

SPECIFICATION

DISPERSANT FOR CALCIUM CARBONATE AND METHOD FOR PRODUCING SAME

[FIELD OF THE INVENTION]

[0001]

The present invention relates to a dispersant for calcium carbonate and a method for producing the same. More specifically, the present invention relates to a dispersant for calcium carbonate, which leads to a calcium carbonate slurry having a reduced initial viscosity by wet grinding and prevents a significant increase in viscosity of the slurry with passage of time, and a method for producing the same.

[BACKGROUND ART]

[0002]

A calcium carbonate slurry obtained by wet grinding of calcium carbonate has been widely used as a paper coating agent, a paint pigment, and the like. When the slurry of calcium carbonate is produced, a dispersant for calcium carbonate is used in order to decrease the viscosity of the resulting calcium carbonate slurry.

A dispersant containing an acryl-based polymer is used as the dispersant for calcium carbonate in order to obtain a slurry having low viscosity.

Patent Literature 1 discloses a method for producing a dispersant wherein a polymer having a low molecular weight is produced using water as a solvent, and using a large amount of a chain transfer agent such as sodium hypophosphite. However, a calcium carbonate slurry prepared using a dispersant obtained by this method exhibits high viscosity, and may show a significant increase in viscosity with passage of time.

As a method without using a chain transfer agent such as sodium hypophosphite, Patent Literature 2 discloses a method wherein a dispersant containing a polymer that contains a structural unit from a vinyl group-containing monocarboxylic acid (salt) and has a weight average molecular weight of 4,000 to 40,000 and a Mw/Mn of 1.2 to 2.0 is produced using a persulfate as an initiator, and using isopropyl alcohol as both a reaction solvent and a chain transfer agent. Patent Literatures 3 and 4 also disclose a same method as that in Patent Literature 2 wherein a dispersant is produced using a persulfate as an initiator, and using isopropyl alcohol as a reaction solvent and a chain transfer agent.

[PRIOR ART LITERATURE]

[PATENT LITERATURE]

[0003]

Patent Literature 1: JP A S60-174793

Patent Literature 2: JP A 2004-306022

Patent Literature 3: JP A 2004-315359

Patent Literature 4: WO 2004-087574

[SUMMARY OF THE INVENTION]

[PROBLEMS THAT THE INVENTION IS TO SOLVE]

[0004]

However, since the methods disclosed in Patent Literatures 2 to 4 utilize only a large amount of persulfate as an initiator, the resulting dispersants do not exhibit satisfactory performance.

The invention was conceived in view of the above problem. An object of the present invention is to provide a dispersant for calcium carbonate, which leads to a calcium carbonate slurry having a reduced initial viscosity and being excellent in long-term dispersion stability and prevents a significant increase in viscosity of the slurry with passage of time, and a method for producing the same.

[0005]

The inventors conducted extensive studies in order to solve the above-mentioned problems and to obtain a dispersant for calcium carbonate in which an initial viscosity of a calcium carbonate slurry obtained by wet grinding or the like is low and there is no significant increase in viscosity with passage of time. The inventors found that a dispersant for calcium carbonate can be produced which leads to a calcium carbonate slurry having a reduced initial viscosity and being excellent in long-term dispersion stability and prevents a significant increase in viscosity of the slurry with passage of time by a method including a polymerization process in which a monomer containing acrylic acid is polymerized in the presence of a hypophosphite and a persulfate using an isopropyl alcohol aqueous solution as a solvent, and a neutralization process in which 15 to 95 mol% of carboxyl groups included in a structural unit derived from the monomer constituting a formed polymer is neutralized.

[0006]

The present invention is as follows.

1. A dispersant for calcium carbonate characterized in that the dispersant comprises an acryl-based polymer that is obtained by a method comprising a polymerization process in which

a monomer containing acrylic acid is polymerized in the presence of a hypophosphite and a persulfate using an isopropyl alcohol aqueous solution as a solvent, and a neutralization process in which 15 to 95 mol% of carboxyl groups included in a structural unit derived from the monomer constituting a formed polymer is neutralized, that the hypophosphite is used in an amount from 1.4 to 4.4 parts by mass based on 100 parts by mass of the monomer, that the persulfate is used in an amount from 0.3 to 1.5 parts by mass based on 100 parts by mass of the monomer, that a reaction temperature employed in the polymerization process is between 68°C and 82°C, and that a weight average molecular weight of the acryl-based polymer is in the range from 4,500 to 8,500.

2. The dispersant for calcium carbonate according to 1 above, wherein the method further comprises, after the polymerization process, a concentration process in which the isopropyl alcohol is distilled off.

3. The dispersant for calcium carbonate according to 2 above, wherein the isopropyl alcohol recovered after the concentration process is used in the polymerization process.

4. The dispersant for calcium carbonate according to any one of 1 to 3 above, wherein a concentration of the isopropyl alcohol in the isopropyl alcohol aqueous solution is in the range from 15% to 55% by mass.

5. The dispersant for calcium carbonate according to any one of 1 to 4 above, wherein a content of the acrylic acid used in the polymerization process is in the range from 80% to 100% by mass based on 100% by mass of a total amount of the monomer.

6. A production method of a dispersant for calcium carbonate containing an acryl-based polymer, characterized in that the method comprises a polymerization process in which a monomer containing acrylic acid is polymerized in the presence of a hypophosphite and a persulfate using an isopropyl alcohol aqueous solution as a solvent, and a neutralization process in which 15 to 95 mol% of carboxyl groups included in a structural unit derived from the monomer constituting a formed polymer is neutralized, that the hypophosphite is used in an amount from 1.4 to 4.4 parts by mass based on 100 parts by mass of the monomer, that the persulfate is used in an amount from 0.3 to 1.5 parts by mass based on 100 parts by mass of the monomer, that a reaction temperature employed in the polymerization process is between 68°C and 82°C, and that a weight average molecular weight of the acryl-based polymer is in the range from 4,500 to 8,500.

7. The production method of the dispersant for calcium carbonate according to 6 above, further comprising a concentration process in which the isopropyl alcohol is distilled off, after the polymerization process.

8. The production method of the dispersant for calcium carbonate according to 7 above,

wherein the isopropyl alcohol recovered after the concentration process is used in the polymerization process.

9. The production method of the dispersant for calcium carbonate according to any one of 6 to 8 above, wherein a concentration of the isopropyl alcohol in the isopropyl alcohol aqueous solution is in the range from 15% to 55% by mass.

10. The production method of the dispersant for calcium carbonate according to any one of 6 to 9 above, wherein a content of the acrylic acid used in the polymerization process is in the range from 80% to 100% by mass based on 100% by mass of a total amount of the monomer.

[EFFECT OF THE INVENTION]

[0007]

The dispersant for calcium carbonate of the present invention is excellent in dispersibility of calcium carbonate and exhibits excellent dispersibility and long-term dispersion stability particularly as a dispersant for wet grinding of calcium carbonate. In the dispersant for calcium carbonate of the present invention, since isopropyl alcohol that functions as a chain transfer agent is used as a solvent, and a hypophosphite as a chain transfer agent and a persulfate as an initiator are used in combination, the amount of by-products (e.g., phosphites and phosphates) produced from the hypophosphite and the amount of by-products (e.g., sulfates) produced from the persulfate are reduced. Use of the dispersant for calcium carbonate of the present invention leads to a calcium carbonate slurry that has low initial viscosity and does not show a significant increase in viscosity with passage of time.

In the case where the content of acrylic acid used in the polymerization process is in the range from 80% to 100% by mass based on 100% by mass of a total amount of the monomer, the dispersant for calcium carbonate can more advantageously disperse calcium carbonate.

[0008]

The production method of a dispersant for calcium carbonate of the present invention can efficiently produce a dispersant for calcium carbonate leading to a calcium carbonate slurry that exhibits low initial viscosity, and does not show a significant increase in viscosity with passage of time.

In the case where a concentration process in which isopropyl alcohol is evaporated is provided after polymerization process, a dispersant for calcium carbonate can be efficiently produced in which the amount of isopropyl alcohol is reduced.

In the case where the isopropyl alcohol recovered after the concentration process is used in the polymerization process, evaporated isopropyl alcohol can be reused in the polymerization process and need not be disposed of. Therefore, it is possible to provide a method for

producing a dispersant for calcium carbonate that can reduce cost, and achieve excellent production efficiency and environmental protection.

In the case where the content of the acrylic acid used in the polymerization process is in the range from 80% to 100% by mass based on 100% by mass of a total amount of the monomer, it is possible to efficiently produce a dispersant for calcium carbonate that can more advantageously disperse calcium carbonate.

[DESCRIPTION OF EMBODIMENTS]

[0009]

In the following, the dispersant for calcium carbonate of the present invention (hereinafter, referred to also as “dispersant” simply) and a production method of a dispersant for calcium carbonate of the present invention are described in detail.

The present invention is characterized in that an acryl-based polymer is included by a production method having a polymerization process in which a monomer containing acrylic acid is polymerized in the presence of a hypophosphite and a persulfate using an isopropyl alcohol aqueous solution as a solvent, and a neutralization process in which 15 to 95 mol% of carboxyl groups included in a structural unit derived from the monomer constituting a formed polymer (hereinafter referred to also as “a first polymer”) is neutralized, that the hypophosphite is used in an amount from 1.4 to 4.4 parts by mass based on 100 parts by mass of the monomer, that the persulfate is used in an amount from 0.3 to 1.5 parts by mass based on 100 parts by mass of the monomer, that a reaction temperature employed in the polymerization process is between 68°C and 82°C, and that a weight average molecular weight of the acryl-based polymer is in the range from 4,500 to 8,500.

In the description of the present invention, “(meth)acryl” means acryl and/or methacryl.

[0010]

The dispersant of the present invention is obtained by the polymerization process and the neutralization process. The raw materials used for the production of the dispersant of the present invention are described below.

[0011]

In the polymerization process, the monomer that is subjected to polymerization includes at least acrylic acid. Specifically, the monomer may include only acrylic acid, or may further include a monomer other than acrylic acid.

The monomer other than acrylic acid (hereinafter referred to as “other monomer”) is not particularly limited so long as the monomer is copolymerizable with the acrylic acid. Examples of the other monomer include a radically-polymerizable vinyl-based monomer

(polymerizable unsaturated compound). Examples of the vinyl-based monomer include an ethylenically unsaturated carboxylic acid other than acrylic acid, a neutralized salt of ethylenically unsaturated carboxylic acid, an alkyl (meth)acrylate compound, an aromatic vinyl compound, an acid anhydride, an amino group-containing vinyl compound, an amide group-containing vinyl compound, a sulfonic acid group-containing vinyl compound, a polyoxyalkylene group-containing vinyl compound, an alkoxy group-containing vinyl compound, a cyano group-containing vinyl compound, a cyanidated vinyl compound, a vinyl ether compound, a vinyl ester compound, a conjugated diene, and the like. These compounds may be used singly or in combination of two or more types thereof.

Among these, an alkyl (meth)acrylate compound and a polyoxyalkylene group-containing vinyl compound are preferable from the viewpoint of the properties (e.g., dispersibility and suppression of coloration) of the resulting dispersant.

[0012]

Examples of the ethylenically unsaturated carboxylic acid other than acrylic acid include methacrylic acid, ethacrylic acid, maleic acid, fumaric acid, itaconic acid, crotonic acid, a half-ester of phthalic anhydride with an alkyl alcohol, a half-ester of itaconic anhydride with an alkyl alcohol, and the like.

[0013]

Examples of the neutralized salt of ethylenically unsaturated carboxylic acid include ethylenically unsaturated carboxylates obtained by neutralizing a carboxyl group in acrylic acid, methacrylic acid, ethacrylic acid, maleic acid, fumaric acid, itaconic acid, crotonic acid, and the like. The ethylenically unsaturated carboxylate may be an alkali metal salt, an alkaline-earth metal salt, an ammonium salt, an organic amine salt, or the like.

[0014]

Examples of the alkyl (meth)acrylate compound include methyl (meth)acrylate, ethyl (meth)acrylate, n-propyl (meth)acrylate, isopropyl (meth)acrylate, n-butyl (meth)acrylate, isobutyl (meth)acrylate, sec-butyl (meth)acrylate, tert-butyl (meth)acrylate, n-pentyl (meth)acrylate, isoamyl (meth)acrylate, n-hexyl (meth)acrylate, 2-methylpentyl (meth)acrylate, n-octyl (meth)acrylate, 2-ethylhexyl (meth)acrylate, n-decyl (meth)acrylate, n-dodecyl (meth)acrylate, n-octadecyl (meth)acrylate, isobornyl (meth)acrylate, cyclohexyl (meth)acrylate, phenyl (meth)acrylate, benzyl (meth)acrylate, and the like.

[0015]

Examples of the aromatic vinyl compound include styrene, 2-methylstyrene, 3-methylstyrene, 4-methylstyrene, α -methylstyrene, 2,4-dimethylstyrene, 2,4-diisopropylstyrene, 4-tert-butylstyrene, tert-butoxystyrene, vinyltoluene, vinylnaphthalene, halogenated styrene,

styrenesulfonic acid, α -methylstyrenesulfonic acid, and the like.

[0016]

Examples of the acid anhydride include maleic anhydride, itaconic anhydride, citraconic anhydride, and the like.

[0017]

Examples of the amino group-containing vinyl compound include dimethylaminomethyl (meth)acrylate, diethylaminomethyl (meth)acrylate, 2-dimethylaminoethyl (meth)acrylate, 2-diethylaminoethyl (meth)acrylate, 2-(di-n-propylamino)ethyl (meth)acrylate, 2-dimethylaminopropyl (meth)acrylate, 2-diethylaminopropyl (meth)acrylate, 2-(di-n-propylamino)propyl (meth)acrylate, 3-dimethylaminopropyl (meth)acrylate, 3-diethylaminopropyl (meth)acrylate, 3-(di-n-propylamino)propyl (meth)acrylate, and the like.

[0018]

Examples of the amide group-containing vinyl compound include (meth)acrylamide, N,N-dimethyl(meth)acrylamide, N,N-dimethylaminopropyl(meth)acrylamide, N-methylol(meth)acrylamide, and the like.

[0019]

Examples of the sulfonic acid group-containing vinyl compound include methallylsulfonic acid, acrylamide-2-methyl-2-propanesulfonic acid, and the like.

