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(54) RESINOUS PARTICLES FOR COATING COMPOSITION

(71) We, NIPPON PAINT CO LTD., of No. 2—1—2, Oyodo, Kita, Oyodo-ku, Osaka-shi, Osaka-fu, Japan, a joint stock company organized under the laws of Japan, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:

The present invention relates to a process for preparing resinous particles for a coating composition. More particularly, it relates to an improved process for preparing a coating composition in a slurry or powdery form.

From the viewpoint of avoiding the use of organic solvents which may cause environmental pollution, there have been proposed various coating compositions such as water-soluble coating compositions, emulsion coating compositions, slurry coating compositions and powder coating compositions. Among them, the last two coating compositions are particularly advantageous in scarce inclusion of harmful or polluting materials, simplicity of after treatment, easy recovery for reuse, etc.

For the preparation of slurry coating compositions and powder coating compositions, there is known a method which comprises admixing a liquid composition comprising a resin dissolved in an organic solvent with a coagulating liquid which is miscible with the organic solvent and does not dissolve the resin therein, whereby the resin is deposited; collecting the deposited resin, and drying the recovered resin or dispersing it into an aqueous medium. Alternatively, the liquid composition may be subjected to spray drying, optionally followed by dispersion of the resulting dried resin into an aqueous medium. However, these methods essentially require the use of organic solvents in large amounts, and are therefore disadvantageous from the viewpoints of environmental pollution, firing, economy, etc.

The present invention is directed to the improvement of the above method for the production of a coating composition in a slurry or powdery form not materially using any organic solvent. Thus, a main feature of the present invention is to provide an improved process for preparing resinous particles suitable for a coating composition not materially using any organic solvent. Another feature of this invention is to provide an improved process for preparing a slurry or powder coating composition not materially using any organic solvent. These and other features of this invention will be apparent to those skilled in the art from the foregoing and subsequent descriptions.

The process for preparing resinous particles for a coating composition according to this invention is one in which (1) an aqueous resin composition in which a resin having at least one ionic group but substantially without any material solubility or dispersibility relative to water is dissolved or dispersed in an aqueous medium by at least partial neutralization of the ionic group or groups in the resin is subjected to treatment with a powdering agent and to simultaneous pulverization treatment whereby the resin is deposited in particulate form; and in which (2) the deposited resinous particles are recovered from the aqueous medium.

In the process of the invention, a resin is dissolved or dispersed in an aqueous medium by utilization of the neutralization reaction. Thus, the resin may be any resin conventionally employed for the formation of a coating film but which is required to have at least one ionic group in the molecule so as to be soluble or dispersible in an aqueous medium by neutralization of a part or all of the ionic groups. Examples of the ionic group include an acidic group such as carboxyl and a basic group such as primary, secondary or tertiary amino. In addition to the ionic group or groups, the resin may include any other functional group such as an epoxy group or a hydroxyl group. Specific examples of resins in which the ionic group is acidic are acrylic resins having carboxyl groups, alkyd resins having carboxyl groups, acrylic resins having carboxyl groups and crosslinkable functional groups (e.g. epoxy or hydroxyl), alkyd resins having carboxyl groups and crosslinkable functional groups (e.g. epoxy or hydroxyl), acryl-modified alkyd resins obtained by copolymerization of the alkyd resins with polymerizable acrylic monomers, etc. Specific examples of resins which the ionic group is basic are epoxy resins having primary, secondary or tertiary amino groups (prepared, for instance, by adding organic amino compounds to the epoxy groups in epoxy resins or epoxy-modified resins such as copolymers of phenol glycidyl ether, glycidyl ethers of phenol-aldehyde condensates, glycidyl acrylate or glycidyl methacrylate with other polymerizable monomers), acrylic or vinylic resins having primary, secondary or tertiary amino groups (prepared, for instance, by copolymerization or nitrogen-containing vinyl compounds such as amino group-containing acrylates, amino group-containing methacrylates, vinylpyridines or vinyl-imidazoles with vinyl compounds having no free carboxyl group), polyamide resins having primary, secondary or tertiary amino groups (prepared, for instance, by polycondensing dibasic acids with polyvalent amines), etc.

