United States Patent Office 2,742,436
Patented Apr. 17, 1956

2,742,436

PREPARATION OF NON-DUSTING ORGANIC DETERGENT COMPOSITIONS

Russell L. Jenkins, Webster Groves, Mo., assignor to Monsanto Chemical Company, St. Louis, Mo., a corporation of Delaware

No Drawing. Application July 16, 1952, Serial No. 299,243

15 Claims. (Cl. 252—161)

The present invention relates to non-dusting synthetic organic detergents and to a novel method of preparing same.

Heretofore, numerous synthetic organic detergents have been developed as soap substitutes and while many of them have possessed advantages over soap for certain applications, they have a number of undesirable properties which impair their utility and marketability.

For example, one of these undesirable properties is the tendency of these synthetic organic detergents to form dust when they are converted into flakes, grains, agglomerates, chips or other physical forms having at least one short dimension. This is a serious disadvantage since, in addition to the undesirability from a merchandising standpoint, the very fine dust sifts from the packages in which the detergents are sold, thus producing a shortage in weight to the consumer. Moreover, the dust from the detergent causes a very objectionable irritation of the eyes and nose of those engaged in its manufacture and also the consumer who, in the case of most packaged goods, is generally the housewife. In addition, excessive dusting results in a substantial reduction in the yield of these products to say nothing of the unsightly appearance created by the accumulation of dust in adjacent parts of the plant.

Because of the complaints made by manufacturers and members of the consuming public as a result of the tendency of these products to dust, considerable effort has been made to solve this problem. Substantial progress in this direction has been made, but there is still an urgent demand in the art for an improved dedusting agent and also for a dedusting agent which will not have a deleterious effect on the lathering and detergent properties of the detergent with which it is associated.

It is, therefore, the primary object of the instant invention to provide a new synthetic organic detergent composition characterized by a substantially reduced tendency to dust as compared with the active material or the untreated detergent composition containing same.

Another object of the invention is to provide a novel non-dusting synthetic organic detergent composition which retains the lathering and detergent properties of the active material and/or the untreated detergent composition containing same.

Another object of the invention is to provide a substantially non-dusting synthetic organic detergent which does not tend to cake excessively.

A further object of the invention is to provide dedusting agents which, when associated with normally-dusting synthetic organic detergents, minimizes the loss in yield of the product which accompanies excessive dusting of the active material or the untreated detergent compositions containing same.

Other objects and advantages will become apparent to those skilled in the art as the description of the invention proceeds.

I have discovered that normally-dusting, solid, synthetic organic detergents of the type hereinafter described may be rendered substantially non-dusting by incorporating therewith about 0.5% to about 5% by weight of a phenol-ethylene oxide condensation product containing

from 1 to 25 moles of ethylene oxide per mole of phenol. These condensation products are oily liquids to waxy solids and are prepared by the method described in Patent 1,970,578 to I. G. Farbenindustrie.

The condensation products of the instant invention may be incorporated in the product in any suitable manner such as by mixing with the solid detergent; by spraying onto the solid detergent; and by adding same to the neutralized solution or slurry of the detergent and then drum drying or spray drying the resulting product.

For a more complete understanding of the present invention reference is made to the following specific examples.

EXAMPLE I

Samples of a normally-dusting detergent containing approximately 40% dodecylbenzene sodium sulfonate and 60% sodium sulfate, in the form of particles passing a 20 mesh screen, were sprayed with the following amounts of a condensate of 12 moles of ethylene oxide with 1 mole of phenol, using spraying times of 3.3, 8.0, 6.5 and 8.0 minutes, respectively. The products thus obtained were tested by the method hereinafter described to determine the dedusting action of the above condensate. The following results were obtained.

Percent Additive	Spraying Time, Minutes	Dust Value, M. A. S.
None		2,513
1.0	3.3	880
1.0	8.0	367
1.25	6.5	182
1.50	8.0	70

This example demonstrates that the phenol-ethylene oxide condensation product markedly reduces the tendency of the above detergent composition to dust; it also shows the importance of not spraying too fast.

