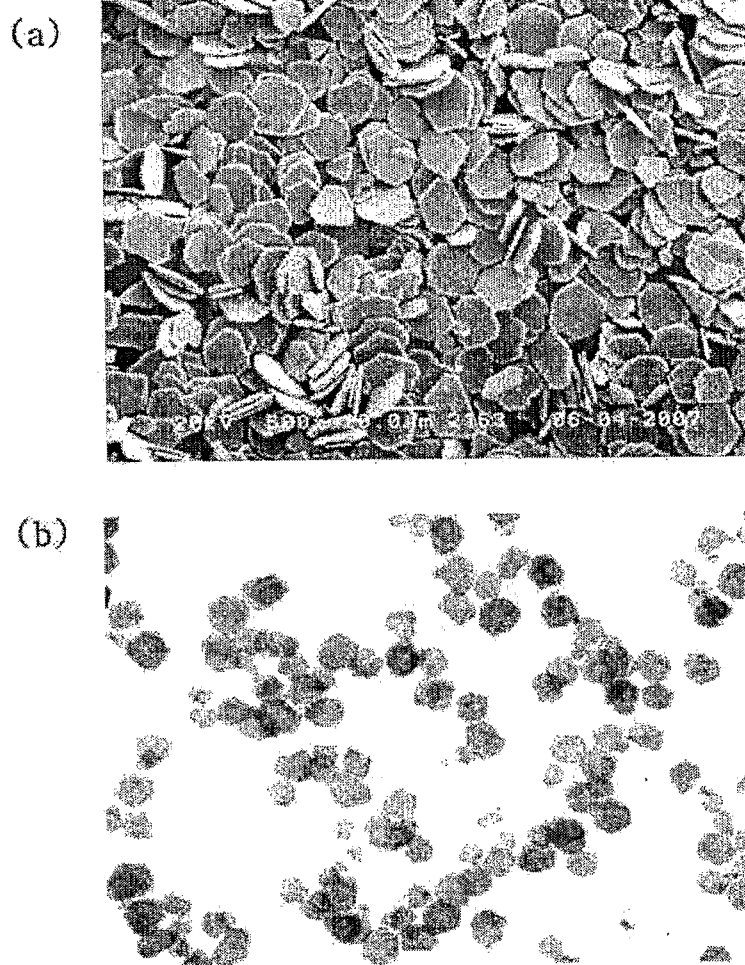


[Fig 2]

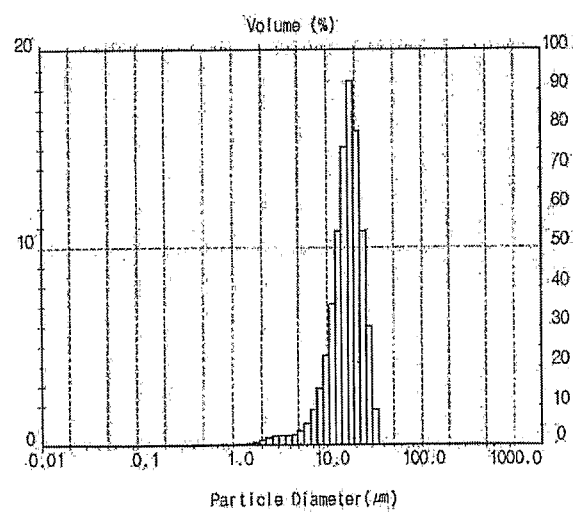




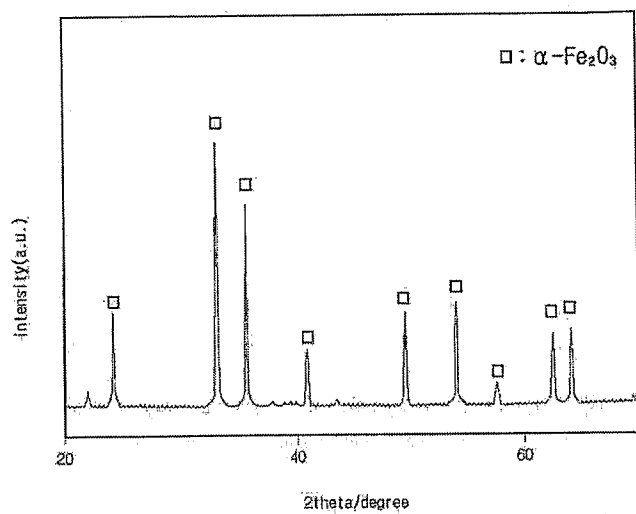
[Fig 3]



