BROOKES & MARTIN

CHARTERED PATENT AGENTS
TRADE MARK AGENTS
EUROPEAN PATENT ATTORNEYS

RICHARD C. MARTIN, M.A.CANTAB.
DAVID C. WOODCRAFT, B.Sc.(CHEM.)
ERIC E. BARNARD, C.ENG., M.I.E.E., C.DIP. A.F.
JOHN F. SYMONDS, LL.B. LOND., SOLICITOR
JOHN H. SLAKE, B.Sc.,(CHEM.)
HUGH R. WRIGHT, M.A. OXON.
PETER C. SANDERS, B.Sc.

DONALD J. ATTFIELD, B.Sc. C.CHEM M.R.S.C.(BIRMINGHAM)
ASSISTED BY:
TIMOTHY M. A. RICKARD, B.Sc. PH D.
GRAHAM F. MATTHEWS, B.A., C.ENG. F.I.E.E. (CONSULTANT)
H. NICHOLAS MATTHEWS, M.INST. T.M.A. (CONSULTANT)
GEOFFREY E. BROOME (CONSULTANT)

OFFICE MANAGER - A.J. JELLY
ACCOUNTS - MRS C. OLSINIA

TELEPHONES (071) 242 9631
INTERNATIONAL +44 71 242 9631
FACSIMILE (071) 831 0586
INTERNATIONAL +44 71 831 0586
TELEX 267541 BROOKMAR G
LDX 318

HIGH HOLBORN HOUSE
52/54, HIGH HOLBORN
LONDON, WC1V 6SE

OUR REF JR/CH1370

YOUR REF CH 1370

RECEIVED FEB 19 1991

RECEIVED DJG
FEB 18 1991
PATENT RECORDS CENTER

6 February 1991

E.I. Du Pont de Nemours & Company
Legal Department, Patent Records Center,
1007 Market Street,
Wilmington,
Delaware 19898,
U.S.A.

For the attention of Mr. David J. Gould

Dear Sirs,

European Patent No. 0 245 984

We have pleasure in enclosing the Certificate of Grant of European Patent No. 0245984, granted on Application No. 87303682.6, together with a copy of the published patent specification.

Publication of the grant took place on 16 January 1991 and the patent will be open to opposition for a period of nine months from that date.

The patent will remain in force for a term of 20 years from the application filing date, i.e. from 27 April 1987 subject to the payment of annual renewal fees in each of the designated states in which it has been registered, i.e. Belgium, Germany, Spain, France, Great Britain and the Netherlands.

The patent may be maintained in all or any of these designated states. The Associates in each of these countries have been instructed to attend to the payment of the first renewal fee after grant and also to forward the certificate of renewal and their debit notes direct to Du Pont. We have entered the European Patent (UK) on our maintenance system.

Yours faithfully,

Miss Jill Routh
BROOKES & MARTIN

TUNBRIDGE WELLS: 1 BOYNE PARK, TUNBRIDGE WELLS, KENT, TN4 8EL. TEL.(0892) 510000 TELEX 957078 FACSIMILE (0892) 510666
BIRMINGHAM: PRUDENTIAL BUILDINGS, 5 ST. PHILIP'S PLACE, BIRMINGHAM, B3 2AF. TEL.(021) 236 6262 TELEX 335381 FACSIMILE (021) 2001473
MUNICH: 15 ZWEIBRÜCKENSTRASSE, 8000 MUNICH 22. TEL.(089) 22 20 10

FOLDER NOT UPDATED
CHECKED D.C. 2-IR-91 SR

N 30919

Europäisches Patentamt | European Patent Office | Office européen des brevets

URKUNDE

Es wird hiermit bescheinigt, daß für die in der beigefügten Patentschrift beschriebene Erfindung ein europäisches Patent für die in der Patentschrift bezeichneten Vertragsstaaten erteilt worden ist.

CERTIFICATE

It is hereby certified that a European patent has been granted in respect of the invention described in the annexed patent specification for the Contracting States designated in the specification.

CERTIFICAT

Il est certifié qu'un brevet européen a été délivré pour l'invention décrite dans le fascicule de brevet ci-joint, pour les Etats contractants désignés dans le fascicule de brevet.