For neutralization of the ionic group or groups in the resin so as to dissolve or disperse such resin into an aqueous medium, there may be used an inorganic basic substance (e.g. potassium hydroxide, sodium hydroxide, potassium carbonate or ammonia) or an organic basic substance (e.g. methylamine, triethylamine, monoethanolamine, dimethylethanolamine, triethanolamine, ethylenediamine or diethylenetriamine) when the ionic group is acidic, or an inorganic acidic substance (e.g. hydrochloric acid, sulphuric acid, phosphoric acid or carbonic acid) or an organic acidic substance (e.g. formic acid, acetic acid, monochloroacetic acid, dichloroacetic acid, trichloroacetic acid, lactic acid, butyric acid or glycollic acid) when the ionic group is basic. The extent of the neutralization of the ionic group so as to make the resin dissolved or dispersed into an aqueous medium is considerably varied with the kind of the resin and may be usually not less than about 10 percent, preferably not less than about 30 percent.

In addition to the resin dissolved or dispersed in an aqueous medium, the aqueous resin composition may comprise any conventional additive(s) such as a colouring agent, a crosslinking agent or a modifier.

The colouring agent may be pigments, dyes or the like.

As the crosslinking agent, there may be used any conventional crosslinkable resin or compound which can react with the functional group (e.g. carboxyl, primary, secondary or tertiary amino, hydroxy or epoxy) present in the resin. A particularly preferred agent is one which is itself either miscible with the resin or miscible with such resin at least on baking to afford a uniform coating film. Examples of crosslinking agent capable of reacting with a carboxyl group or a primary, secondary or tertiary amino group are two or more epoxy group-containing compounds such as epoxy resins or triglycidyl isocyanurate. Examples of the crosslinking agent capable of reacting with a hydroxyl group are aminoplast resins such as urea resins, melamine resins (e.g. hexamethoxymethylmelamine) and benzoguanamine, and polyisocyanate compounds containing a blocked isocyanate group.

As the modifier, there may be used a substance which is miscible with the resin and shows an effect of improving the quality of a coating film. Specific examples are natural resins, cellulose derivatives such as cellulose acetate butyrate, alkyd resins, acrylic resins, vinyl resins, etc.

On practical use, the crosslinking agent and the modifier may be as such incorporated into the aqueous resin composition when they are in a liquid form. In the case of these agents being solid and hardly miscible as such, they may be melted or dissolved in a suitable solvent on admixture with the aqueous resin composition.

The preparation of the aqueous resin composition may be effected in a conventional manner. For example, an aqueous solution or dispersion of the resin may be admixed with a colouring agent and subjected to a conventional dissolving or dispersing treatment, if necessary, followed by the addition of a crosslinking agent, a modifier and the like to obtain a desired aqueous resin composition. The content of non-volatile components in such aqueous resin composition is usually designed to be from substantially 5 to 80% by weight, preferably from substantially 10 to 60% by weight.

The thus prepared aqueous resin composition is then subjected to treatment with an agent capable of acting on the neutralized ionic group (hereinafter referred to as "powdering agent") to make it free (hereinafter referred to "separation treatment"), if necessary, followed by particle formation treatment. As used herein, the term "pulverization treatment" means any operation which makes the resin deposit in a particulate or powdery state. Stirring is a typical example of such an operation. Other typical examples are described under a) to e) hereinafter, and thus spraying and contacting in a mist form may also be adopted for the same purpose.

When the ionic group in the resin is acidic, the separation treatment is effected with an acidic substance. As the acidic substance, there may be used any such substance which can be dissolved in water to afford hydrogen ions. Specific examples are inorganic acids such as sulfuric acid, hydrochloric acid, phosphoric acid and carbonic acid, organic acids such as formic acid, acetic acid, monochloroacetic acid, dichloroacetic acid, trichloroacetic acid, lactic acid, butyric acid and glycolacetic acid, acidic gases such as carbon dioxide and sulfur dioxide, etc. The inorganic or organic acid may be used as an aqueous solution, usually in a concentration of 1 N or less. The acidic gas may be employed alone or in combination with air or an inert gas such as nitrogen in an appropriate proportion. The amount of the acidic substance to be used may be approximately equivalent to the neutralized ionic groups in the resin.