[0020]

Examples of the polyoxyalkylene group-containing vinyl compound include esters of (meth)acrylic acid and an alcohol having a polyoxyethylene group and/or a polyoxypropylene group, and the like.

[0021]

Examples of the alkoxy group-containing vinyl compound include 2-methoxyethyl (meth)acrylate, 2-ethoxyethyl (meth)acrylate, 2-(n-propoxy)ethyl (meth)acrylate, 2-(n-butoxy)ethyl (meth)acrylate, 3-methoxypropyl (meth)acrylate, 3-ethoxypropyl (meth)acrylate, 2-(n-propoxy)propyl (meth)acrylate, 2-(n-butoxy)propyl (meth)acrylate, and the like.

[0022]

Examples of the cyano group-containing (meth)acrylate compound include cyanomethyl (meth)acrylate, 1-cyanoethyl (meth)acrylate, 2-cyanoethyl (meth)acrylate, 1-cyanopropyl (meth)acrylate, 2-cyanopropyl (meth)acrylate, 3-cyanopropyl (meth)acrylate, 4-cyano butyl (meth)acrylate, 6-cyano hexyl (meth)acrylate, 2-ethyl-6-cyano hexyl (meth)acrylate, 8-cyano octyl (meth)acrylate, and the like.

[0023]

Examples of the cyanidated vinyl compound include acrylonitrile, methacrylonitrile, ethacrylonitrile, and the like.

[0024]

Examples of the vinyl ether compound include vinyl methyl ether, vinyl ethyl ether, vinyl n-butyl ether, vinyl phenyl ether, vinyl cyclohexyl ether, and the like. These compounds may be used singly or in combination of two or more types thereof.

Examples of the vinyl ester compound include vinyl formate, vinyl acetate, vinyl propionate, and the like.

[0025]

Examples of the conjugated diene include 1,3-butadiene, isoprene, 2,3-dimethyl-1,3-butadiene, 1,3-pentadiene, 2-methyl-1,3-pentadiene, 1,3-hexadiene, 4,5-diethyl-1,3-octadiene, 3-butyl-1,3-octadiene, chloroprene, and the like.

[0026]

Other examples include a maleimide compound such as maleimide, N-methylmaleimide, N-butylmaleimide, N-phenylmaleimide, and N-cyclohexylmaleimide; a maleate compound; an itaconate compounds; a N-vinyl heterocyclic compound such as vinylpyridine; and the like.

[0027]

Among these, methyl acrylate and butyl acrylate are preferable. A dispersant that advantageously suppresses coloration can be obtained when using methyl acrylate, butyl acrylate, or the like in combination with acrylic acid.

[0028]

In the case where the monomer used in the polymerization process includes other monomer other than acrylic acid, the content of acrylic acid in the monomer is preferably 70% or more by mass, more preferably 80% or more by mass, and further preferably 90% or more by mass. It is particularly preferable that the monomer is acrylic acid (100% by mass). When the content of acrylic acid in the monomer is 70% or more by mass, the resulting dispersant exhibits sufficient solubility in water.

[0029]

In the polymerization process, the hypophosphite is used as a chain transfer agent. The hypophosphite is not particularly limited so long as the hypophosphite is a compound having a chain transfer effect. Specific examples of the hypophosphite include sodium hypophosphite, potassium hypophosphite, lithium hypophosphite, calcium hypophosphite, magnesium hypophosphite, barium hypophosphite, and the like. These compounds may be used singly or in combination of two or more types thereof. Among these, sodium

hypophosphite is preferable.

[0030]

The usage amount of the hypophosphite is in the range from 1.4 to 4.4 parts by mass, preferably from 2.0 to 4.0 parts by mass, and more preferably from 2.5 to 3.5 parts by mass based on 100 parts by mass of the monomer. When the hypophosphite is used in an amount within the above range, a polymer having a weight average molecular weight of 4,500 to 8,500 can be efficiently obtained. When the usage amount of the hypophosphite is 4.4 parts or less by mass, a dispersant containing a small amount of by-product phosphites and phosphates is obtained. The resulting dispersant leads to a calcium carbonate slurry having a reduced initial viscosity by wet grinding, prevents a significant increase in viscosity of the slurry with passage of time, and exhibits excellent long-term dispersibility. If the usage amount of the hypophosphite is too large (e.g., more than 4.4 parts by mass), the calcium carbonate slurry exhibits high initial viscosity, and shows a significant increase in viscosity with passage of time. It is inferable that this is because phosphite and phosphate as by-products produced from the hypophosphite increase, and the by-products and calcium carbonate form a hardly soluble compound to thereby lead to viscosity increase.

[0031]

In the polymerization process, the persulfate is used as an initiator (radical initiator). Examples of the persulfate include sodium persulfate, ammonium persulfate, potassium persulfate, and the like. These compounds may be used singly or in combination of two or more types thereof. It is preferable to use sodium persulfate or potassium persulfate since a volatile component is rarely generated in the neutralization process performed after the polymerization process.

[0032]

The usage amount of the persulfate is in the range from 0.3 to 1.5 parts by mass, preferably from 0.4 to 1.4 parts by mass, and more preferably from 0.6 to 1.2 parts by mass based on 100 parts by mass of the monomer. When the persulfate is used in an amount within the above range, the amount of unreacted monomers can be reduced. When the amount of the persulfate is 1.5 parts or less by mass, the resulting dispersant contains only a small amount of by-product sulfates. If the usage amount of the persulfate is too large (e.g., more than 1.5 parts by mass), the calcium carbonate slurry exhibits high initial viscosity, and shows a significant increase in viscosity with passage of time. It is inferable that this is because a sulfate as by-products produced from the persulfate increases in the same manner as the hypophosphite, and the by-products and calcium carbonate form a hardly soluble compound to thereby lead to viscosity increase.

[0033]

In the polymerization process, an isopropyl alcohol aqueous solution is used as a solvent.

Isopropyl alcohol also functions as a chain transfer agent in the polymerization process. Therefore, the isopropyl alcohol aqueous solution is used as a reaction solvent and a chain transfer agent.

A concentration of isopropyl alcohol in the isopropyl alcohol aqueous solution is preferably 5% or more by mass and 90% or less by mass, more preferably in the range from 10% to 80% by mass, further preferably from 15% to 60% by mass, and particularly from 15% to 55% by mass. The concentration thereof may be from 20% to 50% by mass, or from 30% to 50% by mass.

When the isopropyl alcohol concentration is 5% or more by mass, the isopropyl alcohol effectively exhibits the chain transfer effect (i.e., effectively functions as the chain transfer agent). The chain transfer effect is improved as the isopropyl alcohol concentration increases.

[0034]

In the polymerization process, the usage amount of isopropyl alcohol is preferably in the range from 15 to 80 parts by mass, and more preferably from 45 to 75 parts by mass based on 100 parts by mass of the monomer. When isopropyl alcohol is used in an amount of 15 parts or more by mass, the chain transfer effect of isopropyl alcohol is effectively exhibited. When isopropyl alcohol is used in an amount of 80 parts or less by mass, a solubility of the raw material may be improved.

[0035]

The polymerization process is a process in which a monomer containing acrylic acid is polymerized in the presence of a hypophosphite and a persulfate using an isopropyl alcohol aqueous solution as a reaction solvent to form a first polymer having a structural unit derived from the monomer.

[0036]

The reaction temperature employed in the polymerization process is between 68°C and 82°C, and more preferably between 70°C and 80°C. When the reaction temperature is 82°C or lower, polymerization proceeds smoothly, and oxidation of hypophosphorous acid (chain transfer agent) to phosphites and phosphates can be suppressed, so that a dispersant containing only a small amount of by-product phosphites and phosphates can be obtained. When the reaction temperature is 68°C or higher, a dispersant containing only a small amount of unreacted monomers can be obtained.

[0037]

The polymerization process may be implemented by batch polymerization or continuous polymerization. In the case of batch polymerization, a feeding time of the raw materials (raw material composition) including the monomer and the like is preferably in the range from 2 to 12 hours, and more preferably from 3 to 8 hours. When the feeding time is 2 hours or longer, the heat of polymerization can be easily removed. When the feeding time is 12 hours or shorter, productivity is improved.

A known reactor for batch polymerization may be used as a reaction apparatus. It is preferable to use a reaction apparatus equipped with a stirrer and a temperature control system. The stirrer is not specifically limited so long as a publicly known stirring blade is installed, and examples of the stirring blade include an anchor blade, a paddle blade, a propeller blade, a turbine blade, a ribbon blade, a large flat-plate blade, and the like. Examples of the device for temperature control include a jacket, an inner coil, a well-known internal and/or external heat exchanger such as a plate type heat exchanger. For the purpose of enhancing stirring efficiency and so on, a baffle and the like may be arranged in the reactor if necessary.

In the case of continuous polymerization, it is preferable to use a multi-stage CSTR (continuous vessel-type reactor having a plurality of reaction tanks). In this case, the average residence time in each reaction tank is preferably in the range from 60 to 240 minutes, and more preferably from 80 to 180 minutes. When the average residence time is 60 minutes or longer, the amount of unreacted monomers can be reduced. When the average residence time is 240 minutes or shorter, the size of the reaction tank can be decreased.

For the continuous polymerization, the stirrer, temperature control system, and the like used for batch polymerization may also be used.

[0038]

A polymerization method for the first polymer is not particularly limited in the polymerization process. Example thereof includes the following polymerization methods (1) to (3).

- (1) A predetermined amount of the isopropyl alcohol aqueous solution is put in a reaction vessel. After that, a raw material mixture consisting of a monomer, an isopropyl alcohol aqueous solution, and a hypophosphite, and a persulfate are respectively added dropwise to the reaction vessel to conduct polymerization.
- (2) A raw material mixture consisting of a monomer, an isopropyl alcohol aqueous solution, and a hypophosphite is prepared, and then the raw material mixture and a persulfate are added dropwise to a reaction vessel to conduct polymerization.
- (3) An isopropyl alcohol aqueous solution is put in a reaction vessel, and is kept at a temperature equal to or close to the reaction temperature. After that, a monomer, a

hypophosphite, and a persulfate are added dropwise to the reaction vessel to conduct polymerization.

Among these, it is preferable to use the polymerization method (1) since a homogenous polymer is obtained.

[0039]

Specific process of the embodiment (1) is as follows. A predetermined amount (20% to 80% by mass based on the total amount) of the isopropyl alcohol aqueous solution is put in a reaction vessel, and is kept at a temperature equal to or close to the reaction temperature. On the other hand, the remaining isopropyl alcohol aqueous solution, a monomer, and a hypophosphite are mixed to prepare a raw material mixture. The raw material mixture and a persulfate are added dropwise to the reaction vessel including the isopropyl alcohol aqueous solution to conduct polymerization.

[0040]

In the polymerization process, an isopropyl alcohol having a function as a chain transfer agent is used as the solvent while using the hypophosphite (chain transfer agent) and the persulfate (initiator) in combination. Therefore, when the dispersant is used, a calcium carbonate slurry can be obtained in which an initial viscosity is low and a significant viscosity increase with passage of time can be suppressed. In this regard, when usage amounts of the hypophosphite and persulfate are reduced, an amount of by-products such as a phosphite and phosphate generated from hypophosphite and a sulfate generated from persulfate is small, formation of a hardly soluble compound from the by-products and calcium carbonate can be suppressed, whereby an initial viscosity of a slurry becomes low, and a significant viscosity increase with passage of time can be suppressed.

[0041]

According to the polymerization process, the monomer containing acrylic acid forms the first polymer. A weight average molecular weight of the first polymer is preferably in the range from 4,500 to 8,500. The weight-average molecular weight can be measured by gel permeation chromatography (GPC), using a standard material such as sodium polyacrylate. The first polymer having a weight average molecular weight of 4,500 to 8,500 leads to the acryl-based polymer obtained by the neutralization process having a weight average molecular weight of 4,500 to 8,500.

[0042]

The neutralization process is a process in which a carboxyl group included in a structural unit derived from the monomer constituting the first polymer obtained in the polymerization process is neutralized. In the neutralization process, some of the carboxyl

groups are neutralized by the neutralization process to obtain an acryl-based polymer that is to be included in the dispersant.

[0043]

In the neutralization process, the carboxyl groups are neutralized using a basic compound. The basic compound is not particularly limited so long as the basic compound can neutralize a carboxyl group. Examples of the basic compound include an inorganic basic compound and an organic basic compound. These compounds may be used singly or in combination of two or more types thereof.

Examples of the inorganic basic compound include an alkali metal hydroxide such as sodium hydroxide, potassium hydroxide, and lithium hydroxide; an alkaline-earth metal hydroxide such as calcium hydroxide and magnesium hydroxide; aqueous ammonia, and the like.

Examples of the organic basic compound include an organic amine compound such as monoethanolamine and triethanolamine. Among these, an alkali metal hydroxide is preferable since a volatile component is generated to only a small extent. Sodium hydroxide is more preferable.

[0044]

In the neutralization process, the neutralization rate of the carboxyl groups included in the first polymer is in the range from 15 to 95 mol%, and preferably from 20 to 90 mol%. When the neutralization rate is in the range from 15 to 95 mol%, a dispersant can be obtained which advantageously disperses calcium carbonate.

[0045]

The acryl-based polymer is a polymer which is obtained by polymerizing the monomer containing acrylic acid, and neutralizing some of carboxyl groups derived from the monomer. The acryl-based polymer may include a unit derived from the hypophosphite and the persulfate used for polymerization at the end thereof, for example.

A weight average molecular weight of the acryl-based polymer is in the range from 4,500 to 8,500, and preferably from 5,000 to 8,000. The weight-average molecular weight can be, as mentioned above, measured by GPC using a standard material such as sodium polyacrylate. When the weight average molecular weight of the acryl-based polymer is within the above range, a dispersant can be obtained which advantageously disperses calcium carbonate. And when the dispersant is used, a calcium carbonate slurry can be obtained in which an initial viscosity is low and a significant viscosity increase with passage of time can be suppressed.

[0046]

For the dispersant of the present invention, in addition to the polymerization process and neutralization process, a concentration process may be provided after the polymerization process. The concentration process is preferably provided between the polymerization process and neutralization process.