Europäisches Patent Nr.: 0245984
European patent No.:
Brevet européen n°:

Patentinhaber: E.I. DU PONT DE NEMOURS AND COMPANY
Proprietor of the patent: 1007 Market Street
Titulaire du brevet: Wilmington Delaware 19898/US

München, den 16.01.91
Munich,
Fait à Munich, le

A. Goeller

Generaldirektion 2 – Formalprüfungsstelle
Directorate-General 2 – Formalities Section
Direction générale 2 – Section des formalités

EPA/EPO/OEB Form 2031 03.83

N 30919.01

(19) Europäisches Patentamt
European Patent Office
Office européen des brevets

(11) Publication number: **0 245 984**
B1

(12) **EUROPEAN PATENT SPECIFICATION**

(45) Date of publication of patent specification: **16.01.91**

(51) Int. Cl.[5]: **C 09 C 1/36**, C 09 C 3/06

(21) Application number: **87303682.6**

(22) Date of filing: **27.04.87**

(54) Titanium dioxide pigment coated with boria-modified silica.

(30) Priority: **30.04.86 US 857324**

(43) Date of publication of application:
19.11.87 Bulletin 87/47

(45) Publication of the grant of the patent:
16.01.91 Bulletin 91/03

(84) Designated Contracting States:
BE DE ES FR GB NL

(56) References cited:
EP-A-0 073 340
DE-A-2 740 561

CHEMICAL ABSTRACTS, vol. 88, no. 2, 9th January 1978, page 85, abstract no. 8602k, Columbus, Ohio, US; & FN-A-53 133 (KEMIRA OY) 31-10-1977

(73) Proprietor: **E.I. DU PONT DE NEMOURS AND COMPANY**
1007 Market Street
Wilmington Delaware 19898 (US)

(72) Inventor: **Jacobson, Howard Wayne**
2009 Longcome Drive
Wilmington Delaware 19810 (US)

(74) Representative: **Woodcraft, David Charles et al**
BROOKES & MARTIN High Holborn House
52/54 High Holborn
London, WC1V 6SE (GB)

EP 0 245 984 B1

Courier Press, Leamington Spa, England.

Description

Background of the Invention

The present invention relates generally to coated TiO_2 particles for use as pigments, and particularly to TiO_2 particles having densified silica coatings.

TiO_2 has a high refractive index for its density, which renders it a superior pigment for use in coatings, e.g., paints. However, TiO_2 is photoactive; exposure to ultraviolet radiation results in generation of free radicals on the surface of TiO_2 particles. Thus, where a TiO_2 pigment is employed in a paint exposed to sunlight, free radicals are generated which can migrate to the film-forming component of the paint, resulting in coating degradation or failure.

Therefore, minimizing free radical migration is desirable in order to provide lightfast and stable pigments for coating use. A widely-used approach to the problem of providing lightfastness involves coating TiO_2 particles with a layer of densified silica. The following patent references disclose conventional silica coating densification techniques.

Werner, U.S. Patent 3,437,502, discloses TiO_2 pigment having high opacity and dispersibility, which is obtained by applying a dense silica coating to TiO_2 followed by topcoating with alumina. The silica coating is applied by increasing the pH of a slurry of TiO_2 particles to above 7. A predetermined quantity of sodium silicate is then added to the slurry, resulting in a rise in pH of the slurry to above 8, generally above 11. The pH is then gradually reduced over a period of several hours by addition of dilute acid, and the resulting silica-coated particles are cured at pH 6.0 to 7.5, at 60° to 100°C, for 30 minutes to several hours. The resulting product is then coated with alumina.

West, U.S. Patent 4,125,412, discloses preparation of durable TiO_2 pigments by slurrying silica-coated TiO_2 particles at pH 9 to 10.5 while maintaining slurry temperature at 80° to 100°C.

European Patent Application 73,340 describes a similar method for coating TiO_2 particles with amorphous dense silica prior to topcoating with alumina. Silica is applied by adding a soluble silicate to a TiO_2 slurry at a temperature of at least 85°C and at a pH of from 9.8 to 10.1. The slurry is then neutralized in at least three neutralization steps by addition of acid.