When the ionic group in the resin is basic, the separation treatment is effected with a basic substance. As the basic substance, there may be employed any such substance which can be dissolved in water to accept hydrogen ions. Specific examples are inorganic bases such as sodium hydroxide, potassium hydroxide, potassium carbonate and ammonia, organic bases such as triethylamine and dimethylaminoethanol, basic gases such as gaseous ammonia, etc. The inorganic or organic base may be used as an aqueous solution, usually in a concentration of 1N or less. The basic gas may be employed alone or in combination with air or an inert gas such as nitrogen in an appropriate proportion. The amount of the basic substance to be used may be approximately equivalent to the neutralized ionic groups in the resin.

The separation treatment may be effected by an appropriate procedure depending on the form and the size of the resinous particles to be formed. For example, the following procedures may be adopted:

(a) a procedure wich comprises gradually introducing the acidic or basic gas into the aqueous resin composition with vigorous stirring;

(b) a procedure which comprises gradually adding the aqueous resin composition to an aqueous solution of the acidic or basic substance with vigorous stirring, or *vice versa;* in this case, the presence in the aqueous solution of the acidic or basic substance of a water-soluble high polymer having a dispersing ability such as polyethylene oxide or poval, a cellulose derivative such as hydroxyethylcellulose or a dispersing agent of a low molecular weight such as an anionic, cationic or nonionic surfactant makes the resulting resinous particles more spherical; when the coating composition comprising such spherical resin particles is applied to the surface of a substrate to be coated, the resin particles are arranged on the surface more densely so that a smoother and more lustrous coating film can be obtained;

(c) a procedure which comprises spraying the aqueous resin composition onto or into an aqueous solution of the acidic or basic substance, if necessary, while stirring; in this case, it is advantageous to make the dispersing agent present as in the above mentioned procedure (b);

(d) a procedure which comprises spraying the aqueous resin composition into the atmosphere of the acidic or basic gas; or

(e) a procedure which comprises contacting the aqueous resin composition in a mist form with an aqueous solution of the acidic or basic substance in a mist form.

By the above separation treatment, the neutralized ionic groups in the resin are made free carboxyl groups, and resinous particles are separated. As to the particle form of the resinous particles, the procedures (c), (d) and (e) bring about a rounder, more globular particle form as compared with the procedures (a) and (b). The particle sizes of the resinous particles can be appropriately controlled depending on the conditions of the separation treatment (e.g. the viscosity of the resin, the speed of stirring, the diameter of the spraying nozzle, the spraying viscosity).

The resinous particles formed in the separation treatment are then recovered by a conventional separation procedure such as filtration, sieving and centrifuging. If necessary, the mixture after the separation treatment may be treated with a neutralizing agent so as to neutralize the excessive acidic or basic substance.

The thus recovered resinous particles can be used as a coating composition. For a powder coating composition, the resinous particles may be employed as such. For a slurry coating composition, the resinous particles are dispersed in an aqueous medium and then used. When the resinous particles are contaminated with unfavourable impurities such as salts, the impurities may be removed by a conventional elimination procedure, for instance, by washing with water.

The application of the coating composition in a powdery form onto a substrate to be coated may be carried out by various procedures, e.g. air spraying, electrostatic coating, fluid bed coating etc. The applied coating composition is then dried and baked under suitable conditions whereby a smooth and lustrous coating film is formed.

When the coating composition in a slurry form is applied by a conventional spraying or roll coating procedure, evaporation of water from the wet film is faster than is the case when using a conventional water-soluble or emulsion type aqueous paint for baking, so that a coating film having a larger film thickness is readily obtainable, and thus the workability on the coating operation is greatly improved. Furthermore, it is confirmed that the formed coating film after backing is superior in smoothness and lustre to a coating film obtained from a conventional powdery paint.

The coating composition of the invention can be obtained according to the invention substantially without noise and the scattering of dust, the explosion of dust and other dangers at the pulverizing step, unlike the conventional process which comprises the step of pulverizing a solid composition. Besides, the particle form and the particle size can be readily controlled as mentioned above, and the kind of resin to be used may be optionally chosen in a wide range.