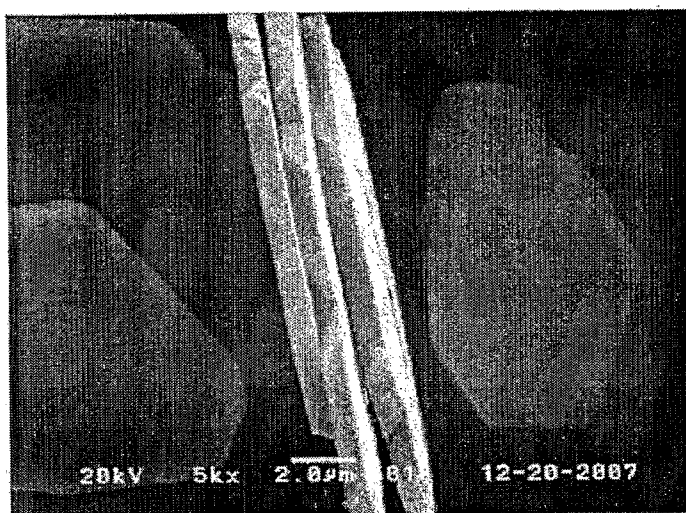
[Fig 4]



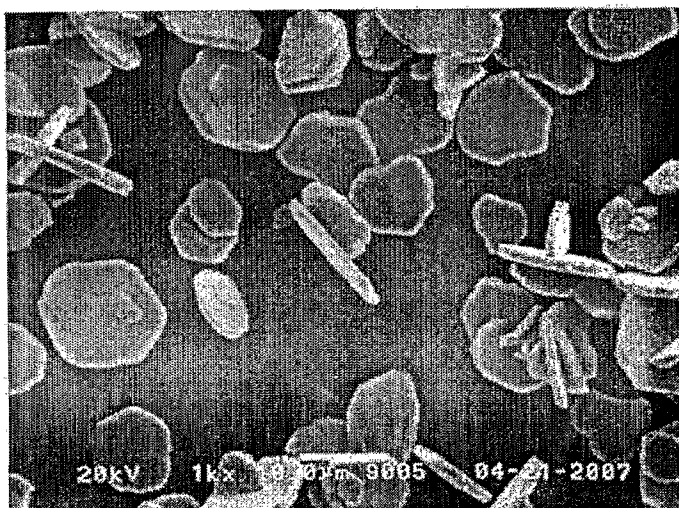
[Fig 5]



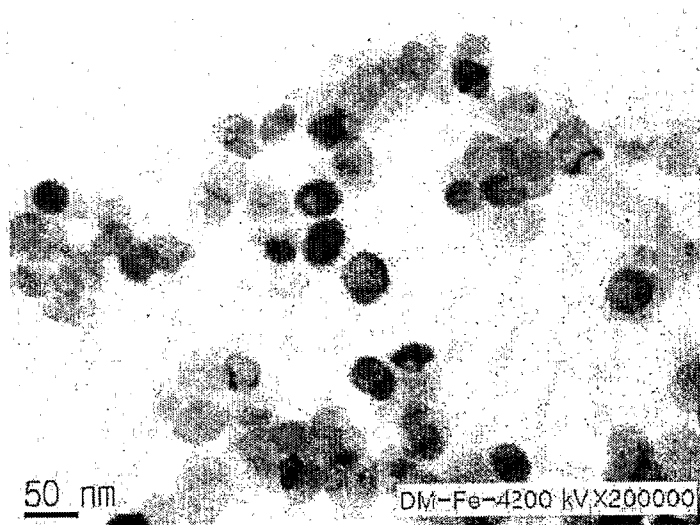
[Fig 6]



