608032

COMMONWEALTH OF AUSTRALIA

Patents Act 1952-1969

CONVENTION APPLICATION FOR A PATENT

(1) Here insert (in full) Name or Names of Applicant or Applicants, followed by Address (es).

I/We (1) WACKER-CHEMIE GMBH

of Prinzregentenstrasse 22, D-8000 Munchen 22,

Federal Republic of Germany

(2) Here insert Title of Invention.

hereby apply for the grant of a Patent for an invention entitled: (2)

ANTIFOAM AGENTS

(3) Here insert number(s) of basic application(s)

which is described in the accompanying complete specification. This application is a Convention application and is based on the application numbered (3)

P37 25 322.0

(4) Here insert Name of basic Country or Countries, and basic date or dates

for a patent or similar protection made in (4) Federal Republic of Germany on 30th July 1987

APPLICATION ACCEPTED AND AMENDMENTS ALLOWED 20.12.90

My/Our address for service is Messrs. Edwd. Waters & Sons, Patent Attorneys, 50 Queen Street, Melbourne, Victoria, Australia.

AU001541 29/07/88

DATED this 28th day of July 1988

(5) Signature (s) of Applicant (s) or Seal of Company and Signatures of its Officers as prescribed by its Articles of Association.

(5) WACKER-CHEMIE GMBH

by

Ian A. Scott

Registered Patent Attorney

To:

THE COMMISSIONER OF PATENTS.

Edwd. Waters & Sons, Melbourne.

COMMONWEALTH OF AUSTRALIA

Patents Act 1952-1969

DECLARATION IN SUPPORT OF A CONVENTION APPLICATION FOR A PATENT OR PATENT OF ADDITION

(1) Here insert (in full) Name of Company.

In support of the Convention Application made by(1) WACKER-CHEMIE GMBH

(hereinafter referred to as the applicant) for a Patent

(2) Here insert title of Invention.

for an invention entitled:(2) ANTIFOAM AGENTS

(3) Here insert full Name and Address, of Company official authorized to make declaration.

I/~~We~~(3) We, Dr. Erich Franke and Hans J. Klinger

of Wacker-Chemie GmbH Fed. Republic of Germany

Prinzregentenstrasse 22, D-8000 München 22

do solemnly and sincerely declare as follows:

1. ~~I am~~ We are authorised by the applicant for the patent to make this declaration on its behalf.

2. The basic application as defined by Section 141 of the Act was made in(4) Federal Republic of Germany on the 30th day of July 1987, by WACKER-CHEMIE GMBH

~~on the~~ ~~day of~~ ~~19~~, ~~by~~

(4) Here insert basic Country or Countries followed by date or dates and basic Applicant or Applicants.

(5) Here insert (in full) Name and Address of Actual Inventor or Inventors.

3.(5) ERNST INNERTSBERGER, Marie-Eberth-Strasse 15, D-8263 Burghausen, JAKOB SCHMIDLKOFER, Ahornweg 3, D-8261 Mehring-Od, PETER HUBER, Gluckstrasse 39, D-8263 Burghausen, WILLIBALD BURGER, Stadtplatz 95/96, D-8263 Burghausen and JOACHIM SCHULZE, Paganiniweg 14, D-8263 Burghausen, Federal Republic of Germany is/are the actual inventor of the invention and the facts upon which the applicant is entitled to make the application are as follow:

The applicant is the assignee of the said actual inventors

4. The basic application referred to in paragraph 2 of this Declaration was the first application made in a Convention country in respect of the invention the subject of the application.

DECLARED at Munich

this 4 th day of May 19 90

(6) Signature.

To: THE COMMISSIONER OF PATENTS.

Dr. Erich Franke
Head of Patent, Trademark and Licensing Dept.

Hans J. Klinger
Marketing Manager

Edwd. Waters & Sons, Melbourne.

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ANTIFOAM AGENTS

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(57) Claim

1. An antifoam agent comprising:
an essentially linear organopolysiloxane; a silicone resin consisting primarily of triorganosiloxy and $SiO_{4/2}$ units; and filler(s):
characterised in that at least part of the organopolysiloxane contains, in addition to SiC-bonded organic radicals, identical or different SiOC-bonded radicals having at least 6 carbon atoms built up from carbon and hydrogen atoms and/or from carbon, hydrogen and at least 2 oxygen atoms and optionally at least one Si atom; and
not more than 10% by weight of 2,2,4-trimethyl-1, 3-diisobutyryloxypentane based on the weight of the organopolysiloxane, silicone resin and filler.