According to the process of the invention, the formation of a large amount of a solvent mixture as the byproduct is not caused unlike the conventional method in which the resin particles are precipitated by utilizing the difference between the dissolving abilities of solvents relative to the resin components, so that environmental pollution is prevented. Besides, it is advantageous from the economical viewpoint that the operations required for recycling, recovery or removal of such solvent mixture in the production or coating step would be simplified. In addition, the risk of explosion to be apprehended in the case of using acetone or other solvents can be minimized.

Since the aqueous resin composition before the separation treatment is in a solution or dispersion state, conventional techniques for the dispersion of a pigment into a liquid medium can be utilized as such. The adsorption of the resin on to the pigment is thus effectively attained, as compared with a conventional powdery paint or a paint in slurry form obtained by dispersing a powdery paint in water, so that a coating film having excellent luster and smoothness can be obtained. In addition, a conventional colour matching procedure may be adopted to realize a matching with ease and a high degree of precision.

Practical and presently preferred embodiments of the invention are illustratively shown in the following Examples wherein parts and percentages are by weight.

EXAMPLE 1

Thirty parts of an aqueous solution comprising a water-soluble acrylic resin (which comprises units of acrylic acid, hydroxpropyl acrylate, methyl methacrylate, *n*-butyl methacrylate, styrene and *n*-butyl acrylate in a weight ratio of 14.5:8.9:14.9:16.1:26:16.6, respectively, 100% of the carboxyl groups in the molecule being neutralized with dimethylethanolamine) in a concentration of 38%, 2.9 parts of hexamethoxymethylmelamine and 0.71 part of water-dispersible aluminium powder are mixed together, and the resulting mixture is admixed with 30 parts of water to make an aqueous resin composition.

The aqueous resin composition (63.61 parts) is added dropwise to a 1/20 N aqueous hydrochloric acid solution (500 parts) in about 15 minutes while stirring by means of a high-speed mixer. After completion of the addition, excessive hydrochloric acid in the resulting mixture was neutralized with a 1 N aqueous potassium hydroxide solution and sieved through a 250 mesh sieve to obtain a dispersion of resinous particles hving a particle size of 10 to 30μ. The resin particles were washed with water and dispersed in water to make a coating composition in a slurry form having a solids content of 50%.

The coating composition in slurry form was applied to a polished steel plate by air spraying and baked at 160°C for 30 minutes, whereby a smooth and lustrous metallic finish coating film was formed. When the coating film was subjected to an impact test of the DuPont method, a value of 20 cm or more (500 g) was obtained. It was thus confirmed that sufficient crosslinking had been attained.

EXAMPLE 2

The dispersion of resin particles obtained in Example 1 was washed with water and dried at 50°C for 3 hours in a fluidized bed drier to make a powder coating composition. The powder coating composition was applied to a steel plate by electrostatic coating and baked at 160°C for 30 minutes, whereby a well crosslinked, smooth and lustrous metallic finish coating film was formed.

EXAMPLE 3

One hundred parts of an isopropanol solution comprising a polyester resin (which has a number average molecular weight of 1500 and an acid value of 110, and in which 50% of the carboxyl groups were neutralized with triethylamine), in a concentration of 70%, 30 parts of benzoguanamine 60 parts of titanium oxide ("Tipake R–820", trade mark of Ishihara Sangyo Kaisha, Ltd.) and 640 parts of water were mixed together to form an aqueous resin composition.

The aqueous resin composition was air sprayed onto the surface of a 1/20 N aqueous hydrochloric acid solution charged into a vessel while stirring. The precipitated materials were collected by centrifuging, washed with water and dispersed in water to make a coating composition in a slurry form having a solids content of 50%.

The coating composition in slurry form was applied to a polished steel plate by air spraying and baked at 160°C for 30 minutes, whereby a smooth and lustrous coating film was formed. When the coating film was subjected to an extrusion test using an Erichsen tester, a value of 3 mm or more was obtained. It was thus confirmed that sufficient crosslinking had been attained.