[0047]

The concentration process is a process in which isopropyl alcohol is distilled off from a reaction liquid containing the first polymer obtained in the polymerization process to prepare a concentrated composition containing reduced isopropyl alcohol.

[0048]

In the concentration process, the reaction system is subjected to depressurizing and/or heating to distill isopropyl alcohol out of the reaction system. Isopropyl alcohol can thus be evaporated off from the reaction liquid. Isopropyl alcohol is normally evaporated off as an azeotrope with water. Therefore, isopropyl alcohol is distilled off from the reaction liquid as an aqueous solution thereof to form a concentrated composition having a reduced isopropyl alcohol content and a reduced water content.

[0049]

In the concentration process, isopropyl alcohol may be distilled off by an arbitrary method. For example, when the reaction system is subjected to depressurizing, and the internal temperature of the reaction system is kept at a temperature equal to or higher than the azeotropic temperature of isopropyl alcohol to evaporate water and isopropyl alcohol off from the reaction system. Alternatively, water and isopropyl alcohol may be evaporated off from the reaction system by passing the reaction mixture through a depressurized thin film evaporator.

[0050]

A content of isopropyl alcohol in the concentrated composition obtained by the concentration process is preferably 1% or less by mass, more preferably 5,000 ppm or less by mass, further preferably 2,000 ppm or less by mass, and particularly 1,000 ppm or less by mass.

[0051]

The isopropyl alcohol aqueous solution evaporated in the concentration process may be recovered. The recovered isopropyl alcohol aqueous solution may be repeatedly used in the polymerization process. When the isopropyl alcohol aqueous solution is reused, evaporated isopropyl alcohol need not be disposed of. Therefore, it is possible to provide a dispersant for calcium carbonate that can reduce cost, and achieve excellent production efficiency and environmental protection, and a method for producing a dispersant for calcium carbonate.

In the case of recovering an isopropyl alcohol aqueous solution, the concentration of

isopropyl alcohol after recovery may differ from the one before recovery. Specifically, since isopropyl alcohol is evaporated off as an azeotrope of isopropyl alcohol and water, and then recovered, the concentration of isopropyl alcohol in the isopropyl alcohol aqueous solution recovered becomes normally about 60% or lower by mass, even if a high-concentration isopropyl alcohol aqueous solution is used in the polymerization process.

In the polymerization process, it is preferable to use the isopropyl alcohol aqueous solution having a constant isopropyl alcohol concentration in order to improve the production efficiency, even when the isopropyl alcohol aqueous solution is reused. Specifically, when the polymerization process is performed in a state in which the concentration of isopropyl alcohol in the isopropyl alcohol aqueous solution is 60% or higher by mass, the isopropyl alcohol concentration in the recovered isopropyl alcohol aqueous solution may be reduced due to evaporation. In this case, it is necessary to concentrate the recovered isopropyl alcohol aqueous solution so that the isopropyl alcohol aqueous solution has the original isopropyl alcohol concentration. On the other hand, the isopropyl alcohol concentration in the recovered isopropyl alcohol aqueous solution depends on the isopropyl alcohol concentration in the isopropyl alcohol aqueous solution to be used and may be higher than the one before recovery. In the case where the isopropyl alcohol concentration in the recovered isopropyl alcohol aqueous solution is higher than the one before recovery, the isopropyl alcohol concentration may be adjusted by adding water. However, when the recovered isopropyl alcohol aqueous solution is subjected to concentration so that the isopropyl alcohol aqueous solution has the original isopropyl alcohol concentration, it is necessary to provide concentration equipment, so that the production efficiency decreases.

Regarding the concentration of isopropyl alcohol in the isopropyl alcohol aqueous solution to be used, when the concentration of isopropyl alcohol is lower than that in the isopropyl alcohol aqueous solution by the concentration process, the resulting isopropyl alcohol aqueous solution can be reused. The isopropyl alcohol concentration when recovering or reusing the isopropyl alcohol aqueous solution is preferably in the range from 15% to 55% by mass, more preferably from 20% to 50% by mass, and particularly from 30% to 50% by mass. It is noted that the recovered isopropyl alcohol aqueous solution is preferably reused in the polymerization process after adjusting the isopropyl alcohol concentration.

[0052]

When the concentrated composition obtained by the concentration process is subjected to the neutralization process to neutralize some of carboxyl groups included in the first polymer, an acryl-based polymer can be obtained which is to be included in the dispersant.

[0053]

The dispersant of the present invention includes the acryl-based polymer obtained by a method having the polymerization process, neutralization process and the like. The dispersant may be a solution in which the acryl-based polymer is dissolved in an aqueous medium, or may be a dispersion in which the acryl-based polymer is dispersed in an aqueous medium. The aqueous medium may contain water, and be only water or a mixture containing water.

[0054]

The dispersant of the present invention may be (a) a reaction liquid based on a reaction production obtained by the polymerization process and neutralization process, or (b) a reaction liquid based on a reaction production obtained by the polymerization process, concentration process and neutralization process. Among these, the reaction liquid (b) is preferable for the dispersant since the dispersant can be efficiently obtained. These dispersants may further include an aqueous medium.

[0055]

The reaction liquid (b) is normally an aqueous solution in which the acryl-based polymer is dissolved in an aqueous medium, and may include the phosphite used in the polymerization process and/or by-products produced from the phosphite, the persulfate used in the polymerization process and/or by-products produced from the persulfate, and the basic compound used in the neutralization process. The reaction liquid (b) may also include isopropyl alcohol in an amount corresponding to the amount after the concentration process.

A content of isopropyl alcohol in the reaction liquid (b) may be 1% or less by mass, preferably 5,000 ppm or less by mass, more preferably 2,000 ppm or less by mass, and further preferably 1,000 ppm or less by mass.

[0056]

When the dispersant of the present invention is the reaction mixture (b), pH and solid content of the dispersant are not particularly limited. The pH of the dispersant is preferably in the range from 3 to 8, and more preferably from 3 to 7. The solid content in the dispersant is preferably in the range from 35% to 55%, and more preferably 40% to 50%.

[0057]

The dispersant of the present invention may further include an additional component such as antifoaming agent and preservative.

Examples of the antifoaming agent include a polyether compound, a mineral oil compound, a silicone compound, an amide compound, and the like. When the dispersant of the present invention includes the antifoaming agent, the content thereof is preferably in the range from 0.01% to 1.0% by mass based on 100 parts by mass of the acryl-based polymer.

[0058]

Examples of the preservative include an isothiazoline compound, a paraben, and the like. When the dispersant of the present invention includes the preservative, the content thereof is preferably in the range from 0.001% to 1.0% by mass based on 100 parts by mass of the acryl-based polymer.

[0059]

The dispersant of the present invention may be a solution or a dispersion in which the acryl-based polymer, and an optional additional component are included in an aqueous medium.

In the case where the dispersant of the present invention contains the additional component, the additional component is added and mixed with the aqueous medium and the acryl-based polymer. The mixing method is not particularly limited and may be a publicly known method.

[0060]

In the case of preparing a calcium carbonate slurry using the dispersant of the present invention, the dispersant is preferably used so that the content of the acryl-based polymer is in the range from 0.1 to 10.0 parts by mass, and the content of the aqueous medium is in the range from 25 to 100 parts by mass, based on 100 parts by mass of calcium carbonate.

After that, the dispersant mixture containing calcium carbonate and the acryl-based polymer is subjected to wet grinding by a known method to produce a calcium carbonate slurry.

[0061]

The dispersant of the present invention is excellent in dispersibility of calcium carbonate, and is suitably used as a dispersant for wet grinding of calcium carbonate to obtain a calcium carbonate slurry. Use of the dispersant for calcium carbonate of the present invention leads to a calcium carbonate slurry that has low initial viscosity, that does not show a significant increase in viscosity with passage of time, and exhibits excellent long-term dispersion stability.

[0062]

In the case where the dispersant of the present invention contains the additional component, the acryl-based polymer can be mixed with the additional component that is optionally added. When the dispersant of the present invention is an aqueous solution or a dispersion that is based on an aqueous medium containing the acryl-based polymer, the additional component may be mixed into the aqueous solution or the dispersion. The mixing method is not particularly limited and may be a publicly known method.

[EXAMPLES]

[0063]

Hereinafter, although Examples of the present invention will be described along with

synthesis examples and Comparative Examples, the scope of the invention is obviously not limited to these examples. Note that in the following examples, “part” and “%” are mass standard unless otherwise designated.

The term “Mw” used in Synthesis Examples, Examples, and Comparative Examples refers to a weight average molecular weight. The Mw was measured by gel permeation chromatography (GPC). “HLC8020 system” (manufactured by Tosoh Corporation), and columns “G4000PW” (×1), “G3000PW” (×1), “G2500PW” (×1) (manufactured by Tosoh Corporation) were used for GPC. A 0.1M NaCl + phosphate buffer (pH7) was used as an eluent, and a calibration curve was drawn using sodium polyacrylate (manufactured by Sowa Scientific Corporation).

[0064]

A viscosity of the ground calcium carbonate slurry after wet grinding, and a viscosity of the slurry after being allowed to stand at 25°C for 7 days were measured at a temperature of 25°C and a rotational speed of 60 rpm using Brookfield viscometer.

A median size of the ground calcium carbonate slurry after wet grinding, and an integrated value of calcium carbonate with a diameter smaller than 1.32 μm were measured using a laser diffraction/scattering particle size distribution analyzer “LA-910” manufactured by Horiba Ltd.

[0065]

<Preparation of dispersant for calcium carbonate and ground calcium carbonate slurry>

Example 1-1 (Preparation of dispersant 1-1, and preparation of slurry using dispersant 1-1)

A flask equipped with a stirrer and a condenser was charged with 500 g of an aqueous solution of an isopropyl alcohol (hereinafter, referred to also as “IPA” simply) at a concentration of 36%, and kept at a temperature of 80°C. A mixture prepared by mixing 600 g of acrylic acid (hereinafter, referred to as “AA”), 20 g of sodium hypophosphite, and 270 g of an aqueous solution of an IPA at a concentration of 36%, and 40 g of an aqueous solution of a sodium persulfate at a concentration of 15% were added dropwise to the flask over 4 hours to effect a reaction (polymerization). After the addition, the reaction liquid was kept at 80°C for an hour to continue a reaction (polymerization). Subsequently, IPA was evaporated off from the reaction liquid under reduced pressure while adding deionized water until a concentration of IPA became not more than 1,000 ppm by mass. The reaction liquid from which IPA was evaporated off was kept at 80°C, and an aqueous solution of a sodium hydroxide at a concentration of 32% was added to the reaction liquid so that a neutralization rate was 22 mol%. A dispersant 1-1 containing an acryl-based polymer 1-1 and having a solid content of 40% and

pH of 4 was thus obtained. Mw of the acryl-based polymer 1-1 contained in the dispersant 1-1 was 6,000. The amount of each raw material used in the preparation of the dispersant, the concentration of each raw material, a ratio of each raw material based on 100 parts of the monomer, and so on are shown in Table 1.

[0066]

17 g of the dispersant 1-1 obtained by the above operation, 320 g of ion-exchanged water, and 900 g of a ground calcium carbonate ("Tankaru A" produced by Maruo Calcium Co., Ltd.) were introduced into a cylindrical vessel and lightly mixed to be uniformly adapted. Subsequently, 3,000 g of a medium (ceramic beads of 1 mm ϕ) was introduced into the cylindrical vessel and mixed for 50 minutes at a rotational speed of 1,000 rpm to be subjected the ground calcium carbonate to wet grinding. After that, the mixture was subjected to filtration using a filter cloth having 200 ME and recovering to obtain a slurry. An ion-exchanged water was added to the slurry to adjust the solid content to 75% and a ground calcium carbonate slurry was obtained. Then, the viscosity of the slurry immediately after wet grinding and the viscosity of the slurry after being left standing for 7 days at a temperature of 25°C were measured in the above-described method. The slurry viscosity of the day of wet grinding was 210 mPa·s, and the slurry viscosity after 7 days was 1,800 mPa·s.

A median size of the slurry immediately after wet grinding and an integrated value of calcium carbonate with a diameter smaller than 1.32 μ m were measured in the above-described methods. The median size was 0.67 μ m, and the integrated value with a diameter smaller than 1.32 μ m was 100%. Those results are shown in Table 1.

In the following Examples and Comparative Examples, the amount of each raw material used in the preparation of the dispersant, the concentration of each raw material, a ratio of each raw material based on 100 parts of the monomer, and so on are shown in Tables 1 to 6. Further, in the following Examples and Comparative Examples, Mw of an acryl-based polymer contained in a dispersant, the viscosity of a ground calcium carbonate slurry after wet grinding, the viscosity of a slurry after left standing for seven days at 25°C, the median size of the ground calcium carbonate slurry after wet grinding, and the integrated value of calcium carbonate with a diameter smaller than 1.32 μ m in the slurry after wet grinding were measured in the same manner as those in Example 1-1, and the measured results were shown in Tables 1 to 6.

[0067]

Example 1-2 (Preparation of dispersant 1-2, and preparation of slurry using dispersant 1-2)

In this Example 1-2, a dispersant 1-2 was prepared using the aqueous solution of IPA distilled off and recovered in the preparation of the dispersant 1-1 in Example 1-1.

When the IPA concentration of the aqueous solution of IPA recovered in Example 1-1 was measured, the IPA concentration was 40%. Deionized water was added to set the same IPA concentration of an IPA aqueous solution as the IPA concentration of the IPA aqueous solution used in Example 1-1, and the IPA aqueous solution was obtained in the form of an aqueous solution of IPA at a concentration of 36%. Subsequently, the concentration-adjusted IPA aqueous solution was used and the same operations including polymerization, reduced-pressure evaporation, and neutralization were conducted as those in Example 1-1 to obtain a dispersant 1-2 which contains an acryl-based polymer 1-2 and has a solid content of 40% and pH of 4.

After that, a ground calcium carbonate slurry was produced using the resulting dispersant 1-2 in the same manner as those in Example 1-1.

[0068]

Example 2-1 (Preparation of dispersant 2-1, and preparation of slurry using dispersant 2-1)

The polymerization, reduced-pressure evaporation, and neutralization were conducted in the same manner as those in Example 1-1, except that an aqueous solution of IPA at a concentration of 48% was used instead of the IPA aqueous solution of 36% in Example 1-1 to obtain a dispersant 2-1 which contains an acryl-based polymer 2-1 and has a solid content of 40% and pH of 4.