2. An antifoam agent according to claim 1, characterised in that the essentially linear organopolysiloxane is built up from units of the formula R_2SiO, $R(R'O)SiO$, $R_3SiO_{1/2}$, $R_2(R'O)SiO_{1/2}$, $RSiO_{3/2}$, $R'OSiO_{3/2}$ and $SiO_{4/2}$, with the proviso that not more than 20% of the siloxane units are of the formulae $RSiO_{3/2}$, $R'OSiO_{3/2}$ and $SiO_{4/2}$, wherein R, R_2 and R_3 denote identical

.../2

or different monovalent or halogen substituted hydrocarbon radicals containing from 1 to 18 carbon actoms per radical, and R' denotes the identical or different SiOC-bonded organic radicals having at least 6 carbon atoms built up from carbon and hydrogen atoms and/or from carbon, hydrogen and at least 2 oxygen atoms and optionally at least one Si atom.

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Form 10

COMMONWEALTH OF AUSTRALIA

PATENTS ACT 1952-69

COMPLETE SPECIFICATION

(ORIGINAL)

This document contains the amendments made under Section 49 and is correct for printing

Class Int. Class

Application Number:
Lodged:

Complete Specification Lodged:
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Related Art :

Name of Applicant : WACKER-CHEMIE GMBH

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Actual Inventor: ERNST INNERTSBERGER, JAKOB SCHMIDLKOFER, PETER HUBER, WILLIBALD BURGER and JOACHIM SCHULZE

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50 QUEEN STREET, MELBOURNE, AUSTRALIA, 3000.

Complete Specification for the invention entitled:

ANTIFOAM AGENTS

The following statement is a full description of this invention, including the best method of performing it known to : us

Antifoam Agents

The invention relates to agents which prevent undesirable high foaming and additives which are particularly suitable for use in detergents. These antifoam agents according to the invention can be prepared on the basis of an essentially linear organopolysiloxane, a silicone resin consisting essentially of triorganosiloxy and $SiO_{4/2}$ units, and filler(s).

Foam-reducing mixtures of a polydimethylsiloxane fluid (sic), a resin consisting of trimethylsiloxy and $SiO_{4/2}$ units, and SiO_2 Aerogel are described in U.S. Patent No. 3,455,839 (published on 15th July 1969, L.A. Rauner, Dow Corning Corp.). Antifoam agents based on organopolysiloxane, filler and dispersing agent which contain substantial amounts of 2,2,4-trimethyl-1,3-diisobutyryloxy-pentane are known from European Patent-A 106,209 (laid open on 25th April, 1984, E. Innertsberger et al., Wacker-Chemie GmbH).

The object of the present invention is to provide new antifoam agents. In particular, the object of the present invention is to provide antifoam agents for detergents, the effect of which decreases only very little during the washing operation. It is furthermore an object of the present invention to provide antifoam agents which are so stable in a detergent slurry that these antifoam agents can already be added to the detergent before spray-drying of the slurry.

The present invention therefore provides an antifoam agent comprising:
an essentially linear organopolysiloxane; a silicone resin consisting primarily of triorganosiloxy and $SiO_{4/2}$ units; and filler(s);
characterised in that at least part of the organopolysiloxane contains, in addition to SiC-bonded organic radicals, identical or different SiOC-bonded radicals having at least 6 carbon atoms built up from carbon and hydrogen atoms and/or from carbon, hydrogen and at least 2 oxygen atoms and

optionally at least one Si atom; and

not more than 10% by weight of 2,2,4-trimethyl-1, 3-diisobutyryloxypentane based on the weight of the organopolysiloxane, silicone resin and filler.

organopolysiloxane containing, in addition to the SiC-bonded organic radicals, identical or different SiOC-bonded radicals built up from carbon and hydrogen atoms and/or from carbon, hydrogen and at least 2 oxygen atoms per radical and if appropriate also at least one Si atom and with in each case at least 6 carbon atoms per radical, and by the antifoam agent containing not more than 10% by weight of 2,2,4-trimethyl-1,3-diisobutyryloxypentane, based on the weight of essentially linear organopolysiloxane, silicone resin and filler.