Common to the teachings of the foregoing references is a requirement for relatively high temperatures and high alkalinity during the silica densification step. An extremely lightfast white pigment for use in paint systems can be made by depositing 4-6 weight percent amorphous dense silica on rutile TiO_2 pigment. To coat the base TiO_2 pigment with this quantity of dense silica requires several hours at 85°-90°C, during which mineral acids are used to precipitate silica from Na_2SiO_3. This high temperature coating operation requires use of steel tanks, since less costly fiberglass tanks are not rated for use in the 85°-90°C temperature range. Methods for providing dense coatings of more than 4 weight percent silica at lower temperatures, e.g., 65°-80°C, to allow use of fiberglass tanks, would be of significant interest to the TiO_2 pigment industry.

It has now been found that codeposition of B_2O_3 with SiO_2 provides dense silica coatings at process temperatures which permit use of fiberglass slurry tanks. The boria-containing pigments resulting from this process are highly lightfast, and exhibit excellent gloss and dispersibility.

Summary of the Invention

The present invention provides a pigment consisting essentially of TiO_2 particles bearing coatings comprising SiO_2 and B_2O_3. In a process aspect, the present invention provides a process for preparing TiO_2 particles bearing coatings of boria-modified silica, comprising:

(a) forming an aqueous slurry of rutile TiO_2 at a temperature from about 65°C to about 90°C;

(b) adjusting pH of the slurry to the range 7-10.5;

(c) adding a predetermined quantity of a solution comprising Na_2SiO_3 and B_2O_3, under conditions which maintain silicate and boria ions in solution;

(d) gradually lowering pH of the slurry to about 7.5-8.5 by addition of acid, thereby depositing a coating of silica and boria; and

(e) curing the resulting coated pigment at a temperature from about 65°C to about 90°C for a period of at least 15 minutes.

Detailed Description of the Invention

In accordance with the present invention, B_2O_3 is codeposited with SiO_2 on TiO_2 particles, providing lightfast, durable pigments. The TiO_2 particles making up the pigments bear dense silica coatings, yet are produced at temperatures significantly lower than those required by other silica densification processes capable of providing dense silica coatings at greater than 4 weight percent.

In practice, silica/boria layers are codeposited from a master solution of Na_2SiO_3 and $Na_2B_2O_4$. A standard acid solubility test can be to measure effectiveness of coating densification. In addition to enhanced densification of SiO_2 in the presence of B_2O_3, an improvement in pigment brightness is provided.

The TiO_2 used to prepare the pigment of the invention can be of the conventional rutile variety, prepared by either the chloride or sulfate process.

The amount of silica or silica topcoated with alumina which the particles bear as coatings can be varied depending primarily upon the pigment's intended use. Generally, the silica coatings will constitute from 2-

10% of the total pigment weight, and an alumina topcoat, if present, will constitute 0.5-5% of the total weight of the particles. Preferably, the silica coating constitutes 4-8% of the total weight of the pigment particles. Preferably, the alumina topcoat, if present, constitutes 1-4% of the total weight.

The amount of alumina the particles bear as coatings, expressed as percent by weight, is calculated by first determining the alumina content of the coated pigment by ion plasma spectroscopy. The alumina content of the uncoated rutile TiO_2 is similarly determined, and the alumina content attributable to the coating is determined by computing the difference between coated and uncoated alumina contents.

The pigment of the present invention can be prepared from an aqueous slurry containing 200-450 grams per liter TiO_2. This slurry is brought to 65°-90°C and is held at that temperature throughout the preparation procedure. The slurry is adjusted to pH 7-10.5, and a sufficient quantity of solution containing Na_2SiO_3 and $Na_2B_2O_4$, or separate solutions of Na_2SiO_3 and $Na_2B_2O_4$, is added to provide a coating of the desired composition and weight. Upon addition of the Na_2SiO_3 and $Na_2B_2O_4$ solution(s), pH of the slurry increases to about 11. Generally, slurry pH must be maintained above about 10 to maintain silicate and boria ions in solution.

Generally, the weight and composition of the coatings to be deposited will be determined by the composition of the master $Na_2SiO_3/Na_2B_2O_4$ solution, the quantity added to the slurry containing TiO_2, and quantity of TiO_2 in the slurry. For TiO_2 slurries containing 400 g/L TiO_2, about 40-50 mL of a master aqueous solution, containing 400 g SiO_2 equivalent and 40 g B_2O_3 equivalent per liter, should be added per liter TiO_2 slurry to provide coatings approximating 5% of pigment weight. The compositions of the coating solutions can be varied as required to provide variations in coating weight and composition.