After that, a ground calcium carbonate slurry was produced using the resulting dispersant 2-1 in the same manner as those in Example 1-1.

[0069]

Example 2-2 (Preparation of dispersant 2-2, and preparation of slurry using dispersant 2-2)

In this Example 2-2, a dispersant 2-2 was prepared using the aqueous solution of IPA distilled off and recovered in the preparation of the dispersant 2-1 in Example 2-1. When the IPA concentration of the aqueous solution of IPA recovered in Example 2-1 was measured, the IPA concentration was 48%. Since the IPA concentration in the recovered IPA aqueous solution was the same as the IPA concentration in the IPA aqueous solution used in Example 2-1, the adjustment of IPA concentration was not performed. Subsequently, the recovered IPA aqueous solution was used and the same operations including polymerization, reduced-pressure evaporation, and neutralization were conducted as those in Example 2-1 to obtain a dispersant 2-2 which contains an acryl-based polymer 2-2 and has a solid content of 40% and pH of 4.

After that, a ground calcium carbonate slurry was produced using the resulting dispersant 2-2 in the same manner as those in Example 2-1.

[0070]

Example 3-1 (Preparation of dispersant 3-1, and preparation of slurry using dispersant 3-1)

The polymerization, reduced-pressure evaporation, and neutralization were conducted in the same manner as those in Example 1-1, except that the usage amount of the sodium hypophosphite was set to 17 g instead of 20g in Example 1-1 to obtain a dispersant 3-1 which contains an acryl-based polymer 3-1 and has a solid content of 40% and pH of 4.

After that, a ground calcium carbonate slurry was produced using the resulting dispersant 3-1 in the same manner as those in Example 1-1.

[0071]

Example 3-2 (Preparation of dispersant 3-2, and preparation of slurry using dispersant 3-2)

In this Example 3-2, a dispersant 3-2 was prepared using the aqueous solution of IPA distilled off and recovered in the preparation of the dispersant 3-1 in Example 3-1.

When the IPA concentration of the aqueous solution of IPA recovered in Example 3-1 was measured, the IPA concentration was 40%. Deionized water was added to set the same IPA concentration of an IPA aqueous solution as the IPA concentration of the IPA aqueous solution used in Example 3-1, and the IPA aqueous solution was obtained in the form of an aqueous solution of IPA at a concentration of 36%. Subsequently, the concentration-adjusted IPA aqueous solution was used and the same operations including polymerization, reduced-pressure evaporation, and neutralization were conducted as those in Example 3-1 to obtain a dispersant 3-2 which contains an acryl-based polymer 3-2 and has a solid content of 40% and pH of 4.

After that, a ground calcium carbonate slurry was produced using the resulting dispersant 3-2 in the same manner as those in Example 3-1.

[0072]

Example 4-1 (Preparation of dispersant 4-1, and preparation of slurry using dispersant 4-1)

The polymerization, reduced-pressure evaporation, and neutralization were conducted in the same manner as those in Example 1-1, except that the usage amount of the sodium persulfate aqueous solution was set to 30 g instead of 40g in Example 1-1 to obtain a dispersant 4-1 which contains an acryl-based polymer 4-1 and has a solid content of 40% and pH of 4.

After that, a ground calcium carbonate slurry was produced using the resulting dispersant 4-1 in the same manner as those in Example 1-1.

[0073]

Example 4-2 (Preparation of dispersant 4-2, and preparation of slurry using dispersant 4-2)

In this Example 4-2, a dispersant 4-2 was prepared using the aqueous solution of IPA distilled off and recovered in the preparation of the dispersant 4-1 in Example 4-1.

When the IPA concentration of the aqueous solution of IPA recovered in Example 4-1 was measured, the IPA concentration was 40%. Deionized water was added to set the same IPA concentration of an IPA aqueous solution as the IPA concentration of the IPA aqueous solution used in Example 4-1, and the IPA aqueous solution was obtained in the form of an aqueous solution of IPA at a concentration of 36%. Subsequently, the concentration-adjusted IPA aqueous solution was used and the same operations including polymerization, reduced-pressure evaporation, and neutralization were conducted as those in Example 4-1 to obtain a dispersant 4-2 which contains an acryl-based polymer 4-2 and has a solid content of 40% and pH of 4.

After that, a ground calcium carbonate slurry was produced using the resulting dispersant 4-2 in the same manner as those in Example 4-1.

[0074]

Example 5-1 (Preparation of dispersant 5-1, and preparation of slurry using dispersant 5-1)

The polymerization, reduced-pressure evaporation, and neutralization were conducted in the same manner as those in Example 1-1, except that the keeping temperature of IPA in the flask, reaction temperature for polymerization, and temperature for neutralization were all set to 75°C instead of 80°C in Example 1-1 to obtain a dispersant 5-1 which contains an acryl-based polymer 5-1 and has a solid content of 40% and pH of 4.

After that, a ground calcium carbonate slurry was produced using the resulting dispersant 5-1 in the same manner as those in Example 1-1.

[0075]

Example 5-2 (Preparation of dispersant 5-2, and preparation of slurry using dispersant 5-2)

In this Example 5-2, a dispersant 5-2 was prepared using the aqueous solution of IPA distilled off and recovered in the preparation of the dispersant 5-1 in Example 5-1.

When the IPA concentration of the aqueous solution of IPA recovered in Example 5-1 was measured, the IPA concentration was 40%. Deionized water was added to set the same IPA concentration of an IPA aqueous solution as the IPA concentration of the IPA aqueous solution used in Example 5-1, and the IPA aqueous solution was obtained in the form of an aqueous solution of IPA at a concentration of 36%. Subsequently, the concentration-adjusted

IPA aqueous solution was used and the same operations including polymerization, reduced-pressure evaporation, and neutralization were conducted as those in Example 5-1 to obtain a dispersant 5-2 which contains an acryl-based polymer 5-2 and has a solid content of 40% and pH of 4.

After that, a ground calcium carbonate slurry was produced using the resulting dispersant 5-2 in the same manner as those in Example 5-1.

[0076]

Example 6-1 (Preparation of dispersant 6-1, and preparation of slurry using dispersant 6-1)

The polymerization, reduced-pressure evaporation, and neutralization were conducted in the same manner as those in Example 1-1, except that the neutralization rate using the 32% sodium hydroxide aqueous solution was set to 40 mol% instead of 22 mol% in Example 1-1 to obtain a dispersant 6-1 which contains an acryl-based polymer 6-1 and has a solid content of 40% and pH of 5.

After that, a ground calcium carbonate slurry was produced using the resulting dispersant 6-1 in the same manner as those in Example 1-1.

[0077]

Example 6-2 (Preparation of dispersant 6-2, and preparation of slurry using dispersant 6-2)

In this Example 6-2, a dispersant 6-2 was prepared using the aqueous solution of IPA distilled off and recovered in the preparation of the dispersant 6-1 in Example 6-1.

When the IPA concentration of the aqueous solution of IPA recovered in Example 6-1 was measured, the IPA concentration was 40%. Deionized water was added to set the same IPA concentration of an IPA aqueous solution as the IPA concentration of the IPA aqueous solution used in Example 6-1, and the IPA aqueous solution was obtained in the form of an aqueous solution of IPA at a concentration of 36%. Subsequently, the concentration-adjusted IPA aqueous solution was used and the same operations including polymerization, reduced-pressure evaporation, and neutralization were conducted as those in Example 6-1 to obtain a dispersant 6-2 which contains an acryl-based polymer 6-2 and has a solid content of 40% and pH of 5.

After that, a ground calcium carbonate slurry was produced using the resulting dispersant 6-2 in the same manner as those in Example 6-1.

[0078]

Example 7-1 (Preparation of dispersant 7-1, and preparation of slurry using dispersant 7-1)

The polymerization, reduced-pressure evaporation, and neutralization were conducted in the same manner as those in Example 1-1, except that an aqueous solution of IPA at a concentration of 20% was used instead of the IPA aqueous solution of 36% in Example 1-1 to obtain a dispersant 7-1 which contains an acryl-based polymer 7-1 and has a solid content of 40% and pH of 4.

After that, a ground calcium carbonate slurry was produced using the resulting dispersant 7-1 in the same manner as those in Example 1-1.

[0079]

Example 7-2 (Preparation of dispersant 7-2, and preparation of slurry using dispersant 7-2)

In this Example 7-2, a dispersant 7-2 was prepared using the aqueous solution of IPA distilled off and recovered in the preparation of the dispersant 7-1 in Example 7-1.

When the IPA concentration of the aqueous solution of IPA recovered in Example 7-1 was measured, the IPA concentration was 25%. Deionized water was added to set the same IPA concentration of an IPA aqueous solution as the IPA concentration of the IPA aqueous solution used in Example 7-1, and the IPA aqueous solution was obtained in the form of an aqueous solution of IPA at a concentration of 20%. Subsequently, the concentration-adjusted IPA aqueous solution was used and the same operations including polymerization, reduced-pressure evaporation, and neutralization were conducted as those in Example 7-1 to obtain a dispersant 7-2 which contains an acryl-based polymer 7-2 and has a solid content of 40% and pH of 4.

After that, a ground calcium carbonate slurry was produced using the resulting dispersant 7-2 in the same manner as those in Example 7-1.

[0080]

Example 8-1 (Preparation of dispersant 8-1, and preparation of slurry using dispersant 8-1)

The polymerization, reduced-pressure evaporation, and neutralization were conducted in the same manner as those in Example 1-1, except that an aqueous solution of IPA at a concentration of 53% was used instead of the IPA aqueous solution of 36% in Example 1-1 to obtain a dispersant 8-1 which contains an acryl-based polymer 8-1 and has a solid content of 40% and pH of 4.

After that, a ground calcium carbonate slurry was produced using the resulting dispersant 8-1 in the same manner as those in Example 1-1.

[0081]

Example 8-2 (Preparation of dispersant 8-2, and preparation of slurry using dispersant

8-2)

In this Example 8-2, a dispersant 8-2 was prepared using the aqueous solution of IPA distilled off and recovered in the preparation of the dispersant 8-1 in Example 8-1.

When the IPA concentration of the aqueous solution of IPA recovered in Example 8-1 was measured, the IPA concentration was 53%. Since the IPA concentration in the recovered IPA aqueous solution was the same as the IPA concentration in the IPA aqueous solution used in Example 8-1, the adjustment of IPA concentration was not performed. Subsequently, the recovered IPA aqueous solution was used and the same operations including polymerization, reduced-pressure evaporation, and neutralization were conducted as those in Example 8-1 to obtain a dispersant 8-2 which contains an acryl-based polymer 8-2 and has a solid content of 40% and pH of 4.

After that, a ground calcium carbonate slurry was produced using the resulting dispersant 8-2 in the same manner as those in Example 8-1.

[0082]

Example 9-1 (Preparation of dispersant 9-1, and preparation of slurry using dispersant 9-1)

The polymerization, reduced-pressure evaporation, and neutralization were conducted in the same manner as those in Example 1-1, except that the usage amount of the sodium hypophosphite was set to 9.5 g instead of 20g in Example 1-1 to obtain a dispersant 9-1 which contains an acryl-based polymer 9-1 and has a solid content of 40% and pH of 4.

After that, a ground calcium carbonate slurry was produced using the resulting dispersant 9-1 in the same manner as those in Example 1-1.

[0083]

Example 9-2 (Preparation of dispersant 9-2, and preparation of slurry using dispersant 9-2)

In this Example 9-2, a dispersant 9-2 was prepared using the aqueous solution of IPA distilled off and recovered in the preparation of the dispersant 9-1 in Example 9-1.

When the IPA concentration of the aqueous solution of IPA recovered in Example 9-1 was measured, the IPA concentration was 40%. Deionized water was added to set the same IPA concentration of an IPA aqueous solution as the IPA concentration of the IPA aqueous solution used in Example 9-1, and the IPA aqueous solution was obtained in the form of an aqueous solution of IPA at a concentration of 36%. Subsequently, the concentration-adjusted IPA aqueous solution was used and the same operations including polymerization, reduced-pressure evaporation, and neutralization were conducted as those in Example 9-1 to obtain a dispersant 9-2 which contains an acryl-based polymer 9-2 and has a solid content of

40% and pH of 4.

After that, a ground calcium carbonate slurry was produced using the resulting dispersant 9-2 in the same manner as those in Example 9-1.

[0084]

Example 10-1 (Preparation of dispersant 10-1, and preparation of slurry using dispersant 10-1)

The polymerization, reduced-pressure evaporation, and neutralization were conducted in the same manner as those in Example 1-1, except that the usage amount of the sodium hypophosphite was set to 26.5 g instead of 20g in Example 1-1 to obtain a dispersant 10-1 which contains an acryl-based polymer 10-1 and has a solid content of 40% and pH of 4.

After that, a ground calcium carbonate slurry was produced using the resulting dispersant 10-1 in the same manner as those in Example 1-1.

[0085]

Example 10-2 (Preparation of dispersant 10-2, and preparation of slurry using dispersant 10-2)

In this Example 10-2, a dispersant 10-2 was prepared using the aqueous solution of IPA distilled off and recovered in the preparation of the dispersant 10-1 in Example 10-1.

When the IPA concentration of the aqueous solution of IPA recovered in Example 10-1 was measured, the IPA concentration was 40%. Deionized water was added to set the same IPA concentration of an IPA aqueous solution as the IPA concentration of the IPA aqueous solution used in Example 10-1, and the IPA aqueous solution was obtained in the form of an aqueous solution of IPA at a concentration of 36%. Subsequently, the concentration-adjusted IPA aqueous solution was used and the same operations including polymerization, reduced-pressure evaporation, and neutralization were conducted as those in Example 10-1 to obtain a dispersant 10-2 which contains an acryl-based polymer 10-2 and has a solid content of 40% and pH of 4.

After that, a ground calcium carbonate slurry was produced using the resulting dispersant 10-2 in the same manner as those in Example 10-1.

