United States Patent Office 3,843,558
Patented Oct. 22, 1974

3,843,558

FOAM CONTROL SUBSTANCE

Kenneth Wilfrid Farminer and Christopher Miles Brooke, Glamorgan, Wales, assignors to Dow Corning Limited, Whitehall, London, England

No Drawing. Filed June 16, 1972, Ser. No. 263,516
Claims priority, application Great Britain, July 1, 1971, 30,964/71
Int. Cl. B01d *19/04;* C11d *3/16*
U.S. Cl. 252—358 1 Claim

ABSTRACT OF THE DISCLOSURE

A foam control composition consisting of a siloxane treated sodium tripolyphosphate, its use as an additive to powder detergents for reducing excess foaming in washing machines and a method for its manufacture are disclosed.

SPECIFICATION

This invention relates to a foam control substance and to a method for the preparation of such a substance. The invention also relates to a detergent composition containing the said foam control substance.

The use of detergent compositions in powder form for the laundering of soiled fabrics is now well known and widely practiced. However, compositions of this type can give rise to excessive foaming when used in automatic washing machines. Such foaming not only interferes with the efficient laundering of the fabrics but also gives rise to overflowing and flooding of the machine.

It has been proposed to overcome this tendency to excessive foaming by modification of the detergent formulation or by inclusion in the wash water of a substance which suppresses foaming in aqueous systems. One such method which has been proposed comprises including in the detergent composition a foam suppressing substance which is encapsulated in an organic material. The known proposals for reducing excessive foaming in detergent compositions have, however, had associated with them certain disadvantages. Accordingly, there has existed a need for an improved means of overcoming the above recited problem.

We have now discovered that sodium tripolyphosphate, having on the surface a silicone antifoam, is an effective foam control agent in detergent compositions of the powdered or granular types.

Accordingly, the invention provides a foam control substance comprising powdered or granular sodium tripolyphosphate having on the surface an organopolysiloxane-containing antifoam composition.

Any organopolysiloxane-containing composition which is effective in controlling foaming in aqueous media may be employed in preparing the foam control substance of this invention. A wide variety of such compositions are known and are described, for example, the U.K. Patent Specification Nos. 689,306; 859,329; 1,020,022; 1,051,687; 1,051,688; 1,079,832 and 1,110,207. They are normally either viscous paste-like materials or aqueous emulsions. Generally, the antifoam composition comprises a diorganopolysiloxane in admixture with from about 1 to about 10 percent of its weight of a finely-divided, high-surface area filler, e.g. fume or precipitated silica or fume aluminum oxide. The diorganopolysiloxane component usually has a viscosity at 25° C. in the range from 50 to 5000 cs. However, diorganopolysiloxanes having viscosities outside this range can also be used.

The silicon-bonded organic radicals in the organopolysiloxane are usually methyl radicals. The organopolysiloxane may, however, also contain other organic radicals, for example, other alkyl radicals, alkenyl, aryl, aralkyl and alkaryl radicals such as ethyl, propyl, octyl, tetradecyl, phenyl, benzyl and 2-phenylpropyl or aminoalkyl radicals as described in U.K. Patent Specification No. 1,050,996. When radicals other than methyl are present, they preferably comprise less than about 50 percent of the total silicon-bonded radicals in the diorganopolysiloxane.

The diorganopolysiloxane employed in the antifoam composition can be terminated, for example, with hydroxyl radicals or with triorganosiloxy radicals, e.g. trimethylsiloxy, phenyldimethylsiloxy or dimethylvinylsiloxy radicals.

Preferred for use in this invention are antifoam compositions based on or containing dimethylpolysiloxanes and a high-surface area silica. Among the preferred antifoam compositions are those comprising a mixture of from 94 to 98 percent by weight of dimethylpolysiloxane and from 2 to 6 percent by weight of a silica having a surface area of at least 50 $m.^2/g$. Also included in the group of preferred antifoam compositions are those comprising a silica, a dimethylpolysiloxane and a diorganopolysiloxane which contains silicon-bonded methyl, ethyl and 2-phenylpropyl radicals, the silica being present in a proportion of from about 1 to about 10 percent by weight based on the total weight of diorganopolysiloxane.