The coatings applied to TiO_2 particles in accordance with the present invention can contain from 60-95 percent by weight SiO_2 and 0.5-30% by weight B_2O_3, based upon coating weight. Preferably, coatings will contain 80-90 percent by weight SiO_2 and 2-20 percent by weight B_2O_3.

Upon addition of the coating solutions, the pH of the TiO_2 slurry will rise to the range 10.5-11.5. Over a period of 2-4 hours, 15% aqueous HCl or other mineral acid is added to gradually decrease slurry pH to about 8.0. As acid is added, SiO_2/B_2O_3 will be deposited and densified on the surfaces of the TiO_2 particles. After all acid is added, the pigment should be cured at 65°-90°C for 30 minutes.

If desired, alumina can be precipitated on silica/boria coated TiO_2 particles by adding enough sodium aluminate to the slurry, at a temperature from about 50°-90°C, to provide a concentration of about 2-8% by weight of the TiO_2. Acid, normally sulfuric or hydrochloric, is added at the same time to maintain the pH of the slurry within the range 6-9 as the alumina is precipitated. After addition of aluminate is complete, the slurry is allowed to cure, with continuous stirring, for 15-30 minutes.

The resulting pigment is then separated from the liquid by filtration or centrifugation, washed with water, and dried.

Coating compositions can be prepared with the pigments of the invention in conventional ways, e.g., by blending the pigment with a film-forming component and a liquid carrier.

The following examples illustrate particular aspects of the present invention. In the examples, all parts and percentages are by weight and all degrees are Celsius unless otherwise indicated.

Example 1

In a large plastic vessel equipped with stirring apparatus and a pH probe, 9000 parts water are mixed with 3000 parts rutile TiO_2.

The resulting slurry is heated to 75°, and the pH is adjusted to about 7.5 by addition of NaOH. 400 parts of an aqueous solution containing 400 parts SiO_2 equivalent (as Na_2SiO_3) and 40 parts B_2O_3 equivalent (as $Na_2B_2O_4$) per 1000 parts water are added. The pH of the resulting mixture is then determined, and sufficient 15% aqueous HCl is added, over a period of about 3 hours, to reduce the slurry pH to about 8.0. After all HCl has been added, the slurry is cured for 30 minutes at pH 8.0 and 75°.

The resulting coating, which constitutes about 5.5% by weight of the total pigment, contains SiO_2 and B_2O_3 in a ratio by weight of about 90:10.

A coating of 2.5% hydrous alumina is then deposited on the pigment by simultaneous gradual addition of 200 mL $NaAlO_2$ solution (containing $NaAlO_2$ equivalent to 400 g Al_2O_3 per liter solution) and HCl, such that the pH of the slurry is continuously maintained from about 7.0 to about 7.5.

The resulting coated pigment is cured by holding for about 30 minutes at ambient temperature and pH 7.5. The pigment is then filtered from the slurry, washed free of salts with water, and air-dried. The pigment is then micronized, using 3 lbs. superheated steam per lb. pigment. The resulting pigment is suitable for use in paint systems where lightfastness is required.

Example 2

In this series of experiments, several batches of pigment were prepared to evaluate the effect of B_2O_3 codeposition and various processing temperatures upon acid solubility. Generally, acid solubility is inversely related to chalk-fade resistance for TiO_2 pigments.

The pigments described below were prepared by procedures substantially similar to that described in Example 1, above, except that processing temperatures were varied as indicated in Table 1, below. Acid solubilities were determined as follows.

10 mL 66% sulfuric acid is added to a test tube containing a magnetic stirring bar, and the tube is

placed in an aluminum heating block and heated to 175°. A 0.2000 g pigment sample is added to the tube, and digested for 1 hour with stirring. At the conclusion of the digestion period, the tube containing the pigment sample is cooled by pouring the acid mixture into a beaker of ice, and the residue in the tube and beaker is washed with distilled water. The pigment residue in the tube and beaker is collected in a 100 mL volumetric flask, the volume made up to 100 mL by addition of distilled water, and the contents mixed thoroughly. The contents of the volumetric flask are then filtered, and 10 mL of the resulting filtrate are added to a 25 mL volumetric flask, to which 2 mL 20% hydrogen peroxide, and sufficient 10% sulfuric acid to make 25 mL, are added. The resulting solution is allowed to stand one hour. Absorbance of the solution is then read at 400 nm using a 10 mm cell path. Soluble TiO_2 is determined by reference to a previously prepared spectrophotometric curve obtained by measurement of samples containing known quantities of dissolved TiO_2.