The essentially linear organopolysiloxane of the antifoam agents according to the invention is preferably built up from units of the formulae R_2SiO, $R(R'O)SiO$, $R_3SiO_{1/2}$, $R_2(R'O)SiO_{1/2}$, $RSiO_{3/2}$, $R'OSiO_{3/2}$ and $SiO_{4/2}$, with the proviso that not more than 20% of the siloxane units are of the formulae $RSiO_{3/2}$, $R'OSiO_{3/2}$ and $SiO_{4/2}$.

The silicone resin in the antifoam agents according to the invention is preferably built up from units of the formula $R_3SiO_{1/2}$, $R_2(R'O)SiO_{1/2}$) and $SiO_{4/2}$ and to the extent of in each case not more than 5% of the siloxane units from units of the formulae R_2SiO, $R_2(RO)SiO_{1/2}$ and $R_2(HO)SiO_{1/2}$. In all the above formulae, R denotes identical or different, monovalent, if appropriate substituted hydrocarbon radicals which preferably contain 1 to 18 carbon atoms per radical, and R' denotes the SiOC-bonded radicals built up from carbon and hydrogen atoms or from carbon, hydrogen and at least 2 oxygen atoms per radical and if appropriate also at least one Si atom, and with at least 6 carbon atoms per radical.

Examples of hydrocarbon radicals R are alkyl radicals, such as the methyl, ethyl, n-propyl, isopropyl, n-butyl and sec.-butyl radical, and octadecyl radicals; alkenyl radicals, such as the vinyl radical; cycloalkyl radicals, such as the cyclohexyl radical and methylcyclohexyl radicals; aryl radicals, such as the phenyl radical, aralkyl radicals, such as the 2-phenylpropyl radical, and alkaryl radicals, such as tolyl radicals.

Examples of substituted hydrocarbon radicals R are, in particular, halogenated hydrocarbon radicals, such as the 3,3,3-trifluoropropyl radical and o-, p- and m-chlorophenyl radicals.

In particular, because of the easier accessibility, preferably at least 80% of the number of SiC-bonded radicals in the organopolysiloxanes of the antifoam agents according to the invention are methyl radicals.

The SiOC-bonded radicals R' built up from carbon, hydrogen and at least 2 oxygen atoms per radical and if appropriate also at least one Si atom and with in each case at least 6 carbon atoms per radical are preferably monovalent. However, they can also be divalent.

The SiOC-bonded radicals built up from carbon and hydrogen atoms and with at least 6 carbon atoms per radical preferably contain not more than 30 carbon atoms per radical. These radicals are furthermore preferably alkyl radicals, such as the n-hexyl, 2-ethylhexyl, lauryl, isotridecyl and 2-octyldodecyl radical, or cycloalkyl radicals, such as methylcyclohexyl radicals. However, these radicals can also be, for example, radicals built up from carbon and hydrogen atoms and with an aliphatic multiple bond, such as the oleyl radical; aryl radicals, such as the phenyl radical; aralkyl radicals, such as the benzyl radical; or alkaryl radicals, such as tolyl radicals.

In the SiOC-bonded radicals built up from carbon, hydrogen and at least 2 oxygen atoms per radical and if appropriate also at least one Si atom and with at least 6 carbon atoms per radical, at least two of the oxygen atoms are preferably ether oxygen atoms. These radicals furthermore preferably have a molecular weight of 100 to 20,000.

Examples of SiOC-bonded radicals built up from carbon, hydrogen and at least 2 oxygen atoms per radical and if appropriate also at least one Si atom and with at least 6 carbon atoms per radical are polyethylene glycols and polypropylene oxide units, it being possible for

these various units to be in blocks or randomly distributed. If these radicals are not divalent, which is in any case not preferred, the oxygen atom on the opposite end to the oxygen atom linked to an Si atom is preferably bonded to an alkyl radical, such as the methyl or n- or tert.-butyl radical. However, it can also be bonded, for example, to an acyl radical, such as the acetyl radical, or, for example, to a trimethylsilyl radical.