In addition to the organopolysiloxane component, the silicone antifoam composition may contain other ingredients, for example, mold growth inhibitors, emulsifying agents and resin copolymers of $(CH_3)_3SiO_{0.5}$ and SiO_2 units as described, for example, in U.K. Patent Specification No. 1,110,107.

Preparation of the foam control substance of this invention may be readily achieved by mixing together the sodium tripolyphosphate and the organopolysiloxane-containing antifoam composition. Conveniently, the mixing is accomplished using a ribbon blender but other mixing techniques and equipment may also be used.

A preferred method of preparing the foam control substance comprises mixing anhydrous sodium tripolyphosphate with an aqueous emulsion of the organopolysiloxane-containing antifoam. The relative proportions of emulsion and sodium tripolyphosphate employed will depend on the active ingredient, i.e. organosiloxane antifoam, content of the emulsion and the proportion of antifoam composition desired in the foam control substance. The active ingredient content of the emulsion is not critical but most conveniently lies within the range from 10 to 40% based on the total weight of the emulsion. Most preferably, enough emulsion is used to just hydrate the sodium tripolyphosphate. Normally, this involves the adsorption of about 6 moles of water per mole of sodium tripolyphosphate. If desired, the proportion of water added as the emulsion may be less than that required to fully hydrate the sodium tripolyphosphate. Alternatively, the proportion of water added may be in excess of that required for hydration. In this case, however, it may be necessary to subject the resulting mixture to drying and comminuting in order to attain free-flowing properties. It will, of course, be understood that the proportion of water added should not be so high that the sodium tripolyphosphate is taken into solution.

Included within the scope of this invention, therefore, is a process for preparing a foam control composition which comprises contacting anhydrous sodium tripolyphosphate with an aqueous emulsion of an organopolysiloxane-containing antifoam composition.

In addition to convenience in preparation of the foam control substance, the use of an emulsion also appears to be advantageous inasmuch as it has been found to reduce the tendency to scum formation in use.

The proportion of organopolysilaxne-containing antifoam composition present on the surface of the sodium

tripolyphosphate may be varied within wide limits. For inclusion in detergent powder compositions, the foam control substance is desirably prepared as a free-flowing powder. In order to retain the free-flowing nature of the product, the proportion of antifoam composition should preferably not exceed about 60 percent by weight based on the weight of the sodium tripolyphosphate, that is, about 37 parts of antifoam composition per 63 parts of sodium tripolyphosphate. Most preferably, the proportion of antifoam composition present in the foam control substance is from about 10 to about 55 percent by weight based on the sodium tripolyphosphate.

The foam control substances of this invention have use as foam control agents in aqueous systems. In particular, they may be incorporated into detergent compositions of the powder type where they function to control the amount of foam generated during washing. Such detergent compositions are well known and typically comprise by weight about 10 to 25 percent of organic surface active agent, for example, a sodium salt of an alkylaryl sulphonate or of a sulphate of a straight chain primary alcohol, e.g. sodium dodecyl benzene sulphonate and sodium lauryl sulphonate, and about 25 to 50 percent of an inorganic phosphate, e.g. sodium tripolyphosphate. Other ingredients such as bleaching agents, optical brighteners, dyes and perfumes are also normally present in the detergent compositions.

The proportion of foam control substance which may be employed in the detergent compositions is not narrowly critical and may be varied between wide limits, for example, up to 50 percent by weight of the total composition. The actual proportion employed will depend, for example, on the content of antifoam composition present in the foam control substance and in the degree of foam suppression desired. We have found that the presence of up to 5 percent, preferably 1 to 4 percent, by weight of a foam control substance containing from about 10 to 35 percent by weight of antifoam composition gives satisfactory foam control with a typical commercial detergent powder.