[0086]

Example 11-1 (Preparation of dispersant 11-1, and preparation of slurry using dispersant 11-1)

The polymerization, reduced-pressure evaporation, and neutralization were conducted in the same manner as those in Example 1-1, except that the usage amount of the sodium persulfate aqueous solution was set to 12 g instead of 40g in Example 1-1 to obtain a dispersant 11-1 which contains an acryl-based polymer 4-1 and has a solid content of 40% and pH of 4.

After that, a ground calcium carbonate slurry was produced using the resulting dispersant 11-1 in the same manner as those in Example 1-1.

[0087]

Example 11-2 (Preparation of dispersant 11-2, and preparation of slurry using dispersant 11-2)

In this Example 11-2, a dispersant 11-2 was prepared using the aqueous solution of IPA distilled off and recovered in the preparation of the dispersant 11-1 in Example 11-1.

When the IPA concentration of the aqueous solution of IPA recovered in Example 11-1 was measured, the IPA concentration was 40%. Deionized water was added to set the same IPA concentration of an IPA aqueous solution as the IPA concentration of the IPA aqueous solution used in Example 11-1, and the IPA aqueous solution was obtained in the form of an aqueous solution of IPA at a concentration of 36%. Subsequently, the concentration-adjusted IPA aqueous solution was used and the same operations including polymerization, reduced-pressure evaporation, and neutralization were conducted as those in Example 11-1 to obtain a dispersant 11-2 which contains an acryl-based polymer 11-2 and has a solid content of 40% and pH of 4.

After that, a ground calcium carbonate slurry was produced using the resulting dispersant 11-2 in the same manner as those in Example 11-1.

[0088]

Example 12-1 (Preparation of dispersant 12-1, and preparation of slurry using dispersant 12-1)

The polymerization, reduced-pressure evaporation, and neutralization were conducted in the same manner as those in Example 1-1, except that the usage amount of the sodium persulfate aqueous solution was set to 60 g instead of 40g in Example 1-1 to obtain a dispersant 12-1 which contains an acryl-based polymer 12-1 and has a solid content of 40% and pH of 4.

After that, a ground calcium carbonate slurry was produced using the resulting dispersant 12-1 in the same manner as those in Example 1-1.

[0089]

Example 12-2 (Preparation of dispersant 12-2, and preparation of slurry using dispersant 12-2)

In this Example 12-2, a dispersant 12-2 was prepared using the aqueous solution of IPA distilled off and recovered in the preparation of the dispersant 12-1 in Example 12-1.

When the IPA concentration of the aqueous solution of IPA recovered in Example 12-1 was measured, the IPA concentration was 40%. Deionized water was added to set the same IPA concentration of an IPA aqueous solution as the IPA concentration of the IPA aqueous

solution used in Example 12-1, and the IPA aqueous solution was obtained in the form of an aqueous solution of IPA at a concentration of 36%. Subsequently, the concentration-adjusted IPA aqueous solution was used and the same operations including polymerization, reduced-pressure evaporation, and neutralization were conducted as those in Example 12-1 to obtain a dispersant 12-2 which contains an acryl-based polymer 12-2 and has a solid content of 40% and pH of 4.

After that, a ground calcium carbonate slurry was produced using the resulting dispersant 12-2 in the same manner as those in Example 12-1.

[0090]

Example 13-1 (Preparation of dispersant 13-1, and preparation of slurry using dispersant 13-1)

The polymerization, reduced-pressure evaporation, and neutralization were conducted in the same manner as those in Example 1-1, except that the neutralization rate using the 32% sodium hydroxide aqueous solution was set to 18 mol% instead of 22 mol% in Example 1-1 to obtain a dispersant 13-1 which contains an acryl-based polymer 13-1 and has a solid content of 40% and pH of 3.

After that, a ground calcium carbonate slurry was produced using the resulting dispersant 13-1 in the same manner as those in Example 1-1.

[0091]

Example 13-2 (Preparation of dispersant 13-2, and preparation of slurry using dispersant 13-2)

In this Example 13-2, a dispersant 13-2 was prepared using the aqueous solution of IPA distilled off and recovered in the preparation of the dispersant 13-1 in Example 13-1.

When the IPA concentration of the aqueous solution of IPA recovered in Example 13-1 was measured, the IPA concentration was 40%. Deionized water was added to set the same IPA concentration of an IPA aqueous solution as the IPA concentration of the IPA aqueous solution used in Example 13-1, and the IPA aqueous solution was obtained in the form of an aqueous solution of IPA at a concentration of 36%. Subsequently, the concentration-adjusted IPA aqueous solution was used and the same operations including polymerization, reduced-pressure evaporation, and neutralization were conducted as those in Example 13-1 to obtain a dispersant 13-2 which contains an acryl-based polymer 13-2 and has a solid content of 40% and pH of 3.

After that, a ground calcium carbonate slurry was produced using the resulting dispersant 13-2 in the same manner as those in Example 13-1.

[0092]

Example 14-1 (Preparation of dispersant 14-1, and preparation of slurry using dispersant 14-1)

The polymerization, reduced-pressure evaporation, and neutralization were conducted in the same manner as those in Example 1-1, except that the neutralization rate using the 32% sodium hydroxide aqueous solution was set to 90 mol% instead of 22 mol% in Example 1-1 to obtain a dispersant 14-1 which contains an acryl-based polymer 14-1 and has a solid content of 40% and pH of 7.

After that, a ground calcium carbonate slurry was produced using the resulting dispersant 14-1 in the same manner as those in Example 1-1.

[0093]

Example 14-2 (Preparation of dispersant 14-2, and preparation of slurry using dispersant 14-2)

In this Example 14-2, a dispersant 14-2 was prepared using the aqueous solution of IPA distilled off and recovered in the preparation of the dispersant 14-1 in Example 14-1.

When the IPA concentration of the aqueous solution of IPA recovered in Example 14-1 was measured, the IPA concentration was 40%. Deionized water was added to set the same IPA concentration of an IPA aqueous solution as the IPA concentration of the IPA aqueous solution used in Example 14-1, and the IPA aqueous solution was obtained in the form of an aqueous solution of IPA at a concentration of 36%. Subsequently, the concentration-adjusted IPA aqueous solution was used and the same operations including polymerization, reduced-pressure evaporation, and neutralization were conducted as those in Example 14-1 to obtain a dispersant 14-2 which contains an acryl-based polymer 14-2 and has a solid content of 40% and pH of 7.

After that, a ground calcium carbonate slurry was produced using the resulting dispersant 14-2 in the same manner as those in Example 14-1.

[0094]

Example 15-1 (Preparation of dispersant 15-1, and preparation of slurry using dispersant 15-1)

The polymerization, reduced-pressure evaporation, and neutralization were conducted in the same manner as those in Example 1-1, except that an aqueous solution of IPA at a concentration of 65% was used instead of the IPA aqueous solution of 36% in Example 1-1 and that the usage amount of the sodium hypophosphite was set to 15 g instead of 20g in Example 1-1 to obtain a dispersant 15-1 which contains an acryl-based polymer 15-1 and has a solid content of 40% and pH of 4.

After that, a ground calcium carbonate slurry was produced using the resulting

dispersant 15-1 in the same manner as those in Example 1-1.

[0095]

Example 15-2 (Preparation of dispersant 15-2, and preparation of slurry using dispersant 15-2)

In this Example 15-2, a dispersant 15-2 was prepared using the aqueous solution of IPA distilled off and recovered in the preparation of the dispersant 15-1 in Example 15-1.

When the IPA concentration of the aqueous solution of IPA recovered in Example 15-1 was measured, the IPA concentration was 55%. Concentration was conducted to set the same IPA concentration of an IPA aqueous solution as the IPA concentration of the IPA aqueous solution used in Example 15-1, and the IPA aqueous solution was obtained in the form of an aqueous solution of IPA at a concentration of 65%. Subsequently, the concentration-adjusted IPA aqueous solution was used and the same operations including polymerization, reduced-pressure evaporation, and neutralization were conducted as those in Example 15-1 to obtain a dispersant 15-2 which contains an acryl-based polymer 15-2 and has a solid content of 40% and pH of 4.

After that, a ground calcium carbonate slurry was produced using the resulting dispersant 15-2 in the same manner as those in Example 15-1.

[0096]

Example 16-1 (Preparation of dispersant 16-1, and preparation of slurry using dispersant 16-1)

The polymerization, reduced-pressure evaporation, and neutralization were conducted in the same manner as those in Example 1-1, except that methyl acrylate was used in addition to acrylic acid and 575 g of acrylic acid and 25 g of methyl acrylate were used instead of 600 g of acrylic acid in Example 1-1 to obtain a dispersant 16-1 which contains an acryl-based polymer 16-1 and has a solid content of 40% and pH of 4.

After that, a ground calcium carbonate slurry was produced using the resulting dispersant 16-1 in the same manner as those in Example 1-1.

[0097]

Example 16-2 (Preparation of dispersant 16-2, and preparation of slurry using dispersant 16-2)

In this Example 16-2, a dispersant 16-2 was prepared using the aqueous solution of IPA distilled off and recovered in the preparation of the dispersant 16-1 in Example 16-1.

When the IPA concentration of the aqueous solution of IPA recovered in Example 16-1 was measured, the IPA concentration was 40%. Deionized water was added to set the same IPA concentration of an IPA aqueous solution as the IPA concentration of the IPA aqueous

solution used in Example 16-1, and the IPA aqueous solution was obtained in the form of an aqueous solution of IPA at a concentration of 36%. Subsequently, the concentration-adjusted IPA aqueous solution was used and the same operations including polymerization, reduced-pressure evaporation, and neutralization were conducted as those in Example 16-1 to obtain a dispersant 16-2 which contains an acryl-based polymer 16-2 and has a solid content of 40% and pH of 4.

After that, a ground calcium carbonate slurry was produced using the resulting dispersant 16-2 in the same manner as those in Example 16-1.

[0098]

Comparative Example 1-1 (Preparation of dispersant 1-1C, and preparation of slurry using dispersant 1-1C)

The polymerization, reduced-pressure evaporation, and neutralization were conducted in the same manner as those in Example 1-1, except that an aqueous solution of IPA at a concentration of 10% was used instead of the IPA aqueous solution of 36% in Example 1-1 to obtain a dispersant 1-1C which contains an acryl-based polymer 1-1C and has a solid content of 40% and pH of 4.

After that, a ground calcium carbonate slurry was produced using the resulting dispersant 1-1C in the same manner as those in Example 1-1.

[0099]

Comparative Example 1-2 (Preparation of dispersant 1-2C, and preparation of slurry using dispersant 1-2C)

In this Comparative Example 1-2, a dispersant 1-2C was prepared using the aqueous solution of IPA distilled off and recovered in the preparation of the dispersant 1-1C in Comparative Example 1-1.

When the IPA concentration of the aqueous solution of IPA recovered in Comparative Example 1-1 was measured, the IPA concentration was 20%. Deionized water was added to set the same IPA concentration of an IPA aqueous solution as the IPA concentration of the IPA aqueous solution used in Comparative Example 1-1, and the IPA aqueous solution was obtained in the form of an aqueous solution of IPA at a concentration of 10%. Subsequently, the concentration-adjusted IPA aqueous solution was used and the same operations including polymerization, reduced-pressure evaporation, and neutralization were conducted as those in Comparative Example 1-1 to obtain a dispersant 1-2C which contains an acryl-based polymer 1-2C and has a solid content of 40% and pH of 4.

After that, a ground calcium carbonate slurry was produced using the resulting dispersant 1-2C in the same manner as those in Comparative Example 1-1.

[0100]

Comparative Example 2-1 (Preparation of dispersant 2-1C, and preparation of slurry using dispersant 2-1C)

The polymerization, reduced-pressure evaporation, and neutralization were conducted in the same manner as those in Example 1-1, except that an aqueous solution of IPA at a concentration of 65% was used instead of the IPA aqueous solution of 36% in Example 1-1 to obtain a dispersant 2-1C which contains an acryl-based polymer 2-1C and has a solid content of 40% and pH of 4.

After that, a ground calcium carbonate slurry was produced using the resulting dispersant 2-1C in the same manner as those in Example 1-1.

[0101]

Comparative Example 2-2 (Preparation of dispersant 2-2C, and preparation of slurry using dispersant 2-2C)

In this Comparative Example 2-2, a dispersant 2-2C was prepared using the aqueous solution of IPA distilled off and recovered in the preparation of the dispersant 2-1C in Comparative Example 2-1.

When the IPA concentration of the aqueous solution of IPA recovered in Comparative Example 2-1 was measured, the IPA concentration was 55%. Concentration was conducted to set the same IPA concentration of an IPA aqueous solution as the IPA concentration of the IPA aqueous solution used in Comparative Example 2-1, and the IPA aqueous solution was obtained in the form of an aqueous solution of IPA at a concentration of 65%. Subsequently, the concentration-adjusted IPA aqueous solution was used and the same operations including polymerization, reduced-pressure evaporation, and neutralization were conducted as those in Comparative Example 2-1 to obtain a dispersant 2-2C which contains an acryl-based polymer 2-2C and has a solid content of 40% and pH of 4.

After that, a ground calcium carbonate slurry was produced using the resulting dispersant 2-2C in the same manner as those in Comparative Example 2-1.

[0102]

Comparative Example 3-1 (Preparation of dispersant 3-1C, and preparation of slurry using dispersant 3-1C)

The polymerization, reduced-pressure evaporation, and neutralization were conducted in the same manner as those in Example 1-1, except that the usage amount of the sodium hypophosphite was set to 6 g instead of 20g in Example 1-1 to obtain a dispersant 3-1C which contains an acryl-based polymer 3-1C and has a solid content of 40% and pH of 4.

After that, a ground calcium carbonate slurry was produced using the resulting

dispersant 3-1C in the same manner as those in Example 1-1.

[0103]

Comparative Example 3-2 (Preparation of dispersant 3-2C, and preparation of slurry using dispersant 3-2C)

In this Comparative Example 3-2, a dispersant 3-2C was prepared using the aqueous solution of IPA distilled off and recovered in the preparation of the dispersant 3-1C in Comparative Example 3-1.

