

[54] **DEFOAMER COMPOSITIONS FOR AQUEOUS SYSTEMS**

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[58] Field of Search **252/321, 358**

[56] **References Cited**

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[57] **ABSTRACT**

Defoamer compositions and method of defoaming aqueous systems such as latex or paper and pulp systems, for example, compositions being composed of certain fatty amides, inorganic particles (talc, for example), hydrocarbon liquid, an oil soluble polymer and a silicone oil.

6 Claims, No Drawings

DEFOAMER COMPOSITIONS FOR AQUEOUS SYSTEMS

BACKGROUND OF THE INVENTION

This invention relates to a defoamer composition and more particularly to an improved defoamer containing an amide.

Materials such as inorganic particles, liquid hydrocarbons and silicone oils have been used in the preparation of defoamer compositions. Although these compositions have been useful in suppressing and controlling foam formation, they have the disadvantage in that the solid particles therein tend to settle on standing. Consequently, after standing for extended periods of time, it was necessary to agitate the compositions immediately prior to use.

It is an object of the present invention to provide improved defoamer compositions. Another object is to provide improved defoamer compositions containing certain amides, inorganic particles, a liquid hydrocarbon, an oil soluble polymer and a silicone oil. Other objects of this invention will become apparent from the detailed description given herein. However, it is intended that the detailed description and specific examples do not limit this invention but merely indicate preferred embodiments.

SUMMARY OF THE INVENTION

The objects of this invention have been accomplished in the following manner. Defoamer compositions have been prepared by using certain amides, inorganic particles, a liquid hydrocarbon, an oil soluble polymer and a silicone oil. The compositions contain from about 1 to about 12 parts by weight of certain amides, from about 5 to about 25 parts by weight of inorganic particles, from about 70 to about 92 parts by weight of a liquid hydrocarbon, from about 0.5 to about 7.5 parts by weight of an oil soluble polymer and from 0 to about 0.2 parts by weight of silicone oil. It has been found that the solid or particulate matter in such compositions tends to remain suspended on standing for significantly longer periods of time.

DESCRIPTION OF THE PREFERRED EMBODIMENTS

Useful amides include those obtained by reaction of a polyamine containing at least one alkylene group having from two to six carbon atoms and a fatty acid having from six to 18 carbon atoms. For example, the amide obtained by reaction of a polyamine and a fatty acid or mixture of fatty acids such as hexanoic, decanoic, lauric, palmitic, oleic and stearic acids, hydroxy acids such as ricinoleic acid or naphthenic acids such as are obtained as byproducts from the refining of petroleum. Natural mixtures of fatty acids such as, tall oil acids, tallow fatty acids and the like can be used. Suitable amines include ethylene diamine, butylene diamine, diethylene triamine, triethylene tetramine, hexamethylene diamine, decamethylene diamine, hydroxyethyl ethylene diamine, 1:3-diamino-2-propanol or the like.

One of the amides useful herein is prepared in the following manner. 95.7 parts by weight of bleached-hydrogenated tallow fatty acids is charged into a stainless steel reactor equipped with a condenser, water trap and agitator. The entire process including charging of reactants and cooling of the reaction product is carried out under a nitrogen atmosphere. The acids charge is heated to about 165° C. to about 175° C. and 10.1 parts by weight of ethylene diamine is added with agitation. After the diamine is added, the reaction mixture is heated to about 180° C. to about 185° C. The mixture is reacted at about 180° C. to about 185° C. until the acid value is less than 5, the alkalinity Scott-Volumeter than 0.6 percent by weight. The resulting reaction product of ethylene diamine and tallow fatty acid is then cooled to room temperature. If desired, the cooled mass can be ground to obtain the product in the form of a very fine powder.

Inorganic particles such as talc or powdered calcium hydroxide can be used. A suitable talc is a fluffy talc (E V Micro Talc — Charles B. Chrystal Co. Inc., N.Y., N.Y.) having a bulking value of 26 pounds per cubic foot or an average

of 3.53 grams per cubic inch on the Scott-Volumeter Test. This talc has an average particle size of 3.5 microns and 99.98 percent by weight of which passes through a 325 mesh screen. It is used in the manufacture of pressed powders and dusting powders. Another useful talc (P-275 — International Talc Co., Inc., N.Y., N.Y.) is a platey talc which has an average particle size of 1.4 microns and 99.99 percent by weight of which passes through a 325 mesh screen. This talc has a Hegman fineness of about 6 and high oil absorption. It is used in housepaints, primers, lacquers and industrial finishes.