The essentially linear organopolysiloxanes which contain, in addition to the SiC-bonded organic radicals, identical or different SiOC-bonded radicals R' built up from carbon and hydrogen atoms and/or from carbon, hydrogen and at least 2 oxygen atoms per radical and if appropriate also at least one Si atom and with in each case at least 6 carbon atoms per radical can be prepared in a known manner or in a manner which is known per se, for example by condensation of organopolysiloxanes containing Si-bonded hydroxyl groups with alcohols of the general formula

R'OH

wherein R' has the abovementioned meaning, in the presence of acid or alkaline catalysts, such as acid-treated bentonite or methanolic potassium hydroxide.

One type of essentially linear organopolysiloxane containing SiOC-bonded organic radicals of the type defined above, ~~can ..... (sic)~~ for the preparation of the antifoam agents according to the invention can, however, also /contain/ at least two different types of organopolysiloxanes with such SiOC-bonded radicals.

The units in the - essentially linear - organopolysiloxane containing SiOC-bonded radicals of the type defined above are preferably to the extent of at least 90% of their number those of the formula R_2SiO and $R_2(R'O)SiO_{1/2}$, wherein R and R' in each case have the meaning given above for these radicals. It is furthermore preferable for such an organopolysiloxane to have an average viscosity of 50 to 500,000 $mm^2.s^{-1}$, preferably 350 to 60,000 mm^2s^{-1}, in each case measured at 25°C.

The amount of essentially linear organopolysiloxane containing, in addition to the SiC-bonded organic radicals, SiOC-bonded radicals built up from carbon and hydrogen atoms and/or from carbon, hydrogen and at least 2 oxygen atoms per radical and if appropriate also at least one Si atom and with in each case at least 6 carbon atoms per radical is preferably 2 to 95 per cent by weight, based on the total weight of all the essentially linear organopolysiloxanes of the silicon resin and fillers present in the particular antifoam agent.

The silicone resin built up essentially from triorganosiloxy and $SiO_{4/2}$ units preferably consists to the extent of at least 90% of the siloxane units of units of the formulae $R_3SiO_{1/2}$ and $SiO_{4/2}$, R having the abovementioned meaning. Because of their easier accessibility, at least 80% of the radicals R are preferably methyl groups. The silicone resins mentioned can be, for example, resins which are solid at room temperature and are built up from $(CH_3)_3SiO_{1/2}$ and $SiO_{4/2}$ units and with 0.25 to 1.25 $(CH_3)_3SiO_{1/2}$ units per $SiO_{4/2}$ unit. Because of their preparation, these preferred silicone resins can contain up to a total of 5% by weight of Si-bonded methoxy, ethoxy or hydroxyl groups. The silicone resins mentioned are as a rule not completely miscible with polydimethylsiloxanes.

~~Although it is not preferred~~, The antifoam agents according to the invention can include ~~(sic)~~ 2,2,4-trimethyl-1,3-diisobutyryloxypentane ~~is~~ present to the extent of not more than 10% by weight, preferably to the extent of not more than 5% by weight, in particular to the extent of not more than 1% by weight and particularly preferably not at all, the above percentages relating to the sum of the weights of the essentially linear organopolysiloxane, silicone resin and filler(s).

Although it is not preferred, the antifoam agents according to the invention can contain surfactants. The amount of surfactants is preferably 0 to 50 per cent by weight, based on the total weight of all the essentially

linear organopolysiloxanes, silicone resin of (sic) filler(s) and dispersing agent present in the particular antifoam agent. In this connection, it should be noted that the percentages by weight of essentially linear organopolysiloxane, silicone resin, filler(s) and dispersing agent actually present in each case and lying within the stated ranges must of course make up 100 per cent by weight.

Examples of preferred surfactants are addition products of ethylene oxide on linear or branched alkanols or arylphenols containing 8 to 20 carbon atoms per molecule, such as isotridecylpolyoxyethylene glycol ether, stearylpolyoxyethylene glycol ether, cetylpolyoxyethylene glycol ether, trimethylnonylpolyoxyethylene glycol ether and nonylphenolpolyoxyethylene glycol ether, and furthermore addition products of ethyl oxide on linear or branched monocarboxylic acids and polymers. Such addition products preferably contain 2 to 15 ethylene oxide units per molecule. Examples of particularly preferred surfactants are, in particular, polyoxyethylene glycol sorbitan esters or polyoxyethylene glycol sorbitol esters with HLB values (compare US Patent 4,076,648, published on 28th February 1978, M.R. Rosen, Union Carbide Corporation, column 4, line 51 to column 6, line 2) of 8 to 14, such as polyoxyethylene glycol sorbitan hexaoleate or polyoxyethylene sorbitol hexaoleate. Examples of preferred surfactants are, finally, also fatty acid mono-, di- and polyglycerides and sodium stearoyllactate or calcium stearoyllactate, diglycerol stearic acid esters and sorbitan monostearate, polyvinyl alcohols, partly saponified polyvinyl acetates, starch, optionally modified types of cellulose and polyacrylates.