When the IPA concentration of the aqueous solution of IPA recovered in Comparative Example 3-1 was measured, the IPA concentration was 40%. Deionized water was added to set the same IPA concentration of an IPA aqueous solution as the IPA concentration of the IPA aqueous solution used in Comparative Example 3-1, and the IPA aqueous solution was obtained in the form of an aqueous solution of IPA at a concentration of 36%. Subsequently, the concentration-adjusted IPA aqueous solution was used and the same operations including polymerization, reduced-pressure evaporation, and neutralization were conducted as those in Comparative Example 3-1 to obtain a dispersant 3-2C which contains an acryl-based polymer 3-2C and has a solid content of 40% and pH of 4.

After that, a ground calcium carbonate slurry was produced using the resulting dispersant 3-2C in the same manner as those in Comparative Example 3-1.

[0104]

Comparative Example 4-1 (Preparation of dispersant 4-1C, and preparation of slurry using dispersant 4-1C)

The polymerization, reduced-pressure evaporation, and neutralization were conducted in the same manner as those in Example 1-1, except that the usage amount of the sodium hypophosphite was set to 33 g instead of 20g in Example 1-1 to obtain a dispersant 4-1C which contains an acryl-based polymer 4-1C and has a solid content of 40% and pH of 4.

After that, a ground calcium carbonate slurry was produced using the resulting dispersant 4-1C in the same manner as those in Example 1-1.

[0105]

Comparative Example 4-2 (Preparation of dispersant 4-2C, and preparation of slurry using dispersant 4-2C)

In this Comparative Example 4-2, a dispersant 4-2C was prepared using the aqueous solution of IPA distilled off and recovered in the preparation of the dispersant 4-1C in Comparative Example 4-1.

When the IPA concentration of the aqueous solution of IPA recovered in Comparative Example 4-1 was measured, the IPA concentration was 40%. Deionized water was added to

set the same IPA concentration of an IPA aqueous solution as the IPA concentration of the IPA aqueous solution used in Comparative Example 4-1, and the IPA aqueous solution was obtained in the form of an aqueous solution of IPA at a concentration of 36%. Subsequently, the concentration-adjusted IPA aqueous solution was used and the same operations including polymerization, reduced-pressure evaporation, and neutralization were conducted as those in Comparative Example 4-1 to obtain a dispersant 4-2C which contains an acryl-based polymer 4-2C and has a solid content of 40% and pH of 4.

After that, a ground calcium carbonate slurry was produced using the resulting dispersant 4-2C in the same manner as those in Comparative Example 4-1.

[0106]

Comparative Example 5-1 (Preparation of dispersant 5-1C, and preparation of slurry using dispersant 5-1C)

The polymerization, reduced-pressure evaporation, and neutralization were conducted in the same manner as those in Example 1-1, except that the usage amount of the sodium persulfate aqueous solution was set to 7 g instead of 40g in Example 1-1 to obtain a dispersant 5-1C which contains an acryl-based polymer 5-1C and has a solid content of 40% and pH of 4.

After that, a ground calcium carbonate slurry was produced using the resulting dispersant 5-1C in the same manner as those in Example 1-1.

[0107]

Comparative Example 5-2 (Preparation of dispersant 5-2C, and preparation of slurry using dispersant 5-2C)

In this Comparative Example 5-2, a dispersant 5-2C was prepared using the aqueous solution of IPA distilled off and recovered in the preparation of the dispersant 5-1C in Comparative Example 5-1.

When the IPA concentration of the aqueous solution of IPA recovered in Comparative Example 5-1 was measured, the IPA concentration was 40%. Deionized water was added to set the same IPA concentration of an IPA aqueous solution as the IPA concentration of the IPA aqueous solution used in Comparative Example 5-1, and the IPA aqueous solution was obtained in the form of an aqueous solution of IPA at a concentration of 36%. Subsequently, the concentration-adjusted IPA aqueous solution was used and the same operations including polymerization, reduced-pressure evaporation, and neutralization were conducted as those in Comparative Example 5-1 to obtain a dispersant 5-2C which contains an acryl-based polymer 5-2C and has a solid content of 40% and pH of 4.

After that, a ground calcium carbonate slurry was produced using the resulting dispersant 5-2C in the same manner as those in Comparative Example 5-1.

[0108]

Comparative Example 6-1 (Preparation of dispersant 6-1C, and preparation of slurry using dispersant 6-1C)

The polymerization, reduced-pressure evaporation, and neutralization were conducted in the same manner as those in Example 1-1, except that the usage amount of the sodium persulfate aqueous solution was set to 80 g instead of 40g in Example 1-1 to obtain a dispersant 6-1C which contains an acryl-based polymer 6-1C and has a solid content of 40% and pH of 4.

After that, a ground calcium carbonate slurry was produced using the resulting dispersant 6-1C in the same manner as those in Example 1-1.

[0109]

Comparative Example 6-2 (Preparation of dispersant 6-2C, and preparation of slurry using dispersant 6-2C)

In this Comparative Example 6-2, a dispersant 6-2C was prepared using the aqueous solution of IPA distilled off and recovered in the preparation of the dispersant 6-1C in Comparative Example 6-1.

When the IPA concentration of the aqueous solution of IPA recovered in Comparative Example 6-1 was measured, the IPA concentration was 40%. Deionized water was added to set the same IPA concentration of an IPA aqueous solution as the IPA concentration of the IPA aqueous solution used in Comparative Example 6-1, and the IPA aqueous solution was obtained in the form of an aqueous solution of IPA at a concentration of 36%. Subsequently, the concentration-adjusted IPA aqueous solution was used and the same operations including polymerization, reduced-pressure evaporation, and neutralization were conducted as those in Comparative Example 6-1 to obtain a dispersant 6-2C which contains an acryl-based polymer 6-2C and has a solid content of 40% and pH of 4.

After that, a ground calcium carbonate slurry was produced using the resulting dispersant 6-2C in the same manner as those in Comparative Example 6-1.

[0110]

Comparative Example 7-1 (Preparation of dispersant 7-1C, and preparation of slurry using dispersant 7-1C)

The polymerization, reduced-pressure evaporation, and neutralization were conducted in the same manner as those in Example 1-1, except that the keeping temperature of IPA in the flask, reaction temperature for polymerization, and temperature for neutralization were all set to 60°C instead of 80°C in Example 1-1 to obtain a dispersant 7-1C which contains an acryl-based polymer 7-1C and has a solid content of 40% and pH of 4.

After that, a ground calcium carbonate slurry was produced using the resulting

dispersant 7-1C in the same manner as those in Example 1-1.

[0111]

Comparative Example 7-2 (Preparation of dispersant 7-2C, and preparation of slurry using dispersant 7-2C)

In this Comparative Example 7-2, a dispersant 7-2C was prepared using the aqueous solution of IPA distilled off and recovered in the preparation of the dispersant 7-1C in Comparative Example 7-1.

When the IPA concentration of the aqueous solution of IPA recovered in Comparative Example 7-1 was measured, the IPA concentration was 40%. Deionized water was added to set the same IPA concentration of an IPA aqueous solution as the IPA concentration of the IPA aqueous solution used in Comparative Example 7-1, and the IPA aqueous solution was obtained in the form of an aqueous solution of IPA at a concentration of 36%. Subsequently, the concentration-adjusted IPA aqueous solution was used and the same operations including polymerization, reduced-pressure evaporation, and neutralization were conducted as those in Comparative Example 7-1 to obtain a dispersant 7-2C which contains an acryl-based polymer 7-2C and has a solid content of 40% and pH of 4.

After that, a ground calcium carbonate slurry was produced using the resulting dispersant 7-2C in the same manner as those in Comparative Example 7-1.

[0112]

Comparative Example 8-1 (Preparation of dispersant 8-1C, and preparation of slurry using dispersant 8-1C)

The polymerization, reduced-pressure evaporation, and neutralization were conducted in the same manner as those in Example 1-1, except that the keeping temperature of IPA in the flask, reaction temperature for polymerization, and temperature for neutralization were all set to 89°C instead of 80°C in Example 1-1 to obtain a dispersant 8-1C which contains an acryl-based polymer 8-1C and has a solid content of 40% and pH of 4.

After that, a ground calcium carbonate slurry was produced using the resulting dispersant 8-1C in the same manner as those in Example 1-1.

[0113]

Comparative Example 8-2 (Preparation of dispersant 8-2C, and preparation of slurry using dispersant 8-2)

In this Comparative Example 8-2, a dispersant 8-2C was prepared using the aqueous solution of IPA distilled off and recovered in the preparation of the dispersant 8-1C in Comparative Example 8-1.

When the IPA concentration of the aqueous solution of IPA recovered in Comparative

Example 8-1 was measured, the IPA concentration was 40%. Deionized water was added to set the same IPA concentration of an IPA aqueous solution as the IPA concentration of the IPA aqueous solution used in Comparative Example 8-1, and the IPA aqueous solution was obtained in the form of an aqueous solution of IPA at a concentration of 36%. Subsequently, the concentration-adjusted IPA aqueous solution was used and the same operations including polymerization, reduced-pressure evaporation, and neutralization were conducted as those in Comparative Example 8-1 to obtain a dispersant 8-2C which contains an acryl-based polymer 8-2C and has a solid content of 40% and pH of 4.

After that, a ground calcium carbonate slurry was produced using the resulting dispersant 8-2C in the same manner as those in Comparative Example 8-1.

[0114]

Comparative Example 9-1 (Preparation of dispersant 9-1C, and preparation of slurry using dispersant 9-1C)

The polymerization, reduced-pressure evaporation, and neutralization were conducted in the same manner as those in Example 1-1, except that the neutralization rate using the 32% sodium hydroxide aqueous solution was set to 10 mol% instead of 22 mol% in Example 1-1 to obtain a dispersant 9-1C which contains an acryl-based polymer 9-1C and has a solid content of 40% and pH of 2.

After that, a ground calcium carbonate slurry was produced using the resulting dispersant 9-1C in the same manner as those in Example 1-1.

[0115]

Comparative Example 9-2 (Preparation of dispersant 9-2C, and preparation of slurry using dispersant 9-2C)

In this Comparative Example 9-2, a dispersant 9-2C was prepared using the aqueous solution of IPA distilled off and recovered in the preparation of the dispersant 9-1C in Comparative Example 9-1.

When the IPA concentration of the aqueous solution of IPA recovered in Comparative Example 9-1 was measured, the IPA concentration was 40%. Deionized water was added to set the same IPA concentration of an IPA aqueous solution as the IPA concentration of the IPA aqueous solution used in Comparative Example 9-1, and the IPA aqueous solution was obtained in the form of an aqueous solution of IPA at a concentration of 36%. Subsequently, the concentration-adjusted IPA aqueous solution was used and the same operations including polymerization, reduced-pressure evaporation, and neutralization were conducted as those in Comparative Example 9-1 to obtain a dispersant 9-2C which contains an acryl-based polymer 9-2C and has a solid content of 40% and pH of 2.

After that, a ground calcium carbonate slurry was produced using the resulting dispersant 9-2C in the same manner as those in Comparative Example 9-1.

[0116]

Comparative Example 10-1 (Preparation of dispersant 10-1C, and preparation of slurry using dispersant 10-1C)

The polymerization, reduced-pressure evaporation, and neutralization were conducted in the same manner as those in Example 1-1, except that the neutralization rate using the 32% sodium hydroxide aqueous solution was set to 98 mol% instead of 22 mol% in Example 1-1 to obtain a dispersant 10-1C which contains an acryl-based polymer 10-1C and has a solid content of 40% and pH of 9.

After that, a ground calcium carbonate slurry was produced using the resulting dispersant 10-1C in the same manner as those in Example 1-1.

[0117]

Comparative Example 10-2 (Preparation of dispersant 10-2C, and preparation of slurry using dispersant 10-2C)

In this Comparative Example 10-2, a dispersant 10-2C was prepared using the aqueous solution of IPA distilled off and recovered in the preparation of the dispersant 10-1C in Comparative Example 10-1.

When the IPA concentration of the aqueous solution of IPA recovered in Comparative Example 10-1 was measured, the IPA concentration was 40%. Deionized water was added to set the same IPA concentration of an IPA aqueous solution as the IPA concentration of the IPA aqueous solution used in Comparative Example 10-1, and the IPA aqueous solution was obtained in the form of an aqueous solution of IPA at a concentration of 36%. Subsequently, the concentration-adjusted IPA aqueous solution was used and the same operations including polymerization, reduced-pressure evaporation, and neutralization were conducted as those in Comparative Example 10-1 to obtain a dispersant 10-2C which contains an acryl-based polymer 10-2C and has a solid content of 40% and pH of 9.

After that, a ground calcium carbonate slurry was produced using the resulting dispersant 10-2C in the same manner as those in Comparative Example 10-1.

[0118]

Comparative Example 11-1 (Preparation of dispersant 11-1C, and preparation of slurry using dispersant 11-1C)

The polymerization, reduced-pressure evaporation, and neutralization were conducted in the same manner as those in Example 1-1, except that an aqueous solution of IPA at a concentration of 10% was used instead of the IPA aqueous solution of 36% in Example 1-1 and

that the usage amount of the sodium persulfate aqueous solution was set to 80 g instead of 40g in Example 1-1 to obtain a dispersant 11-1C which contains an acryl-based polymer 11-1C and has a solid content of 40% and pH of 4.

After that, a ground calcium carbonate slurry was produced using the resulting dispersant 11-1C in the same manner as those in Example 1-1.

[0119]

Comparative Example 11-2 (Preparation of dispersant 11-2C, and preparation of slurry using dispersant 11-2C)

In this Comparative Example 11-2, a dispersant 11-2C was prepared using the aqueous solution of IPA distilled off and recovered in the preparation of the dispersant 11-1C in Comparative Example 11-1.

When the IPA concentration of the aqueous solution of IPA recovered in Comparative Example 11-1 was measured, the IPA concentration was 20%. Deionized water was added to set the same IPA concentration of an IPA aqueous solution as the IPA concentration of the IPA aqueous solution used in Comparative Example 11-1, and the IPA aqueous solution was obtained in the form of an aqueous solution of IPA at a concentration of 10%. Subsequently, the concentration-adjusted IPA aqueous solution was used and the same operations including polymerization, reduced-pressure evaporation, and neutralization were conducted as those in Comparative Example 11-1 to obtain a dispersant 11-2C which contains an acryl-based polymer 11-2C and has a solid content of 40% and pH of 4.