EXAMPLE 4

The aqueous resin composition used in Example 3 was air sprayed onto the surface of a 1/20 N aqueous hydrochloric acid solution charged into a vessel while stirring. The precipitated materials were collected by centrifuging, washed with water and dried at 50°C for 3 hours in a fluidized bed drier to prepare a powder coating composition, which was applied to a steel plate by electrostatic coating, and baked at 160°C for 30 minutes, whereby a well crosslinked, smooth and lustrous coating film was formed.

EXAMPLE 5

The polyester resin dispersion employed in Example 3 (100 parts) and a polyisocyanate compound containing a blocked isocyanate group (14 parts) were melt mixed at a temperature higher than the melting temperature of the polyisocyanate compound (e.g. 90°C), and 70% of the carboxyl groups in the resin mixture were neutralized with dimethylethanolamine. The resulting mixture (114 parts) was admixed with titanium oxide as employed in Example 3 (30 parts) and water (350 parts) to make an aqueous resin composition.

The aqueous resin composition was air sprayed into a stream of carbon dioxide to make resinous spherical particles, which were washed with water and dispersed in water to prepare a coating composition in a slurry form having a solids content of 50%. Alternatively, the resinous spherical particles were washed with water and dried to prepare a powder coating composition.

EXAMPLE 6

Acrylic acid (15 parts), 2-hydroxyethyl methacrylate (15 parts), methyl methacrylate (38 parts), styrene (19 parts) and *n*-butyl acrylate (8 parts) were mixed together to make a liquid composition. The resulting liquid composition (20 parts) was charged into a flask, and, while stirring at 130°C, a mixture of the liquid composition (75 parts) and azobisisobutyronitrile (1.5 parts) was added dropwise thereto in 3 hours. After completion of the addition, stirring was continued at the same temperature for 1 hour. Then, a 10% aqueous solution of sodium hydroxide (36 parts) was added to the resulting mixture at 110°C, and, after stirring for 30 minutes, water (150 parts) was added thereto at a temperature of below 100°C to yield a resin mixture. To the resin mixture (100 parts), hexamethoxymethylmelamine (10 parts) and aluminium powder ("Alpaste 5105 WA", trade mark of Toyo Aluminium K.K.) (30 parts) were added, and the resulting mixture was thoroughly dispersed to make an aqueous resin composition.

The aqueous resin compositon (100 parts) was diluted with water (200 parts), and a 1/10 N aqueous hydrochloric acid solution (700 parts) was added thereto, followed by stirring with a labomixer. The resulting mixture was neutralised with a 1/10 N aqueous sodium hydroxide solution, and the aqueous medium was eliminated to yield resinous particles having a partcle size of 10 to 20μ. The resinous particles were washed with water to eliminate the sodium chloride produced and dispersed in water to make a coating composition in a slurry form having a solids content of 50%.

The coating composition in slurry form was applied to a polished steel plate by air spraying and baked at 160°C for 30 minutes, whereby a smooth and lustrous coating film was formed.

When the resinous particles were washed with water and dried at 50°C for 3 hours in a fluidized bed drier, there was obtained a powder coating composition which also afforded a coating film of satisfactory quality.

EXAMPLE 7

An epoxy resin "Epikote 1001" (trade mark), manufactured by Shell Chemical Corp.) (488 parts) and diethanolamine (105 parts) were reacted in isopropanol (250 parts) under a nitrogen stream at 80°C for 3 hours to make an aminoepoxy resin solution. To the aminoepoxy resin solution (143 parts), glacial acetic acid (62 parts) was added, deionized water (500 parts) was added thereto, and the resulting mixture was admixed with a blocked polyisocyanate resin solution in methylethylketone (solid content, 50%, 16 parts) and titanium oxide ("Tipake R–820" (trade mark) manufactured by Ishihara Sangyo Kaisha, Ltd.) (30 parts) to make an aqueous resin composition.

The aqueous resin composition (143 parts) was diluted with water (250 parts), and the resulting dilution was added dropwise to a 0.05 N aqueous solution of potassium hydroxide with vigorous stirring. After excessive alkali had been neutralized with 0.1 N acetic acid, the resulting mixture was subjected to centrifugal separation to collect resinous particles of not more than 50μ in diameter. The resinous particles were washed with water and then dispersed in water to make a coating composition in a slurry form having a solids content of 50%.