TABLE 1
Acid Solubility: (% dissolved TiO_2)

Processing Temp. (°C)	Coating Composition: 100% SiO_2	90% SiO_2/10% B_2O_3
90	0.11	0.10
80	0.27	0.12
70	3.34	0.17
65	6.0	0.22

Example 3

A mill base is made by mixing

Pigment of Example 1	387 parts
alkyd resin (Syntex ® 3145; Celanese Coatings Co.)	149.6 parts
n-butanol	9.4 parts
xylol	79 parts

This mixture is sand ground, and then the sand is filtered from the mill base. A paint is made by mixing 100 parts of the mill base with the following:

xylol	1.4 parts
alkyd resin (Syntex ® 3145; Celanese Coatings Co.)	71.8 parts
melamine resin solution (Cymel ® 248-8, 55% solids, American Cyanamid Co.)	38.5 parts

Claims

1. A pigment comprising rutile TiO_2 particles bearing coatings comprising SiO_2 and B_2O_3.
2. A pigment according to claim 1, wherein the TiO_2 particles have coatings comprising from 60-98 percent by weight SiO_2 and 0.5-30 percent by weight B_2O_3 based on coating weight.
3. A pigment according to claim 2, wherein the TiO_2 particles have coatings comprising from 80-90 percent by weight SiO_2 and 2-20 percent by weight B_2O_3, based on coating weight.
4. A pigment according to claim 3, wherein the TiO_2 particles have SiO_2/B_2O_3 coatings constituting 2-10% of the total pigment weight.
5. A pigment according to claim 4, wherein the TiO_2 particles have SiO_2/B_2O_3 coatings constituting 4-8% of the total pigment weight.
6. A pigment according to any one of the preceding claims, wherein the TiO_2 particles have outer coatings of alumina constituting 0.5-5% of the total weight of the particles.
7. A pigment according to claim 6, wherein the TiO_2 particles have outer coatings of alumina constituting 1-4% of the total weight of the particles.

8. A process for preparing TiO_2 particles bearing coatings of boria-modified silica, comprising:
(a) forming an aqueous slurry of rutile TiO_2 at a temperature from about 65° to about 90°C;
(b) adjusting pH of the slurry to the range 7-10.5;
(c) adding a predetermined quantity of a solution comprising Na_2SiO_3 and B_2O_3, under conditions which maintain silicate and boria ions in solution;
(d) gradually lowering pH of the slurry to abut 7.5-8.5 by addition of acid, thereby depositing a coating of silica and boria; and
(e) curing the resulting coated pigment at a temperature of from about 65°C to about 90°C for a period of at least 15 minutes.

9. A process according to claim 8, wherein sufficient Na_2SiO_3 and B_2O_3 are added in step (c) to provide a coating comprising from 60-98 percent by weight SiO_2 and 0.5-30 percent by weight B_2O_3 based on coating weight.

10. A process according to claim 9, wherein sufficient Na_2SiO_3 and B_2O_3 are added in step (c) to provide a coating comprising from 80-90 percent by weight SiO_2 and 2-20 percent by weight B_2O_3, based on coating weight.

11. A process according to claim 10, wherein sufficient Na_2SiO_3 and B_2O_3 are added in step (c) to provide a SiO_2/B_2O_3 coating constituting 0.5-10% of the total pigment weight.

12. A process according to claim 11, wherein sufficient Na_2SiO_3 and B_2O_3 are added in step (c) to provide a SiO_2/B_2O_3 coating constituting 4-8% of the total pigment weight.

13. A process according to any one of claims 8 to 12 comprising the following additional step:-
(f) adding sufficient $NaAlO_2$ to the slurry, at a temperature from about 50°-90°C, to provide a concentration of about 2-8% by weight of the TiO_2, while adding mineral acid to maintain the pH of the slurry from 6-9.

14. A coating composition comprising a pigment having a densified SiO_2/B_2O_3 coating according to any one of claims 1 to 7, a film-forming material, and a liquid carrier.