The antifoam agents according to the invention can contain one type of surfactant. However, the antifoam agents according to the invention can also contain mixtures of at least two different types of surfactants.

The fillers in the antifoam agents according to the invention can be the same fillers which the antifoam

agents known to date could also contain. Examples of such fillers are hydrophilic and hydrophobic oxides of silicon, magnesium or zinc, these oxides preferably in each case having a surface area of at least 50 m^2/g, salts of elements of group II or III of the periodic table according to Mendeleev with an atomic number of 12 to 30 with aliphatic monobasic carboxylic acids or hydroxycarboxylic acids containing 12 to 22 carbon atoms per molecule, such as calcium stearate or calcium -12-hydroxystearate, and products which are solid at least at the particular use temperature of the antifoam agent and have been prepared by reaction of at least one mono- or polyvalent isocyanate with at least one organic compound which contains at least one hydrogen atom which is reactive towards the isocyanate group, such as the product of the reaction of naphthylene diisocyanate with cyclohexylamine. The preparation of the latter type of fillers is preferably carried out in the presence of at least one of the organopolysiloxanes used for the preparation of the antifoam agents according to the invention. Further examples of fillers in the antifoam agents according to the invention are lithium stearate, magnesium silicate and magnesium aluminium silicate. Pyrogenically produced or precipitated silicon dioxide, in particular silicon dioxide rendered hydrophobic, with a surface area of at least 50 m^2/g are (sic) particularly preferred.

The antifoam agents according to the invention can contain one type of filler. However, the antifoam agents according to the invention can also contain mixtures of at least two different types of fillers.

The amount of filler is preferably 1 to 20, in particular 2 to 8 per cent by weight, based on the total weight of all the organopolysiloxanes and filler(s) present in the particular antifoam agent. The essentially linear organopolysiloxane with SiOC-bonded radicals referred to, the silicone resin, referred to (called MQ resin below) built up essentially from triorganosiloxy and $SiO_{4/2}$ units, and any additional organopolysiloxanes

present are to be included in all the organopolysiloxanes present in the particular antifoam agent.

These additional organopolysiloxanes are, in particular, polydiorganosiloxanes which carry terminal hydroxyl and/or triorganosiloxy groups, the organo radicals of which are preferably methyl groups to the extent of at least 80%. These polydiorganosiloxanes preferably have a viscosity of 35 to 5,000 mm^2/s^{-1} at 25°C, in particular 50 to 1,000 mm^2/s^{-1} at 25°. They are used, for example, to adjust the viscosity of the antifoam agents according to the invention to a desired degree.

The viscosity of the antifoam agents according to the invention which are ready for commercial use is preferably 1,000 to 500,000, in particular 25,000 to 40,000 mm^2/s^{-1}, measured at 25°. The antifoam agents according to the invention can also contain condensation catalysts in addition to the substances mentioned, in particular LiOH, NaOH, KOH, RbOH or CsOH, KOH being preferred. Condensation catalyst(s) are preferably used in the antifoam agent according to the invention in amounts of 0 to 1%, based on the weight of all its components.

The weight ratio of essentially linear organopolysiloxanes with SiOC-bonded radicals (called SiOC organopolysiloxanes below) to silicone resins built up essentially from triorganosiloxy and $SiO_{4/2}$ units (MQ resins) in the antifoam agents according to the invention is preferably 1 : 10 to 10 : 1, in particular 1 : 5 to 5 : 1 and specifically 1 : 2 to 2 : 1 parts by weight.