The coating composition in slurry form was applied to a polished steel plate by air spraying and baked at 180°C for 20 minutes, whereby a smooth and lustrous coating film was formed. When the coating film was subjected to the Du-Pont impact test, a value of 20 cm or more (500 g) was obtained. It was thus confirmed that sufficient crosslinking had been attained.

EXAMPLE 8

An aqueous dispersion comprising an aminoacrylic resin (comprising as the monomeric constituents dimethylaminoethyl, methacrylate, methyl methacrylate, *n*-butyl acrylate, 2-hydroxyethyl methacrylate and styrene; glass transition point, 65°C; weight average molecular weight: 18,000; content of non-volatile components: 80%; dispersing medium, isopropanol: water = 1:1 by weight) (100 parts) was admixed with hexamethoxymethylmelamine ("Cymel 303" (trade mark) manufactured by American Cyanamid) (20 parts), a mixture of powdery aluminium ("L–579" manufactured by Silverline Mfg. Co.) (5 parts) and isopropanol (5 parts) qws added thereto, and the resulting mixture was admixed with 0.1 N hydrochloric acid to make an aqueous resin composition.

To the aqueous resin composition (100 parts), a mixture of 0.1 N sodium hydroxide solution (500 parts) and polyethylene oxide (10 parts) was added, and the resulting mixture was stirred with the aid of a high-speed mixer for 15 minutes. After excessive alkali had been neutralized with 0.1 N hydrochloric acid, the resulting mixture was subjected to centrifugal separation to collect resinous particles of not more than 50μ in particle size. The resinous particles were washed with water and dispersed in water to make a coating composition in a slurry form having a solids content of 50%.

The coating composition in slurry form was applied to a polished steel plate and baked as in Example 7, whereby a smooth and lustrous metallic finish coating film was formed. By the DuPont impact test method, it was confirmed that sufficient crosslinking had been attained.

EXAMPLE 9

The resinous particles as collected in Example 8 were washed with water and dried by the use of a fluidized bed type dryer to make a coating composition in a powder form. The coating composition was applied electrostatically to a polished steel plate and then baked at 160°C for 20 minutes, whereby a smooth and lustrous coating film with a good metallic feel was formed. By the DuPont impact test method, it was confirmed that sufficient crosslinking had been attained.

WHAT WE CLAIM IS:—

1. A process for preparing resinous particles for a coating composition in which (1) an aqueous resin composition, in which a resin having at least one ionic group but substantially without any material solubility or dispersibility relative to water is dissolved or dispersed in an aqueous medium by at least partial neutralization of the ionic group or groups in the resin is subjected to treatment with a powdering agent and to simultaneous pulverization treatment (as herein defined), whereby the resin is deposited in particulate form; and (2) the deposited resinous particles are recovered from the aqueous medium.

2. A process according to claim 1, wherein the powdering agent has the capability of acting on the neutralized ionic group or groups in the resin to make it or them free.

3. A process according to claim 1 or 2, wherein the aqueous resin composition comprises additionally a water-soluble or water-insoluble resin with or without a pigment.

4. A process according to claim 3, wherein the water-insoluble resin is crosslinkable with one or more functional groups in the ionic group-containing resin.

5. A process according to claim 1 or 2, wherein the resin is a carboxyl group-containing resin, in which the carboxyl groups are at least partly neutralized with an organic or inorganic base, and the powdering agent is an acidic substance.

6. A process according to claim 1 or 2, wherein the resin is a basic group-containing resin, in which the the basic groups are at least partly neutralized with an organic or inorganic acid and the powdering agent is a basic substance.

7. A process for preparing a coating composition in a slurry form, which comprises dispersing in an aqueous medium the resinous particles prepared by a process according to any of claims 1 to 6.

8. A process for preparing a coating composition in a powdery form which comprises drying the resinous particles prepared by a process according to any of claims 1 to 6.

9. Resinous particles prepared by a process according to any of claims 1 to 6.

10. A coating composition in a slurry form prepared by a process according to claim 7.

11. A coating composition in a powdery form prepared by a process according to claim 8.

12. A process for prepring resinous particles for a coating composition substantially as herein described with reference to any specific examples.

13. Resinous particles for a coating composition when prepared by a process as claimed in claim 12.

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