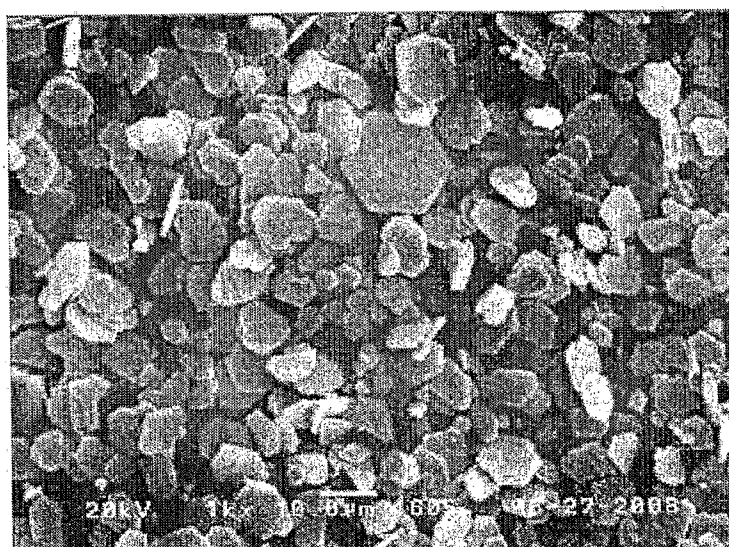
[Fig 7]



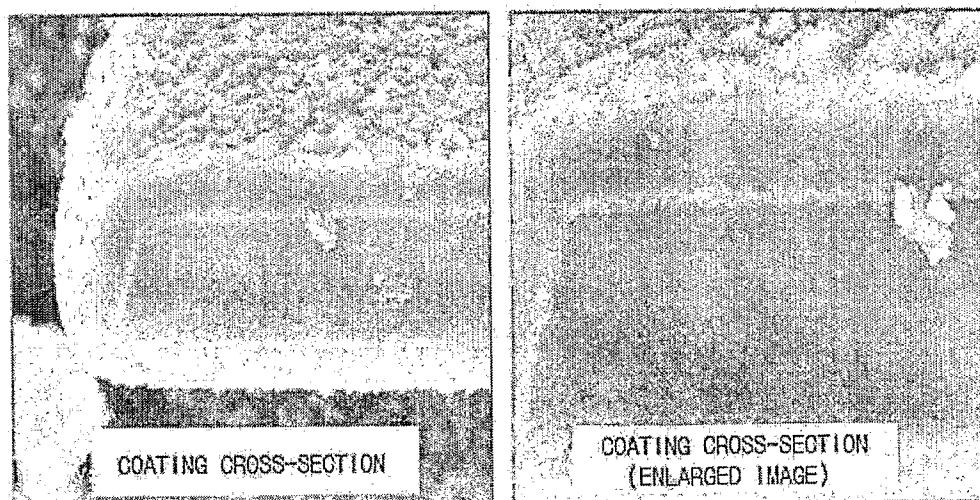
[Fig 8]



[Fig 9]



[Fig 10]



**A. CLASSIFICATION OF SUBJECT MATTER***C09C 1/24(2006.01)i*

According to International Patent Classification (IPC) or to both national classification and IPC

**B. FIELDS SEARCHED**

Minimum documentation searched (classification system followed by classification symbols)

IPC 8 C09C 1/22, C09C 1/24, C09C 3/00, C09C 1/00, C01G 49/00

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Korean Utility models and applications for Utility models since 1975

Japanese Utility models and applications for Utility models since 1975

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

eKIPASS (KIPO Internal)

**C. DOCUMENTS CONSIDERED TO BE RELEVANT**

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	JP01-93427 A (TODA KOGYO CORPORATION ) 12 APRIL 1989	1~6
Y	See claim 1, pages 3,4	7~18
Y	JP06-56429 A (TODA KOGYO CORPORATION) 1 MARCH 1994 See claims 1,2; pages 3~5	7~18
Y	KR 10-2001-0089601A (MERCK PATENT GMBH) 6 OCTOBER 2001 See claims 1~4	15~18
A	JP03-109217 A (BASF AKTIENGESELLSCHAFT) 9 MAY 1991 See claims 1~3; pages 3~5	1



Further documents are listed in the continuation of Box C.



See patent family annex.

\* Special categories of cited documents:

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Date of the actual completion of the international search

26 MARCH 2009 (26.03.2009)

Date of mailing of the international search report

**26 MARCH 2009 (26.03.2009)**

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KIM, Bumsu

Telephone No. 82-42-481-8450



**INTERNATIONAL SEARCH REPORT**

Information on patent family members

International application No.

**PCT/KR2008/004561**

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