The antifoam agents according to the invention can be prepared by mixing their components in any desired sequence. Preferably, the MQ resin is first dissolved in a relatively low-boiling solvent, this solution is combined with the SiOC organopolysiloxane and the mixture thus formed is freed from the solvent by distillation; if appropriate, the organopolysiloxane additionally used and finally fillers and if appropriate substances additionally used are then added.

If the antifoam agents according to the invention

are to be used as a constituent of detergents, which is their preferred field of use, they preferably contain no emulsifiers or dispersing agents. Detergents in any case contain sufficient quantities of these substances. Further addition of these substances would accelerate the dispersion of the antifoam agent in the washing liquor. This would lead, however, to a depletion of the antifoam agent at the washing liquor/air interface and thus to a drop in the action of the antifoam agent.

In addition to organopolysiloxanes and filler, the antifoam agents according to the invention can contain further substances which could also previously be used in the processing of antifoam agents based on organopolysiloxane with a foam suppressant action and filler. Examples of such additional substances are mineral oils, paraffin waxes, vegetable oils, higher alcohols, glycols and ethylene oxide-propylene oxide polymers. The antifoam agents according to the invention can also be adsorbed onto solids or absorbed into solids, which enables them to be formulated as powders. Examples of such solids are plastics, such as polyvinyl acetates, polyvinyl alcohols, starch, polyacrylates, zeolites, inorganic salts, such as phosphates, for example sodium tripolyphosphate and trisodium phosphate, and sulphates, such as sodium sulphate, or a mixture of at least two of the abovementioned substances and other similar substances.

They can also be spray-dried as such or as a mixture with suitable additives, such as pentanediol diisobutyrate, or in dispersed form with thickening agents, for example types of methylcellulose, carboxymethylcelluloses, starch or polyvinyl alcohols and polymer dispersions, for example of polyvinyl acetate. A correspondingly spray-dried powder preferably has the following composition (in per cent by weight):

40% - 70% of polyvinyl acetate, stabilized with protective colloids,

5% - 70% of polyvinyl alcohols or other thickening

agents,

3% - 40% of the antifoam agents according to the invention in bulk or in dissolved or emulsified form and

3% - 25% of antiblocking agent.

Examples of antiblocking agents are finely ground aluminium silicates, kieselguhr, calcium carbonate, precipitated silicic acid and other similar substances.

The antifoam agents according to the invention can also be processed to emulsions with the abovementioned emulsifiers and water.

The antifoam agents according to the invention can be used for preventing or combating foam, not only but in particular on or in aqueous solutions, especially as additives to detergents or one or more constituents of detergents, such as sodium tripolyphosphate or sodium perborate or a mixture of such sodium compounds, and furthermore for example, in the evaporation of alkaline waste liquors of the paper industry, the concentration of rubber latices, in cutting oil emulsions in the metalworking industry, in emulsion paints and other uses of synthetic resin dispersions, in water lacquers, in lubricants, in crude oil production, textile dyeing, including the high temperature process and jet dyeing, effluent treatment, fermentation processes, such as the preparation of antibiotics and ore flotation.

In the following examples, the activity of the antifoam agents is in each case represented by a number. This number is called the "FRA" value (FRA = abbreviation for "foam resistance area") and is determined as follows:

The amount of the antifoam agent to be investigated stated with the appropriate FRA value in the following connection is added to 200 ml of a 4 per cent strength by weight aqueous solution of sodium C_{14-15}-alkylsulphonate in an 800 ml glass beaker with a diameter of 9.5 cm and a height of 13 cm. The mixture thus obtained is foamed by 2 stirrers which run in opposite directions and have several arms each with an unbroken surface at

1,000 revolutions per minute for 1 minute so that no homogeneous liquid remains. 4 light barriers are positioned vertically along the outer wall of the glass beaker each at a distance of in each case 1.5 cm from one another. Such a device is shown in DE-OS 25 51 260 (laid open on 18th May 1977, Wacker-Chemie GmbH). When the foam collapses, the light barriers are released one after the other and a recorder automatically plots a graph showing on the abscissa the times (1 cm = 10 seconds) which elapse until the foam disappears at the top, second from top, third from top and bottom light barrier. 8 cm on the ordinate corresponds to the top light barrier. A line is obtained which runs almost vertically from the point of intersection of the abscissa and ordinate for 8 cm, then horizontally to the time of release of the second highest light barrier, drops vertically at this point in time and so on. Such a graph is also shown in DE-OS 25 51 260. The area which the staircase line thus obtained forms with the abscissa and ordinate is measured and gives the FRA value. The smaller this value, the greater the activity of the antifoam agent.