EXAMPLE II

The salt mixture of Example I including dodecylbenzene sodium sulfonate and sodium sulfate was sprayed with 0.5% by weight of the condensate of one mole of phenol with 12 moles of ethylene oxide. This product was then evaluated from the standpoint of dusting with the following results:

Per cent additive:	Dust value M. A. S.
None	4300
0.5	1850

This example shows that even in percentages as low as 0.5% the condensation products of the instant invention greatly reduce the dust value of the detergent mixture.

The following experimental data indicate the effect of varying the phenol-ethylene oxide molar ratios on the dedusting action of the condensation product when associated with dodecylbenzene sodium sulfonate-containing mixtures of the type employed in Examples I and II.

Table I

Condensation Product (Ethylene Oxide/Phenol Molar Ratio)	Synthetic Detergent	Dust Value, M. A. S.
None	Composition Containing Approximately 40% Dodecylbenzene Sodium Sulfonate and 60% Sodium Sulfate.	4,165
6.0	do	630
8.0	do	530
10.0	do	490
12.0	do	780
14.0	do	990
16.0	do	1,150

The tests involving this series of dedusting agents were carried out in a 20-gallon rotating drum using a charge of 30 lbs. of the detergent mixture. The additives were all heated to 100° C. before application, but they were transferred to a graduated funnel for measuring and then introduced in through a tube so that the temperature of application of the dedusting agents to the detergent mixture was considerably below 100° C. The additive was introduced in a period of about 2.5 minutes and in an amount corresponding to 1.25% by weight. After the dedusting agent had been added, the rotation of the drum was continued for 2 minutes. The product thus obtained was then evaluated from the standpoint of dusting with the results indicated above.

It will be noted from Table I that while all of the given ratios were effective, the condensation product containing 10 moles of ethylene oxide per mole of phenol gave the optimum results.

Typical products of the instant invention were evaluated from the standpoint of dustiness and compared with commercially availabel products with the following results.

Table II

	Dust values M. A. S.
Synthetic detergent:	
Composition containing approximately 40% dodecylbenzene sodium sulfonate and 60% sodium sulfate	3200
Composition containing approximately 40% dodecylbenzene sodium sulfonate and 60% sodium sulfate plus 1.3% Phenol-ethylene oxide condensation product containing 12 moles of ethylene oxide per mole of phenol	250
Commercial detergent *A* based upon a soluble salt of an alkyl aryl sulfonic acid	3200
Commercial detergent *B* based upon a soluble salt of an alkyl aryl sulfonic acid	355
Commercial detergent *C* based upon a soluble salt of an alkyl aryl sulfonic acid	1465
Commercial detergent *D* based upon a soluble salt of a sulfated higher alcohol	1715
Composition containing approximately 40% dodecylbenzene sodium sulfonate and 60% sodium sulfate plus 1.25% phenol-ethylene oxide condensation product containing 12 moles of ethylene oxide per mole of phenol	275
Commercial detergent *E* based upon a soluble salt of a sulfated higher alcohol	305
Commercial detergent *F* based upon a soluble salt of a sulfated higher alcohol	690

The experimental data given in this table indicate that the dedusted products of the insatnt invention represent a substantial advance in the art over the listed commercial products.

The dedusting agents of the instant invention have the property of substantially reducing the tendency of normally-dusting solid synthetic organic detergents to dust without adversely affecting their detergent properties. As evidence of this fact, reference is made to the experimental data set forth in Table III.