The invention is illustrated by the following examples. In Examples 2 to 5, the detergent compositions were tested in a commercial, front-loading, automatic washing machine employing a 90 minute cycle during which the water temperature rose from 25 to 90° C. The detergent composition was employed in a proportion of 4 g. per liter of water.

EXAMPLE 1

A mixture of 5 percent by weight of a fume silica having a surface area to weight ratio of 140 m.²/g. and 95 percent by weight of a hydroxy-terminated polydimethylsiloxane (approximately 1500 cs. viscosity at 25° C.) was emulsified in water using conventional techniques. The oil-in-water type emulsion so obtained contained 30% by weight of the silica/polydimethylsiloxane mixture.

Forty parts by weight of the emulsion were then thoroughly mixed with 60 parts by weight of anhydrous, granular sodium tripolyphosphate. The resulting product was a free-flowing powder which was an effective foam control agent for foaming aqueous systems.

The powder was mixed in a proportion of 1.5% by weight with a powdered detergent composition having the following formulation:

	Percent by weight
Sodium dodecyl benzene sulphonate	12
Sodium tripolyphosphate	40
Sodium silicate	6
Carboxymethyl cellulose	1
Foam builder (coconut acid monoethanolamine)	2
Sodium perborate	20
Optical brightener and water	5
Sodium sulphate	5

The composition was added to water at a rate of 4 g. per liter and the solution agitated for 1 hour at 70° C.

For the duration of this period, no excessive foam build up took place and there was no noticeable surface scum formation.

EXAMPLE 2

An antifoam composition was prepared by mixing 6 parts by weight of fume silica with 94 parts by weight of a trimethylsiloxy end-stopped polydimethylsiloxane having a viscosity of 1000 cs. at 25° C.

Thirty parts by weight of the paste-like composition so obtained were mixed with 70 parts by weight of anhydrous, granular sodium tripolyphosphate to give a free-flowing composition. This composition was effective as a foam control agent in aqueous systems.

The foam control composition was added in a proportion of 2% by weight to the detergent composition described in Example 1. When tested in an automatic washing machine, no excessive foam build up occurred.

EXAMPLE 3

An antifoam composition was prepared by shearing for 3 hours at 175° C. a mixture of 3 parts by weight of a silica having a surface area of about 225 m.²/g. and 97 percent by weight of a polydiorganosiloxane having a viscosity at 25° C. of about 1500 cs., a methyl to silicon ratio of about 1:1, an ethyl to silicon ratio of about 0.8:1 and a 2-phenylpropyl to silicon ratio of about 0.2:1.

Thirty-five parts by weight of the paste-like composition thus obtained was mixed with 65 parts by weight of anhydrous sodium tripolyphosphate to give a free-flowing powder. The powder was an effective foam control agent for aqueous systems. When incorporated in the detergent powder described in Example 1 at a level of 4% by weight, it prevented excessive build up of foam. At lower levels of addition, the foam control agent was also effective in controlling foaming in a commercial "low-foaming" detergent powder.

EXAMPLE 4

A paste-like composition was prepared by mixing 88 parts by weight of a trimethylsiloxy-terminated polydimethylsiloxane having a viscosity of 1000 cs. at 25° C., 10 parts by weight of silica aerogel and 2 parts by weight of a resin copolymer of $(CH_3)_3SiO_{0.5}$ and SiO_2 units in which the ratio of former to latter units was in the range from 0.6/1 to 1.2/1.

The composition was emulsified in water using conventional emulsification techniques, the oil-in-water emulsion obtained contained 30 parts by weight of the composition.

Forty parts by weight of the emulsion were then thoroughly mixed with 60 parts by weight of anhydrous, granular sodium tripolyphosphate to give a free-flowing powder. The powder was effective as a foam control agent in aqueous systems.