Patentansprüche

1. Pigment, welches Rutil-TiO_2-Teilchen enthält, die SiO_2 und B_2O_3 enthaltende Beschichtungen aufweisen.

2. Pigment nach Anspruch 1, worin die TiO_2-Teilchen Beschichtungen aufweisen, die 60-98 Gew.-% SiO_2 und 0,5 bis 30 Gew.-% B_2O_3, bezogen auf das Beschichtungsgewicht, enthalten.

3. Pigment nach Anspruch 2, worin die TiO_2-Teilchen Beschichtungen aufweisen, die 80-90 Gew.-% SiO_2 und 2-20 Gew.-% B_2O_3, bezogen auf das Beschichtungsgewicht, enthalten.

4. Pigment nach Anspruch 3, worin die TiO_2-Teilchen SiO_2/B_2O_3-Beschichtungen aufweisen, die 2-10% vom gesamten Pigmentgewicht ausmachen.

5. Pigment nach Anspruch 4, worin die TiO_2-Teilchen SiO_2/B_2O_3-Beschichtungen aufweisen, die 4-8% vom gesamten Pigmentgewicht ausmachen.

6. Pigment nach einem der vorstehenden Ansprüche, worin die TiO_2-Teilchen äußere Beschichtungen aus Aluminiumoxid aufweisen, die 0,5-5% vom Gesamtgewicht der Teilchen ausmachen.

7. Pigment nach Anspruch 6, worin die TiO_2-Teilchen äußere Beschichtungen aus Aluminiumoxid aufweisen, die 1-4% vom Gesamtgewicht der Teilchen ausmachen.

8. Verfahren zur Herstellung von TiO_2-Teilchen mit Beschichtungen aus Boroxid-modifiziertem Siliziumdioxid durch
(a) Bilden einer wässrigen Aufschlämmung von Rutil-TiO_2 bei einer Temperatur von etwa 65° bis etwa 90°C;
(b) Einstellen des pH-Werts der Aufschlämmung im Bereich von 7-10,5;
(c) Zugeben einer vorbestimmten Menge einer Na_2SiO_3 und B_2O_3 enthaltenden Lösung unter Bedingungen, die Silikat- und Borationen in der Lösung halten;
(d) allmähliches Senken des pH-Werts der Aufschlämmung auf etwa 7,5-8,5 durch Zugabe von Säure, wodurch eine Beschichtung aus Siliziumdioxid und Boroxid niedergeschlagen wird; und
(e) Härten des resultierenden beschichteten pigments bei einer Temperatur von etwa 65°C bis etwa 90°C über einen Zeitraum von wenigstens 15 Minuten.

9. Verfahren nach Anspruch 8, worin in Schritt (c) ausreichend Na_2SiO_3 und B_2O_3 zugesetzt werden, um eine Beschichtung mit 60-98 Gew.-% SiO_2 und 0,5-30 Gew.-% B_2O_3, bezogen auf das Beschichtungsgewicht, zu ergeben.

10. Verfahren nach Anspruch 9, worin in Schritt (c) ausreichend Na_2SiO_3 und B_2O_3 zugesetzt werden, um eine Beschichtung mit 80-90 Gew.-% SiO_2 und 2-20 Gew.-% B_2O_3, bezogen auf das Beschichtungsgewicht, zu ergeben.

11. Verfahren nach Anspruch 10, worin in Schritt (c) ausreichend Na_2SiO_3 und B_2O_3 zugesetzt werden, um eine SiO_2/B_2O_3-Beschichtung zu ergeben, die 0,5-10% vom gesamten pigmentgewicht ausmacht.

12. Verfahren nach Anspruch 11, worin in Schritt (c) ausreichend Na_2SiO_3 und B_2O_3 zugesetzt werden, um eine SiO_2/B_2O_3-Beschichtung zu ergeben, die 4-8% vom gesamten Pigmentgewicht ausmacht.

13. Verfahren nach einem der Ansprüche 8 bis 12, welches den folgenden zusätzlichen Schritt umfaßt
(f) Zugabe von ausreichend $NaAlO_2$ zur Aufschlämmung bei einer Temperatur von etwa 50° bis 90°C,

um eine Konzentration von etwa 2-8% vom TiO_2-Gewicht bereitzustellen, wobei Mineralsäure hinzugefügt wird, um den pH-Wert der Aufschlämmung bei 6-9 zu halten.