Table III

	Detergency At 0.2% Concentration				Wetting Action[1] at 0.125% Concentration—Seconds Required to Wet
	Basis, Gardinol, WA		Basis, Standard Detergent Mixture Containing Approximately 40% Dodecylbenzene Sodium Sulfonate and 60% Sodium Sulfate		
	50 p. p. m.	300 p. p. m.	50 p. p. m.	300 p. p. m.	
Detergent I: Composition Containing Approximately 40% Dodecylbenzene Sodium Sulfonate and 60% Sodium Sulfate Plus 1% of Condensate of 12 Moles of Ethylene Oxide with 1 Mole of Phenol	100	90	102	100	28.1
Detergent II: Composition Containing Approximately 40% Dodecylbenzene Sodium Sulfonate and 60% Sodium Sulfate Plus 1% of Condensate of 6 Moles of Ethylene Oxide with 1 Mole of Phenol	102	90	105	100	38.8
Detergent III: Composition Containing Approximately 40% Dodecylbenzene Sodium Sulfonate and 60% Sodium Sulfate Plus 2% of Condensate of 12 Moles of Ethylene Oxide with 1 Mole of Phenol	100	90	102	100	25.3
Detergent IV: Composition Containing Approximately 40% Dodecylbenzene Sodium Sulfonate and 60% Sodium Sulfate Plus 2% of Condensate of 6 Moles of Ethylene Oxide with 1 Mole of Phenol	101	90	104	100	27.6
Detergent V: Composition Containing Approximately 40% Dodecylbenzene Sodium Sulfonate and 60% Sodium Sulfate	110	99	112	109	30.8

[1] Draves-Clarkson test for wetting out efficiency.

The standard Launderometer procedure employed herein for evaluating detergency is that described by Jay C. Harris in "Soap and Sanitary Chemicals" for August and September 1943. The "hard" water employed had a hardness of 300 p. p. m., the "soft" water a hardness of 50 p. p. m. The detergent was employed in a concentration of 0.2%. However, instead of reporting the results as per cent of soil removed, as described by Harris, the effectiveness of the detergent compositions is compared with that of Gardinol WA (sodium lorol sulfate) and a standard detergent mixture containing approximately 40% dodecylbenzene sodium sulfonate and 60% sodium sulfate, the value of each being taken as 100%.

The Draves-Clarkson test involves a determination of the time required to wet a standard skein sample, using the method described in the 1935–1936 Yearbook of the American Association of Textile Colorists, at pages 162–165.

It will be noted from the data given in Table III that the phenol-ethylene oxide condensation product had no significant effect on the detergency or wetting action of the detergent mixture.

The phenol-ethylene oxide condensation products of the instant invention also have the highly advantageous property of substantially abating the tendency of normally-dusting solid synthetic organic detergents to dust without adversely affecting their lathering properties. In contrast to the action of phenol-ethylene oxide condensation products, the alkylated derivatives thereof have the property of abating dust when associated with these detergents but are subject to the disadvantage of substantially reduc-

ing their lathering action. As evidence of these facts, reference is made to the experimental data given in the following table.

Table IV

	Lather at 0.1%, 300 p. p. m.	
	At Once	5 Min.
Composition Containing Approximately 40% Dodecylbenzene Sodium Sulfonate and 60% Sodium Sulfate	14.8	12.8
	12.7	10.4
	14.0	14.5
Composition Containing Approximately 40% Dodecylbenzene Sodium Sulfonate and 60% Sodium Sulfate Plus 1.25% by Weight of an Alkyl Phenoxy Polyethoxy Ethanol (Triton X155)	6.5	6.0
	6.3	5.5
Composition Containing Approximately 40% Dodecylbenzene Sodium Sulfonate and 60% Sodium Sulfate Plus 1.25% by Weight of Condensate of 12 Moles of Ethylene Oxide With 1 Mole of Phenol	11.5	11.0
	12.5	12.0

It will be observed from the data given in Table IV that the lathering property of the detergent mixture was substantially reduced in hard water by the presence of Triton X155 whereas the phenolethylene oxide condensate had substantially no effect on this property of the detergent mixture.

The phenol-ethylene oxide condensates of the instant invention have a further highly advantageous property which is manifested when the products of the instant invention are employed in the manufacture of detergent compositions containing sodum dichromate to inhibit corrosion of metals. Thus, whereas ethylene oxide condensates with tall oil, even when present in only the proportions required for dedusting, cause serious discoloration of such detergent compositions containing dodecylbenzene sodium sulfonate and sodium dichromate, the phenol-ethylene oxide condensates of the present invention do not discolor such products.