A suitable powdered calcium hydroxide is a hydrate of lime containing about 95% by weight of calcium hydroxide. A typical sample of this material after jet milling gave the following results in a screen test:

% by weight passing	100 mesh	100
	200 mesh	99
	325 mesh	98

The liquid hydrocarbon can be any liquid aliphatic, alicyclic, aromatic hydrocarbon or mixture thereof. The hydrocarbon should be liquid at room temperature and atmospheric pressure, have a viscosity of from about 30 to about 400 SUS (Saybolt Universal Seconds at 100° F.), a minimum boiling point of at least 150° F. and an average from about six to 25 carbon atoms. Suitable hydrocarbons include benzene, hexane, heptane, octane, mineral seal oil, stoddard solvent, petroleum naphtha, toluene, xylene, paraffinic mineral oil, naphthenic mineral oil or the like. If desired mixtures of two or more hydrocarbons can be used.

An example of the oil soluble polymer is a copolymer of vinyl acetate and fumaric acid esterified with a tallow alcohol. Such an oil soluble polymer available from Enjay Chemical Co. in the form of a mineral oil solution thereof under the trade name Paratone 440 which consists of about 25 parts by weight of said copolymer in 75 parts by weight of a liquid hydrocarbon. This product has the following properties:

Viscosity at 210°F, cs	1300
Flash Point, COC, °F	360
Color, ASTM	40
Specific Gravity 60/60°F	0.92

The silicone oil can be a polysiloxane oil such as an alkyl, aryl, alicyclic or aralkyl siloxane or polysiloxane having a viscosity of from about 10 to about 3,000 centistokes at 25° C. Preferred silicone oils include alkyl polysiloxanes having viscosities of from about 40 to about 1,000 centistokes at 25° C. These alkyl polysiloxanes include dimethyl polysiloxane, diethyl polysiloxane, dipropyl polysiloxane, methyl ethyl polysiloxane, dioctyl polysiloxane, dihexyl polysiloxane, methyl propyl polysiloxane, dibutyl polysiloxane, didodecyl polysiloxane or the like having a viscosity of from about 10 to about 3,000 centistokes at 25° C.

The defoamer composition is prepared by mixing together the liquid hydrocarbon and oil soluble polymer and stirring until uniform. The inorganic particles and amide are added next. The mixture is then heated to about 160° C. and held for 15 minutes at about 160° C. After cooling to room temperature, the formulation is homogenized using a hand homogenizer (C. W. Logeman Co., Brooklyn, N.Y.). Finally, if desired, the silicone oil is added with stirring.

The defoamer composition was evaluated for settling in the following manner. Fresh, homogenized formulation was added to a standard capped 2oz. jar to an arbitrary height, that is, about 5 cm. After standing for four days, three layers were observed, i.e., a settlement at the bottom, a gelatinous suspension above the bottom layer and a relatively clear liquid layer at the top. The height of the settlement at the bottom was recorded.

The defoamers of this invention are especially adapted to defoam aqueous systems such as are found in the preparation of latex, glues, resinous materials, starches, paper, pulp and the like. The defoamer compositions are used in small

amounts depending upon the system in which they are employed. In general, from about 0.01 percent to about 0.5 percent by weight of the defoamer based on the foam producing materials present in the aqueous system is satisfactory. Amounts in excess of about 0.5 percent by weight can be utilized to defoam the aqueous systems but generally such amounts are not practical due to the high cost of the defoamer.

For a fuller understanding of the nature and objects of this invention, reference may be made to the following examples. These examples are given merely to illustrate the invention and are not to be construed in a limiting sense. The terms g, percent by weight, sec and cm are used to indicate grams, percent by weight, second and centimeter respectively in these examples.

EXAMPLE I

This example is directed to a comparison of defoamer compositions, both outside of the scope of the present invention and within the scope of the present invention.

Formulations A through F as shown in Table I were prepared and evaluated using the procedure given above. Formulations, A, C and E which are outside the scope of the present invention (contained no Amide) and which contained 74.8 parts by weight of a liquid hydrocarbon, 10 parts by weight of Paratone 440, 0.2 parts by weight of silicone oil and 15 parts by weight of inorganic particles identified as Talc I, Talc II and Calcium hydroxide respectively. Formulations B, D and F in Table I which are within the scope of the present invention contained 72.8 parts by weight of a liquid hydrocarbon, 10 parts by weight of Paratone 440, 0.2 parts by weight of a silicone oil, 15 parts by weight of inorganic particles identified as Talc I, Talc II and Calcium hydroxide respectively and 2 parts by weight of Amide.