14. Überzugszusammensetzung, welche ein Pigment mit einer verdichteten SiO_2/B_2O_3-Beschichtung gemäß einem der Ansprüche 1 bis 7, ein filmbildendes Material und einen flüssigen Träger umfaßt.

Revendications

1. Un pigment comprenant des particules de TiO_2 du type rutile, portant des revêtements comprenant SiO_2 et B_2O_3.

2. Un pigment selon la revendication 1, dans lequel les particules de TiO_2 portent des revêtements comprenant 60 à 98 pour cent en poids de SiO_2 et 0,5 à 30 pour cent en poids de B_2O_3, par rapport au poids de revêtement.

3. Un pigment selon la revendication 2, dans lequel les particules de TiO_2 portent des revêtements comprenant 80 à 90 pour cent en poids de SiO_2 et 2 à 20 pour cent en poids de B_2O_3, par rapport au poids de revêtement.

4. Un pigment selon la revendication 3, dans lequel les particules de TiO_2 portent des revêtements de SiO_2/B_2O_3 constituant 2 à 10% du poids total du pigment.

5. Un pigment selon la revendication 4, dans lequel les particules de TiO_2 portent des revêtements de SiO_2/B_2O_3 constituant 4 à 8% du poids total du pigment.

6. Un pigment selon l'une quelconque des revendications précédentes, dans lequel les particules de TiO_2 portent des revêtements extérieurs d'alumine constituant 0,5 à 5% du poids total des particules.

7. Un pigment selon la revendication 6, dans lequel les particules de TiO_2 portent des revêtements extérieurs d'alumine constituant 1 à 4% du poids total des particules.

8. Un procédé pour préparer des particules de TiO_2 portant des revêtements de silice modifiée par de l'anhydride borique, consistant à:

(a) former une suspension aqueuse de TiO_2 du type rutile à une température d'environ 65° à environ 90°C.

(b) ajuster le pH de la suspension dans l'intervalle de 7 à 10,5;

(c) ajouter une quantité prédéterminée d'une solution comprenant Na_2SiO_3 et B_2O_3, dans des conditions qui maintiennent en solution les ions silicate et d'anhydride borique;

(d) abaisser progressivement le pH de la suspension par addition d'acide jusqu'à atteindre environ 7,5 à 8,5, en déposant ainsi un revêtement de silice et d'anhydride borique; et

(e) faire mûrir le pigment revêtu résultant à une température d'environ 65°C à environ 90°C pendant une période d'au moins 15 minutes.

9. Un procédé selon la revendication 8, dans lequel on ajoute suffisamment de Na_2SiO_3 et de B_2O_3 dans l'étape (c) pour former un revêtement comprenant 60 à 98 pour cent en poids de SiO_2 et 0,5 à 30 pour cent en poids de B_2O_3, par rapport au poids de revêtement.

10. Un procédé selon la revendication 9, dans lequel on ajoute suffisamment de Na_2SiO_3 et de B_2O_3 dans l'étape (c) pour former un revêtement comprenant 80 à 90 pour cent en poids de SiO_2 et 2 à 20 pour cent en poids de B_2O_3, par rapport au poids de revêtement.

11. Un procédé selon la revendication 10, dans lequel on ajoute suffisamment de Na_2SiO_3 et de B_2O_3 dans l'étape (c) pour former un revêtement de SiO_2/B_2O_3 constituant 0,5 à 10% du poids total du pigment.

12. Un procédé selon la revendication 11, dans lequel on ajoute suffisamment de Na_2SiO_3 et de B_2O_3 dans l'étape (c) pour former un revêtement de SiO_2/B_2O_3 constituant 4 à 8% du poids total du pigment.

13. Un procédé selon l'une quelconque des revendications 8 à 12, comprenant l'étape supplémentaire suivante:

(f) ajouter suffisamment de $NaAlO_2$ à la suspension, à une température d'environ 50° à 90°C, pour obtenir une concentration d'environ 2 à 8% par rapport au poids du TiO_2, tout en ajoutant un acide minéral de façon à maintenir le pH de la suspension entre 6 et 9.

14. Une composition de revêtement comprenant un pigment portant un revêtement densifié de SiO_2/B_2O_3 selon l'une quelconque des revendications 1 à 7, une matière filmogène et un véhicule liquide.