After that, a ground calcium carbonate slurry was produced using the resulting dispersant 11-2C in the same manner as those in Comparative Example 11-1.

[0120]

Comparative Example 12-1 (Preparation of dispersant 12-1C, and preparation of slurry using dispersant 12-1C)

The polymerization, reduced-pressure evaporation, and neutralization were conducted in the same manner as those in Example 1-1, except that the usage amount of the sodium hypophosphite was set to 6 g instead of 20g in Example 1-1 and the usage amount of the sodium persulfate aqueous solution was set to 80 g instead of 40g in Example 1-1 to obtain a dispersant 12-1C which contains an acryl-based polymer 12-1C and has a solid content of 40% and pH of 4.

After that, a ground calcium carbonate slurry was produced using the resulting dispersant 12-1C in the same manner as those in Example 1-1.

[0121]

Comparative Example 12-2 (Preparation of dispersant 12-2C, and preparation of slurry

using dispersant 12-2C)

In this Comparative Example 12-2, a dispersant 12-2C was prepared using the aqueous solution of IPA distilled off and recovered in the preparation of the dispersant 12-1C in Comparative Example 12-1.

When the IPA concentration of the aqueous solution of IPA recovered in Comparative Example 12-1 was measured, the IPA concentration was 40%. Deionized water was added to set the same IPA concentration of an IPA aqueous solution as the IPA concentration of the IPA aqueous solution used in Comparative Example 12-1, and the IPA aqueous solution was obtained in the form of an aqueous solution of IPA at a concentration of 36%. Subsequently, the concentration-adjusted IPA aqueous solution was used and the same operations including polymerization, reduced-pressure evaporation, and neutralization were conducted as those in Comparative Example 12-1 to obtain a dispersant 12-2C which contains an acryl-based polymer 12-2C and has a solid content of 40% and pH of 4.

After that, a ground calcium carbonate slurry was produced using the resulting dispersant 12-2C in the same manner as those in Comparative Example 12-1.

[0122]

Comparative Example 13-1 (Preparation of dispersant 13-1C, and preparation of slurry using dispersant 13-1C)

The polymerization, reduced-pressure evaporation, and neutralization were conducted in the same manner as those in Example 1-1, except that an aqueous solution of IPA at a concentration of 10% was used instead of the IPA aqueous solution of 36% in Example 1-1 and that the usage amount of the sodium hypophosphite was set to 33 g instead of 20g in Example 1-1 to obtain a dispersant 13-1C which contains an acryl-based polymer 13-1C and has a solid content of 40% and pH of 4.

After that, a ground calcium carbonate slurry was produced using the resulting dispersant 13-1C in the same manner as those in Example 1-1.

[0123]

Comparative Example 13-2 (Preparation of dispersant 13-2C, and preparation of slurry using dispersant 13-2C)

In this Comparative Example 13-2, a dispersant 13-2C was prepared using the aqueous solution of IPA distilled off and recovered in the preparation of the dispersant 13-1C in Comparative Example 13-1.

When the IPA concentration of the aqueous solution of IPA recovered in Comparative Example 13-1 was measured, the IPA concentration was 20%. Deionized water was added to set the same IPA concentration of an IPA aqueous solution as the IPA concentration of the IPA

aqueous solution used in Comparative Example 13-1, and the IPA aqueous solution was obtained in the form of an aqueous solution of IPA at a concentration of 10%. Subsequently, the concentration-adjusted IPA aqueous solution was used and the same operations including polymerization, reduced-pressure evaporation, and neutralization were conducted as those in Comparative Example 13-1 to obtain a dispersant 13-2C which contains an acryl-based polymer 13-2C and has a solid content of 40% and pH of 4.

After that, a ground calcium carbonate slurry was produced using the resulting dispersant 13-2C in the same manner as those in Comparative Example 13-1.

[0124]

Comparative Example 14-1 (Preparation of dispersant 14-1C, and preparation of slurry using dispersant 14-1C)

The polymerization, reduced-pressure evaporation, and neutralization were conducted in the same manner as those in Example 1-1, except that the keeping temperature of IPA in the flask, reaction temperature for polymerization, and temperature for neutralization were all set to 60°C instead of 80°C in Example 1-1 and that the usage amount of the sodium persulfate aqueous solution was set to 80 g instead of 40 g in Example 1-1 to obtain a dispersant 14-1C which contains an acryl-based polymer 14-1C and has a solid content of 40% and pH of 4.

After that, a ground calcium carbonate slurry was produced using the resulting dispersant 14-1C in the same manner as those in Example 1-1.

[0125]

Comparative Example 14-2 (Preparation of dispersant 14-2C, and preparation of slurry using dispersant 14-2C)

In this Comparative Example 14-2, a dispersant 14-2C was prepared using the aqueous solution of IPA distilled off and recovered in the preparation of the dispersant 14-1C in Comparative Example 14-1.

When the IPA concentration of the aqueous solution of IPA recovered in Comparative Example 14-1 was measured, the IPA concentration was 40%. Deionized water was added to set the same IPA concentration of an IPA aqueous solution as the IPA concentration of the IPA aqueous solution used in Comparative Example 14-1, and the IPA aqueous solution was obtained in the form of an aqueous solution of IPA at a concentration of 36%. Subsequently, the concentration-adjusted IPA aqueous solution was used and the same operations including polymerization, reduced-pressure evaporation, and neutralization were conducted as those in Comparative Example 14-1 to obtain a dispersant 14-2C which contains an acryl-based polymer 14-2C and has a solid content of 40% and pH of 4.

After that, a ground calcium carbonate slurry was produced using the resulting

dispersant 14-2C in the same manner as those in Comparative Example 14-1.
[0126]

Table 1

		Example											
		1-1	1-2	2-1	2-2	3-1	3-2	4-1	4-2	5-1	5-2	6-1	6-2
Acrylic acid (g)		600		600		600		600		600		600	
IPA concentration (%)		36		48		36		36		36		36	
Aqueous solution of IPA (g)		770		770		770		770		770		770	
Number of parts of IPA based on 100 parts of monomer		46		62		46		46		46		46	
Sodium hypophosphite (g)		20		20		17		20		20		20	
Number of parts of sodium hypophosphite based on 100 parts of monomer		3.3		3.3		2.8		3.3		3.3		3.3	
Sodium persulfate concentration (%)		15		15		15		15		15		15	
Sodium persulfate aqueous solution (g)		40		40		40		30		40		40	
Number of parts of sodium persulfate based on 100 parts of monomer		1.0		1.0		1.0		0.8		1.0		1.0	
Reaction condition	Internal temperature in polymerization process (°C)	80		80		80		80		75		80	
	Carboxyl group neutralization rate (mol%)	22		22		22		22		22		40	
	Monomer feeding time (hr)	4		4		4		4		4		4	
Dispersant for calcium carbonate		1-1	1-2	2-1	2-2	3-1	3-2	4-1	4-2	5-1	5-2	6-1	6-2
Physicality/Evaluation	Solid content (% by mass)	40	40	40	40	40	40	40	40	40	40	40	40
	pH	4	4	4	4	4	4	4	4	4	4	5	5
	Weight average molecular weight (Mw)	6000	6100	5300	5400	6600	6800	6400	6500	6000	6200	6000	6100
	<1.32- μ m integrated value of slurry (%)	100	100	100	100	100	100	100	100	100	100	100	100
	Median diameter of slurry (μ m)	0.67	0.67	0.66	0.66	0.67	0.67	0.68	0.68	0.67	0.67	0.68	0.68
	Slurry viscosity at current day of wet grinding (mPa·s)	210	220	200	210	200	200	200	210	200	200	230	230
	Slurry viscosity after 7 days (mPa·s)	1800	1800	2000	2100	1700	1800	1700	1700	1700	1800	2100	2200

Table 2

		Example											
		7-1	7-2	8-1	8-2	9-1	9-2	10-1	10-2	11-1	11-2	12-1	12-2
Acrylic acid (g)		600		600		600		600		600		600	
IPA concentration (%)		20		53		36		36		36		36	
Aqueous solution of IPA (g)		770		770		770		770		770		770	
Number of parts of IPA based on 100 parts of monomer		26		68		46		46		46		46	
Sodium hypophosphite (g)		20		20		9.5		26.5		20		20	
Number of parts of sodium hypophosphite based on 100 parts of monomer		3.3		3.3		1.6		4.4		3.3		3.3	
Sodium persulfate concentration (%)		15		15		15		15		15		15	
Sodium persulfate aqueous solution (g)		40		40		40		40		12		60	
Number of parts of sodium persulfate based on 100 parts of monomer		1.0		1.0		1.0		1.0		0.3		1.5	
Reaction condition	Internal temperature in polymerization process (°C)	80		80		80		80		80		80	
	Carboxyl group neutralization rate (mol%)	22		22		22		22		22		22	
	Monomer feeding time (hr)	4		4		4		4		4		4	
Dispersant for calcium carbonate		7-1	7-2	8-1	8-2	9-1	9-2	10-1	10-2	11-1	11-2	12-1	12-2
Physicality/Evaluation	Solid content (% by mass)	40	40	40	40	40	40	40	40	40	40	40	40
	pH	4	4	4	4	4	4	4	4	4	4	4	4
	Weight average molecular weight (Mw)	7900	8000	4800	4900	8100	8200	4600	4600	8200	8300	5200	5200
	<1.32- μ m integrated value of slurry (%)	100	100	100	100	100	100	100	100	100	100	100	100
	Median diameter of slurry (μ m)	0.68	0.68	0.66	0.66	0.68	0.68	0.66	0.66	0.68	0.68	0.67	0.67
	Slurry viscosity at current day of wet grinding (mPa·s)	280	290	200	200	250	260	230	240	260	270	240	240
	Slurry viscosity after 7 days (mPa·s)	2500	2500	3000	3100	2200	2300	3200	3200	2300	2400	2900	2900

Table 3

		Example							
		13-1	13-2	14-1	14-2	15-1	15-2	16-1	16-2
Acrylic acid (g)		600		600		600		575	
Methyl acrylate (%)		0		0		0		25	
IPA concentration (%)		36		36		65		36	
Aqueous solution of IPA (g)		770		770		770		770	
Number of parts of IPA based on 100 parts of monomer		46		46		83		46	
Sodium hypophosphite (g)		20		20		15		20	
Number of parts of sodium hypophosphite based on 100 parts of monomer		3.3		3.3		2.5		3.3	
Sodium persulfate concentration (%)		15		15		15		15	
Sodium persulfate aqueous solution (g)		40		40		40		40	
Number of parts of sodium persulfate based on 100 parts of monomer		1.0		1.0		1.0		1.0	
Reaction condition	Internal temperature in polymerization process (°C)	80		80		80		80	
	Carboxyl group neutralization rate (mol%)	18		90		22		22	
	Monomer feeding time (hr)	4		4		4		4	
Dispersant for calcium carbonate		13-1	13-2	14-1	14-2	15-1	15-2	16-1	16-2
Physi- city/Evaluation	Solid content (% by mass)	40	40	40	40	40	40	40	40
	pH	3	3	7	7	4	4	4	4
	Weight average molecular weight (Mw)	6000	6100	6000	6100	5800	5700	5900	6000
	<1.32- μ m integrated value of slurry (%)	100	100	100	100	100	100	100	100
	Median diameter of slurry (μ m)	0.67	0.67	0.68	0.68	0.67	0.67	0.67	0.67
	Slurry viscosity at current day of wet grinding (mPa·s)	210	210	310	310	200	200	220	230
	Slurry viscosity after 7 days (mPa·s)	1800	1800	3300	3300	1700	1700	1900	2000

Table 4

		Comparative Example									
		1-1	1-2	2-1	2-2	3-1	3-2	4-1	4-2	5-1	5-2
Acrylic acid (g)		600		600		600		600		600	
IPA concentration (%)		10		65		36		36		36	
Aqueous solution of IPA (g)		770		770		770		770		770	
Number of parts of IPA based on 100 parts of monomer		13		83		46		46		46	
Sodium hypophosphite (g)		20		20		6		33		20	
Number of parts of sodium hypophosphite based on 100 parts of monomer		3.3		3.3		1.0		5.5		3.3	
Sodium persulfate concentration (%)		15		15		15		15		15	
Sodium persulfate aqueous solution (g)		40		40		40		40		7	
Number of parts of sodium persulfate based on 100 parts of monomer		1.0		1.0		1.0		1.0		0.2	
Reaction condition	Internal temperature in polymerization process (°C)	80		80		80		80		80	
	Carboxyl group neutralization rate (mol%)	22		22		22		22		22	
	Monomer feeding time (hr)	4		4		4		4		4	
Dispersant for calcium carbonate		1-1C	1-2C	2-1C	2-2C	3-1C	3-2C	4-1C	4-2C	5-1C	5-2C
Physicoity/Evaluation	Solid content (% by mass)	40	40	40	40	40	40	40	40	40	40
	pH	4	4	4	4	4	4	4	4	4	4
	Weight average molecular weight (Mw)	11000	11000	4300	4200	10900	10800	4300	4200	9100	9900
	<1.32- μ m integrated value of slurry (%)	100	100	100	100	100	100	100	100	100	100
	Median diameter of slurry (μ m)	0.68	0.68	0.66	0.66	0.68	0.68	0.68	0.68	0.68	0.68
	Slurry viscosity at current day of wet grinding (mPa·s)	1400	1400	240	250	1200	1200	1100	1200	1000	1300
		7200	7300	6900	7100	6800	6900	7900	8000	6600	7500
Remarks					Concentration of IPA was necessary					Amount of unreacted AA was large	AA was mixed in IPA