The liquid hydrocarbon was a mineral oil having the following properties:

Viscosity at 100°F, SUS	100-110
Viscosity at 210°F, SUS	40
API Gravity at 60°F	33.5
Sp.Gr. at 60°F	0.8760
lb/gal.	7.24
Flash Point °F	380
Pour Point °F	0
Kauri Butanol Value	20.0
% Aromatics	4
% Naphthenics	24
% Paraffinics	72
Distillation Range °F	50% distilled at 720°F

The silicone oil was L-45 (Union Carbide Corp.), a dimethyl siloxane polymer having the following properties:

Viscosity (centistokes)	50
Specific Gravity at 25°C	0.960
Volatile	1% maximum
Pour Point	-67°F
Flash Point	500°F
Surface tension	20.8 dynes/cm
Appearance	Clear water white

Properties of Talc I (E. V. Micro Talc), Talc II (P-275) and Calcium hydroxide used as the inorganic particles in the formulations are given above.

The Amide used in Formulations B, D and F was the above described reaction product of ethylene diamine and tallow fatty acids.

Data in Table I show after four days standing at room temperature, a marked reduction in the height of bottom settlement in those formulations containing the Amide.

It has been found that in commercial wood pulp production, the presence of the Amide enhances the effectiveness of the

defoamer composition to suppress foam.

TABLE I

Formulation	Percent by weight of -			Bottom settlement height (cm.) after 4 days standing
	Talc I ^a	Talc II ^b	Calcium hydroxide	
A	15			0.50
B	15			0.05
C		15		0.55
D		15		0.05
E			15	0.80
F			15	0.10

^a E.V. Micro Talc (Charles B. Chrystal, Co., Inc., New York, New York).

^b P-275 (International Talc Co., Inc., New York, New York).

What is claimed is:

1. A method of defoaming aqueous systems used in the preparation of latex, glues, resinous materials, starches, paper and pulp, consisting essentially of the addition of from about 0.01 percent to about 0.5 percent by weight, based on the foam producing materials present in the aqueous system, of a defoamer composition comprising

- from about 1 to about 12 parts by weight of an amide which is the reaction product of (i) polyamine selected from a group consisting of ethylene diamine, butylene diamine, diethylene triamine, triethylene tetramine, hexamethylene diamine, decamethylene diamine, hydroxyethyl ethylene diamine, and 1:3-diamine-2-propanol, and (ii) fatty acid having from six to 18 carbon atoms,
- from about 5 to about 25 parts by weight of inorganic particles selected from the group consisting of talc ranging in size from about 1.4 to about 3.5 microns and calcium hydroxide, 98 percent by weight of which is capable of passing a 325 mesh screen and 100 percent by weight of which is capable of passing a 100 mesh screen,
- from about 70 to about 92 parts by weight of hydrocarbon, liquid at room temperature and atmospheric pressure,
- from about 0.5 to about 7.5 parts by weight of an oil soluble copolymer of vinyl acetate and fumaric acid esterified with a tallow alcohol, and
- from 0 to about 0.2 parts by weight of silicone oil selected from the group consisting of alkyl, aryl, alicyclic, and aralkyl, siloxane or polysiloxane.

2. The method of claim 1 wherein said amide is the reaction product of ethylene diamine and tallow fatty acids.

3. The method of claim 1 wherein said inorganic particles are talc.

4. The method of claim 1 wherein said inorganic particles are calcium hydroxide.

5. The method of claim 1 wherein said liquid hydrocarbon is mineral oil.

6. The method of claim 7 wherein the aqueous system is black liquor used in paper preparation and the defoamer composition comprises

- from about 1 to about 12 parts by weight of the reaction product of ethylene diamine and tallow fatty acids,
- from about 5 to about 25 parts by weight of talc ranging in size from about 1.4 to about 3.5 microns,
- from about 70 to about 92 parts by weight of mineral oil,
- from about 0.5 to about 7.5 parts by weight of an oil soluble copolymer of vinyl acetate and fumaric acid esterified with a tallow alcohol, and
- from 0 to about 0.2 parts by weight of silicone oil selected from the group consisting of alkyl polysiloxanes having viscosities of from about 40 to about 1,000 centistokes at 25° C.

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UNITED STATES PATENT OFFICE
CERTIFICATE OF CORRECTION

Patent No. 3,673,105 Dated June 27, 1972

Inventor(s) James H. Curtis and Fred E. Woodward

It is certified that error appears in the above-identified patent and that said Letters Patent are hereby corrected as shown below:

Column 1, line 67, change "Scott-Volumeter" to --less--. Column 4, claim 6, line 54, change "claim 7" to --claim 1--.

Signed and sealed this 22nd day of May 1973.

(SEAL)
Attest:

EDWARD M. FLETCHER, JR.
Attesting Officer

ROBERT GOTTSCHALK
Commissioner of Patents

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