In the following examples, all the parts and percentages data relate to the weight, unless indicated otherwise.

Examples

A: Preparation of the SiOC organopolysiloxane

650 g of an alpha-omega-bis-hydroxypolydimethylsiloxane with a viscosity of 80 $mm^2.s^{-1}$ at 25°C, 220 g of 2-octyldodecanol and 4.5 g of 20% by weight methanolic KOH solution were mixed and the mixture was heated to 140°C, the water formed by the condensation being distilled off continuously. After cooling, the mixture was neutralized with dimethyldichlorosilane, 18 g of sodium hydrogencarbonate were added and the mixture was dried over sodium sulphate and finally filtered. The oil thus obtained had a viscosity of about 180 $mm^2.s^{-1}$ at 25°C. NMR measurements gave a content of 2-octyldodecyl groups in the oil of 23.5% by weight.

B: Foam suppressant base

One part by weight of the oil prepared according to A would be (sic) mixed with 2 parts by weight of a 50% strength by weight solution of MQ resin in tolnol (sic), the MQ resin consisting of trimethylsiloxy units to the extent of 40 mol % and of $SiO_{4/2}$ units to the extent of 60 mol %. Volatile constituents was (sic) distilled off from this mixture at 120°C and under a pressure of 100 to 1.6 kPa (absolute). An oil with a viscosity of 3,200 $mm^2.s^{-1}$ at 25°C was obtained. According to ^{29}Si-NMR measurements, the oil contained 23.9 mol % of trimethylsiloxy and 37 mol % of $SiO_{4/2}$ units.

Example 1:

A mixture of 89.3 parts by weight of an alpha-omega-bis-trimethylsilylpolydimethylsiloxane with a viscosity of 5,000 $mm^2.s^{-1}$, 5 parts by weight of a foam suppressant base prepared according to B, 5 parts by weight of a pyrogenically prepared highly disperse silicic acid with a BET surface area of about 300 m^2/g and 0.7 parts by weight of a 20% strength by weight methanolic KOH solution was kept at 150°C for 2 hours and, after cooling, was homogenized. An antifoam agent with a viscosity of about 25,600 $mm^2.s^{-1}$ at 25°C was obtained.

Example 2:

A mixture of 94 parts by weight of the foam suppressant base prepared according to B, 3 parts by weight of a precipitated silicic acid with a BET surface area of about 100 m^2/g and 3 parts by weight of a pyrogenically prepared silicic acid with a BET surface area of about 50 m^2/g was kept at 150°C for 2 hours and, after cooling, was homogenized.

An antifoam agent with a viscosity of 10,350 $mm^2.s^{-1}$ at 25°C was obtained.

Example 3:

Example 1 was repeated with the modification that instead of an alpha-omega-bis-trimethylsilylpolydimethylsiloxane, 89.3 parts by weight of an alpha-omega-bis-

hydropolydimethylsiloxane with a viscosity of about 6,000 mm^2/s at 25°C was used. The mass thus obtained with a viscosity of about 400,000 $mm^2.s^{-1}$ was brought to a viscosity of about 30,000 mm^2/s at 25°C with a methylsilicone oil with a viscosity of about 50 mm^2/s at 25°C (commercially obtainable from Wacker-Chemie GmbH as silicone oil AK 50).

Example 4:

A mixture of 90 parts by weight of an alpha-omega-bis-trimethylsilylpolydimethylsiloxane with a viscosity of about 350 mm^2/s at 25°C, 5 parts by weight of a pyrogenically prepared silicic acid with a BET surface area of about 300 m^2/g and 5 parts by weight of the foam suppressant base prepared according to B was kept at 150°C for 2 hours and, after cooling, was homogenized. An antifoam agent with a viscosity of 11,300 mm^2/s at 25°C was obtained.