Table 5

		Comparative Example									
		6-1	6-2	7-1	7-2	8-1	8-2	9-1	9-2	10-1	10-2
Acrylic acid (g)		600		600		600		600		600	
IPA concentration (%)		36		36		36		36		36	
Aqueous solution of IPA (g)		770		770		770		770		770	
Number of parts of IPA based on 100 parts of monomer		46		46		46		46		46	
Sodium hypophosphite (g)		20		20		20		20		20	
Number of parts of sodium hypophosphite based on 100 parts of monomer		3.3		3.3		3.3		3.3		3.3	
Sodium persulfate concentration (%)		15		15		15		15		15	
Sodium persulfate aqueous solution (g)		80		40		40		40		40	
Number of parts of sodium persulfate based on 100 parts of monomer		2.0		1.0		1.0		1.0		1.0	
Reaction condition	Internal temperature in polymerization process (°C)	80		60		89		80		80	
	Carboxyl group neutralization rate (mol%)	22		22		22		10		98	
	Monomer feeding time (hr)	4		4		4		4		4	
Dispersant for calcium carbonate		6-1C	6-2C	7-1C	7-2C	8-1C	8-2C	9-1C	9-2C	10-1C	10-2C
Physicality/Evaluation	Solid content (% by mass)	40	40	40	40	40	40	40	40	40	40
	pH	4	4	4	4	4	4	2	2	9	9
	Weight average molecular weight (Mw)	4900	4800	8900	9900	5500	5500	6000	6100	6000	6100
	<1.32- μ m integrated value of slurry (%)	100	100	100	100	100	100	-	-	100	100
	Median diameter of slurry (μ m)	0.68	0.68	0.68	0.68	0.68	0.68	-	-	0.68	0.68
	Slurry viscosity at current day of wet grinding (mPa·s)	1000	1100	900	1200	1000	1000	-	-	1000	1000
	Slurry viscosity after 7 days (mPa·s)	7400	7500	7100	8000	6600	6700	-	-	9100	9200
Remarks				Amount of unreacted AA was large	AA was mixed in IPA			Could not be ground	Could not be ground		

[0132]

In Examples 1-1 to 16-2, usage amounts of sodium hypophosphite and sodium persulfate were respectively in the range from 1.4 to 4.4 parts by mass and 0.3 to 1.5 parts by mass based on 100 parts by mass of the monomer, the reaction temperature employed in the polymerization process was between 68°C and 82°C, the weight average molecular weight of the acryl-based polymer was in the range from 4,500 to 8,500, and the neutralization rate of carboxyl groups was 15 to 95 mol%. According to the above results, it was found that dispersants of Examples 1-1 to 16-2 were ones in which an initial viscosity of a calcium

Table 6

		Comparative Example							
		11-1	11-2	12-1	12-2	13-1	13-2	14-1	14-2
Acrylic acid (g)		600		600		600		600	
IPA concentration (%)		10		36		10		36	
Aqueous solution of IPA (g)		770		770		770		770	
Number of parts of IPA based on 100 parts of monomer		13		46		13		46	
Sodium hypophosphite (g)		20		6.0		33		20	
Number of parts of sodium hypophosphite based on 100 parts of monomer		3.3		1.0		5.5		3.3	
Sodium persulfate concentration (%)		15		15		15		15	
Sodium persulfate aqueous solution (g)		80		80		40		80	
Number of parts of sodium persulfate based on 100 parts of monomer		2.0		2.0		1.0		2.0	
Reaction condition	Internal temperature in polymerization process (°C)	80		80		80		60	
	Carboxyl group neutralization rate (mol%)	22		22		22		22	
	Monomer feeding time (hr)	4		4		4		4	
Dispersant for calcium carbonate		11-1C	11-2C	12-1C	12-2C	13-1C	13-2C	14-1C	14-2C
Physicality/Evaluation	Solid content (% by mass)	40	40	40	40	40	40	40	40
	pH	4	4	4	4	4	4	4	4
	Weight average molecular weight (Mw)	7400	7500	6900	7000	7100	7200	7300	7400
	<1.32- μ m integrated value of slurry (%)	100	100	100	100	100	100	100	100
	Median diameter of slurry (μ m)	0.68	0.68	0.68	0.68	0.68	0.68	0.68	0.68
	Slurry viscosity at current day of wet grinding (mPa·s)	1000	1000	900	900	1200	1300	1000	1000
	Slurry viscosity after 7 days (mPa·s)	7000	7100	6500	6600	8000	8000	6900	7000

carbonate slurry obtained by wet grinding was low, and a significant increase in the viscosity with passage of time was suppressed.

Meanwhile, in Comparative Examples 1-1 and 1-2, the weight average molecular weight of the acryl-based polymer was high (11,000). Slurry viscosities at the time of grinding of calcium carbonate were high, and a significant increase was observed in slurry viscosities after seven days. In Comparative Examples 2-1 and 2-2, slurry viscosities at the time of grinding of calcium carbonate were low. Since weight average molecular weights of the acryl-based polymer were too low (4,300 and 4,200), a significant increase was observed in slurry viscosities after seven days. The weight average molecular weights of the acryl-based polymer in Comparative Examples 1-1 and 1-2 were high, and the weight average molecular weights of the acryl-based polymer in Comparative Examples 2-1 and 2-2 were low. When the usage amounts of hypophosphite and persulfate are specified, the molecular weight of the acryl-based polymer (first polymer) can be adjusted according to the usage amount of IPA. Specifically, since the usage amounts of IPA used in Comparative Examples 1-1 and 1-2 were each 13 parts by mass and were smaller than 46 parts by mass of Example 1-1 based on 100 parts by mass of the monomer. The usage amounts of IPA used in Comparative Examples 2-1 and 2-2 were each 83 parts by mass and were larger than 46 parts by mass of Example 1-1 based on 100 parts by mass of the monomer. Accordingly, the molecular weight of the acryl-based polymer was affected. Since the concentrations of IPA in the IPA aqueous solution used in Comparative Examples 2-1 and 2-2 were high (65%), it was necessary to concentrate the recovered IPA aqueous solution (azeotrope) in order to recycle the recovered IPA.

[0133]

In Comparative Examples 3-1 and 3-2, the usage amounts of sodium hypophosphite were as small as 1 part by mass based on 100 parts by mass of the monomer. The weight average molecular weights of the acryl-based polymer were high (10,900 and 10,800). Slurry viscosities at the time of grinding of calcium carbonate were high, and a significant increase was observed in slurry viscosities after seven days.

In Comparative Examples 4-1 and 4-2, the usage amount of sodium hypophosphite was as large as 5.5 parts by mass based on 100 parts by mass of the monomer, and the weight average molecular weights of the acryl-based polymer were low. Slurry viscosities at the time of grinding of calcium carbonate were high, and a significant increase was observed in slurry viscosities after seven days.

[0134]

In Comparative Examples 5-1 and 5-2, the usage amounts of sodium persulfate were as small as 0.2 part by mass based on 100 parts by mass of the monomer. The weight average

molecular weights of the acryl-based polymer were high (9,100 and 9,900), and the amounts of unreacted acrylic acid were increased. Slurry viscosities at the time of grinding of calcium carbonate were high, and a significant increase was observed in slurry viscosities after seven days. Since a mixing of acrylic acid was found in the recovered IPA aqueous solution in Comparative Example 5-1, it is considered that it is inappropriate to recycle the IPA aqueous solution recovered in Comparative Example 5-1.

In Comparative Examples 6-1 and 6-2, the usage amounts of sodium persulfate were as large as 2.0 parts by mass based on 100 parts by mass of the monomer. The weight average molecular weights of the acryl-based polymer were within the appropriate range, however, slurry viscosities at the time of grinding of calcium carbonate were high, and a significant increase was observed in slurry viscosities after seven days.

[0135]

In Comparative Examples 7-1 and 7-2, the weight average molecular weights of the acryl-based polymer were increased to some extent, and the amounts of unreacted acrylic acid were increased since the reaction temperature employed in the polymerization process was as low as 60°C. Slurry viscosities at the time of grinding of calcium carbonate were high, and a significant increase was observed in slurry viscosities after seven days. Since a mixing of acrylic acid was found in the recovered IPA aqueous solution in Comparative Example 7-1, it is considered that it is inappropriate to recycle the IPA aqueous solution recovered in Comparative Example 7-1.

In Comparative Examples 8-1 and 8-2, the reaction temperature employed in the polymerization process was as high as 89°C. The weight average molecular weights of the acryl-based polymer were within the appropriate range, however, slurry viscosities at the time of grinding of calcium carbonate were high, and a significant increase was observed in slurry viscosities after seven days.

[0136]

In Comparative Examples 9-1 and 9-2, it was impossible to grind calcium carbonate, and a slurry could not be obtained since the neutralization rates of carboxyl groups were as low as 10 mol%. In Comparative Examples 10-1 and 10-2, the neutralization rates of carboxyl groups were as high as 98 mol%. Calcium carbonate could be ground, however, slurry viscosities at the time of grinding of calcium carbonate were high, and a significant increase was observed in slurry viscosities after seven days.

[0137]

In Comparative Examples 11-1 and 11-2 in which usage amount of sodium persulfate was increased to be 2 parts by mass based on 100 parts by mass of the monomer, and IPA was

used in an amount of 13 parts by mass based on 100 parts by mass of the monomer in order to obtain an acryl-based polymer having a weight average molecular weight within the appropriate range, the weight average molecular weights of the resulting acryl-based polymer were within the appropriate range (around 7,400). Since the amounts of sodium persulfate were large, slurry viscosities at the time of grinding of calcium carbonate were high, and a significant increase was observed in slurry viscosities after seven days.

In Comparative Examples 12-1 and 12-2 in which usage amount of sodium persulfate was increased to be 2 parts by mass based on 100 parts by mass of the monomer, and sodium hypophosphite was used in an amount of 1 part by mass based on 100 parts by mass of the monomer in order to obtain an acryl-based polymer having a weight average molecular weight within the appropriate range, the weight average molecular weights of the resulting acryl-based polymer were within the appropriate range (around 6,900). Since the amount of sodium persulfate was large, slurry viscosities at the time of grinding of calcium carbonate were high, and a significant increase was observed in slurry viscosities after seven days.

[0138]

In Comparative Examples 13-1 and 13-2 in which usage amount of sodium hypophosphite was increased to be 5.5 parts by mass based on 100 parts by mass of the monomer, and IPA was used in an amount of 13 parts by mass based on 100 parts by mass of the monomer in order to obtain an acryl-based polymer having a weight average molecular weight within the appropriate range, the weight average molecular weights of the resulting acryl-based polymer were within the appropriate range (around 7,100). Since the amount of sodium hypophosphite was large, slurry viscosities at the time of grinding of calcium carbonate were high, and a significant increase was observed in slurry viscosities after seven days.

In Comparative Examples 14-1 and 14-2 in which usage amount of sodium persulfate was increased to be 2 parts by mass based on 100 parts by mass of the monomer, and the reaction temperature employed in the polymerization process was set to 60°C, the weight average molecular weights of the resulting acryl-based polymer were within the appropriate range (around 7,300). Since the amount of sodium persulfate was large, slurry viscosities at the time of grinding of calcium carbonate were high, and a significant increase was observed in slurry viscosities after seven days.

[INDUSTRIAL APPLICABILITY]

[0139]

Clearly from the above description, the dispersion for calcium carbonate of the present invention exhibits excellent dispersibility and long-term dispersion stability. In particular, the

dispersion is effectively used as a dispersant for wet grinding of calcium carbonate.

According to the production method of a dispersant for calcium carbonate of the present invention, the above dispersant for calcium carbonate can be efficiently produced. When the recovered isopropyl alcohol from the concentration process according to the present invention is used in the polymerization process, evaporated isopropyl alcohol need not be disposed of.

Therefore, it is possible to provide a method for producing a dispersant for calcium carbonate that can reduce cost, and achieve excellent production efficiency and environmental protection.

What is claimed is:

1. A dispersant for calcium carbonate characterized in that said dispersant comprises an acryl-based polymer that is obtained by a method comprising a polymerization process in which a monomer containing acrylic acid is polymerized in the presence of a hypophosphite and a persulfate using an isopropyl alcohol aqueous solution as a solvent, and a neutralization process in which 15 to 95 mol% of carboxyl groups included in a structural unit derived from said monomer constituting a formed polymer is neutralized, that said hypophosphite is used in an amount from 1.4 to 4.4 parts by mass based on 100 parts by mass of said monomer, that said persulfate is used in an amount from 0.3 to 1.5 parts by mass based on 100 parts by mass of said monomer, that a reaction temperature employed in said polymerization process is between 68°C and 82°C, and that a weight average molecular weight of said acryl-based polymer is in the range from 4,500 to 8,500.
2. The dispersant for calcium carbonate according to Claim 1, wherein said method further comprises, after said polymerization process, a concentration process in which said isopropyl alcohol is distilled off.
3. The dispersant for calcium carbonate according to Claim 2, wherein said isopropyl alcohol recovered after said concentration process is used in said polymerization process.
4. The dispersant for calcium carbonate according to any one of Claims 1 to 3, wherein a concentration of said isopropyl alcohol in said isopropyl alcohol aqueous solution is in the range from 15% to 55% by mass.
5. The dispersant for calcium carbonate according to any one of Claims 1 to 4, wherein a content of said acrylic acid used in said polymerization process is in the range from 80% to 100% by mass based on 100% by mass of a total amount of said monomer.
6. A production method of a dispersant for calcium carbonate containing an acryl-based polymer, characterized in that said method comprises a polymerization process in which a monomer containing acrylic acid is polymerized in the presence of a hypophosphite and a persulfate using an isopropyl alcohol aqueous solution as a solvent, and a neutralization process in which 15 to 95 mol% of carboxyl groups included in a structural unit derived from said monomer constituting a formed polymer is neutralized, that said hypophosphite is used in an amount from 1.4 to 4.4 parts by mass based on 100 parts by mass of said monomer, that said persulfate is used in an amount from 0.3 to 1.5 parts by mass based on 100 parts by mass of said monomer, that a reaction temperature employed in said polymerization process is between 68°C and 82°C, and that a weight average molecular weight of said acryl-based polymer is in the

range from 4,500 to 8,500.

7. The production method of the dispersant for calcium carbonate according to Claim 6, further comprising a concentration process in which said isopropyl alcohol is distilled off, after said polymerization process.

8. The production method of the dispersant for calcium carbonate according to Claim 7, wherein said isopropyl alcohol recovered after said concentration process is used in said polymerization process.

9. The production method of the dispersant for calcium carbonate according to any one of Claims 6 to 8,

wherein a concentration of said isopropyl alcohol in said isopropyl alcohol aqueous solution is in the range from 15% to 55% by mass.

10. The production method of the dispersant for calcium carbonate according to any one of Claims 6 to 9,

wherein a content of said acrylic acid used in said polymerization process is in the range from 80% to 100% by mass based on 100% by mass of a total amount of said monomer.