The powder was mixed in a proportion of 1% by weight with a powdered detergent composition having the following formulation by weight:

	Percent
Alkyl lauryl sulphonate	24
Sodium tripolyphosphate	38
Sodium silicate	6
Carboxymethyl cellulose	2
Sodium sulphate	29

When the detergent powder was tested in an automatic washing machine, no excessive build up of foam occurred.

EXAMPLE 5

A foam control composition was prepared by mixing by weight 65 parts of anhydrous, granular sodium tripolyphosphate and 17.5 parts each of the paste-like antifoam compositions prepared in Examples 3 and 4. The resulting composition was a free-flowing powder.

The powder was incorporated in an amount of 1% by weight into a powdered detergent composition having the following formulation by weight:

	Percent
Sodium dodecylbenzene sulphonate	15
Sodium tripolyphosphate	40
Sodium perborate	20
Sodium silicate	6
Magnesium silicate	0.5
Sodium carbonate	5
Carboxy methyl cellulose	1
Sodium sulphate	11.5

When the detergent powder was tested in an automatic washing machine, the foam was stabilized at a height of 12 cm.

That which is claimed is:

1. A foam control substance consisting essentially of powdered or granular sodium tripolyphosphate having on the surface thereof an antifoam composition consisting essentially of a mixture of (1) dimethylpolysiloxane, (2) a diorganopolysiloxane in which the silicon-bonded organic radicals are methyl, ethyl and 2-phenylpropyl and said ethyl and 2-phenylpropyl groups comprise less than about 50 percent of the total silicon-bonded radicals in the diorganopolysiloxane, (1) and (2) being present in about equal proportions and being in the viscosity range from 50 to 5000 cs. at 25° C. and (3) from 1 to 10 percent by weight based on the weights of (1) and (2) of silica which has a surface area of at least 50 $m.^2/g.$, the mixture of (1), (2) and (3) being present in amount of from 10 to 60 percent by weight based on the weight of the tripolyphosphate.

References Cited

UNITED STATES PATENTS

3,691,091	9/1972	Koerner	252—358
3,501,408	3/1970	Gabler	252—135
3,421,940	1/1969	Wedell	134—29
3,383,327	5/1968	Sullivan	252—358
3,314,891	4/1967	Schmolka	252—135 XR
3,306,858	2/1967	Oberle	252—135 XR

FOREIGN PATENTS

525,433	5/1956	Canada	252—358

OTHER REFERENCES

"Synthetic Detergents" by A. Davidsohn et al., Leonard Hill, London 1967, p. 53.

LEON D. ROSDOL, Primary Examiner

D. L. ALBRECHT, Assistant Examiner

U.S. Cl. X.R.

252—135, 321, 539

UNITED STATES PATENT AND TRADEMARK OFFICE

CERTIFICATE OF CORRECTION

PATENT NO. : 3,843,558

DATED : October 22, 1974

INVENTOR(S) : KENNETH WILFRID FARMINER ET AL.

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

Under Rule 322 (37 CFR 1.322)

Column 2, line 31, "No. 1,110,107" should read --No. 1,110,207--.

Signed and Sealed this

twentieth Day of January 1976

[SEAL]

Attest:

RUTH C. MASON
Attesting Officer

C. MARSHALL DANN
Commissioner of Patents and Trademarks

UNITED STATES PATENT AND TRADEMARK OFFICE

CERTIFICATE OF CORRECTION

PATENT NO. : 3,843,558

DATED : October 22, 1974

INVENTOR(S) : KENNETH WILFRID FARMINER ET AL.

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

Under Rule 322 (37 CFR 1.322)

Column 2, line 31, "No. 1,110,107" should read --No. 1,110,207--.

Signed and Sealed this

twentieth Day of January 1976

[SEAL]

Attest:

RUTH C. MASON
Attesting Officer

C. MARSHALL DANN
Commissioner of Patents and Trademarks