Comparison Examples 1 to 4:

A mixture of 95 parts by weight of an alpha-omega-bis-trimethylsilylpolydimethylsiloxane with a viscosity of 350 mm^2/s (Comparison Example 1) or 5,000 mm^2/s (Comparison Example 2 and 4) or 10,000 mm^2/s (Comparison Example 3) and 5 parts by weight of a pyrogenically prepared silicic acid with a BET surface area of 200 m^2/g and, exclusively in Comparison Example 4, 0.1 parts by weight of a 20% strength by weight methanolic KOH solution would be (sic) left at 180°C for 3 hours and, after cooling, homogenized.

Action Examples:

The FRA values were in each case determined by the process described above for the examples and comparison examples given above, in each case 0.1 g of the antifoam agent being used as a 10% strength by weight solution in ethyl acetate. These FRA values are shown in the following table. In this table, FRA 1 means the FRA value determined as stated above, FRA 2 means the values determined in the first and FRA 3 the value determined in the second repetition of the test, without further

addition of antifoam agent.

Table

Example	FRA1	FRA2	FRA3
1	250	220	300
2	450	560	820
3	180	200	240
4	485	510	590
Comparison Example			
1	950	1,610	2,580
2	800	1,450	2,160
3	750	1,300	2,050
4	700	1,250	1,920

As already mentioned, the foam suppressant action is greater the smaller the FRA value of the particular agent.

Example 5:

4,000 g of a 55% strength polyvinyl acetate dispersion stabilized with protective colloid, 2,200 g of a 20% strength solution of polyvinyl alcohol with a saponification number of 140 and a viscosity of 5 mPa.s at 20°C, measured on a 4% strength aqueous solution, and 656 g of an antifoam agent prepared in accordance with Example 1, were dispersed in 3,000 g of water in a high-speed stirrer (dissolver).

The dispersion thus prepared was pulverized in a spray-drier with a discharge temperature of 80°C and the powder thus obtained (3,300 g) was mixed with 330 g of antiblocking agent (an aluminium silicate).

The FRA values of a suspension of 0.075 g of the antifoam agent prepared in this manner in 200 ml of a 4% strength by weight aqueous solution of alkyl sulphonate were:

FRA1: 1410; FRA2: 1480; FRA3: 1540.

Comparison Example 5:

Example 5 was repeated with the modification that instead of the antifoam agent prepared according to

Example 1, the same amount of the antifoam agent prepared according to Comparison Example 2 was used.

The FRA values determined in accordance with Example 5 were:

FRA1: 2020; FRA2: 2510; FRA3: 2950.

THE CLAIMS DEFINING THE INVENTION ARE AS FOLLOWS:

1. An antifoam agent comprising:
an essentially linear organopolysiloxane; a silicone resin consisting primarily of triorganosiloxy and $SiO_{4/2}$ units; and filler(s);
characterised in that at least part of the organopolysiloxane contains, in addition to SiC-bonded organic radicals, identical or different SiOC-bonded radicals having at least 6 carbon atoms built up from carbon and hydrogen atoms and/or from carbon, hydrogen and at least 2 oxygen atoms and optionally at least one Si atom; and
not more than 10% by weight of 2,2,4-trimethyl-1, 3-diisobutyryloxypentane based on the weight of the organopolysiloxane, silicone resin and filler.

2. An antifoam agent according to claim 1, characterised in that the essentially linear organopolysiloxane is built up from units of the formula R_2SiO, $R(R'O)SiO$, $R_3SiO_{1/2}$, $R_2(R'O)SiO_{1/2}$, $RSiO_{3/2}$, $R'OSiO_{3/2}$ and $SiO_{4/2}$, with the proviso that not more than 20% of the siloxane units are of the formulae $RSiO_{3/2}$, $R'OSiO_{3/2}$ and $SiO_{4/2}$, wherein R, R_2 and R_3 denote identical or different monovalent or halogen substituted hydrocarbon radicals containing from 1 to 18 carbon actoms per radical, and R' denotes the identical or different SiOC-bonded organic radicals having at least 6 carbon atoms built up from carbon and hydrogen atoms and/or from carbon, hydrogen and at least 2 oxygen atoms and optionally at least one Si atom.

3. A process for the preparation of a solid formulation antifoam agent, characterised in that the antifoam agent according to Claim 1 or 2 is spray-dried.

4. An antifoam agent substantially as hereinbefore described with reference to any one of the Examples.

5. A process for the preparation of an antifoam agent substantially as hereinbefore described with reference to any one of the Examples.

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