Finally, when incorporated in an aqueous slurry of the detergent, the phenol-ethylene oxide condensates have the desirable property of solubilizing the detergent, that is, they increase the solubility of the detergent in the aqueous medium over that of the same detergent not containing the condensate.

In the practice of the instant invention, the phenol-ethylene oxide condensates are employed in an amount varying from about 0.5% to about 5% by weight or, more specifically, within the range of about 1% to about 3% by weight and within the above limits about 1.25% to about 2% by weight is preferred. The above figures are based on the total solids content of the detergent.

When the phenol-ethylene oxide condensate is incorporated in a solution or slurry of the detergent and the product spray dried, larger amounts are required than when the condensate is mixed with the dry detergent. Roughly it requires the use of 1.25%, 2.5%, 5.0%, 7.5%, 10.0% and 12.5% of the condensate, basis total solids content, in order to obtain a dedusting action equivalent to that obtainable by incorporating the additive to the dry detergent in the proportions of 0.5%, 1%, 2%, 3%, 4% and 5%, respectively.

The phenol-ethylene oxide condensates within the scope of the present invention are those having a molar ratio of ethylene oxide to phenol of from 1:1 to about 25:1 and within the foregoing limits those products having a molar ratio range of from 6:1 to 16:1 and particularly those having a molar ratio range of 6:1 to 10:1 are preferred.

The normally-dusting, solid organic detergents within the scope of the present invention include long-chain primary alkyl hydrogen sulfates; long-chain primary alkyl sulfonates; long-chain secondary alkyl hydrogen sulfates; salts of monocarboxylic acid amides of taurine; salts of long-chain alkyl amides of sulfo-acetic acid; sulfo-succinic esters; alkyl aryl sulfonates; sulfonated, long-chain alkylated phenols; salts of petroleum sulfonates; salts of diphenyl and long-chain alkylated diphenyl mono- and polysulfonic acids; salts of the monosulfate of glycol ethers and monocarboxylic acid esters; salts of monoethers of isethionic acid and salts of monocarboxylic acid esters of isethionic acid, etc. Illustrative examples of compounds within the above classes are sodium cetyl sulfate, sodium dedecyl sulfate, sodium salt of sulfated fatty alcohols obtained by hydrogenation of cocoanut oil; sodium cetane sulfonate, sodium dodecyl sulfonate, and sodium tetradecyl sulfonate; sodium secondary dodecyl sulfate; sodium salt of the amide from oleic acid and methyl taurine, sodium dioctyl sulfo succinate; sodium dibutyl naphthalene sulfonate, sodium dodecyl benzene sulfonate and sodium hexyl naphthalene sulfonate; salts of aryl naphthene sulfonates with short and long chain substituents, salts of sulfonated aromatic mineral oil extracts in sulfur dioxide and salts of sulfonated aliphatic mineral oils; and the sodium salt of the sulfate of diethylene glycol monolaurate. The above detergents have an increased tendency to dust when mixed with inorganic salts such as sodium carbonate, borax, sodium silicate, sodium phosphates or sodium sulfates and in many instances, the last two salts are formed in the product during the process of neutralizing the sulfonated or sulfated product.

Although the above examples are directed to the treatment of the sodium salts of the organic detergents, the invention is applicable to the dedusting of other salts including those of calcium, barium, magnesium and strontium; alkaline metals; earth metals; ammonium and substituted ammonium such as ethanolamine, and mono-, di- and triamylamine; and other organic nitrogen bases. Moreover, although the above examples are directed to the processing of mixtures containing synthetic organic detergents, it is to be understood that the invention is applicable to the dedusting of the organic detergents per se.

Other materials may be added before, during or after producing the final detergent. Examples of these are soaps, coloring agents, inert fillers, builders such as sodium carbonate, ammonia and ammonium salts, perfume, fats, oils, waxes, gums, resins, germicides and deodorants, water softening agents such as pyro- and hexametaphosphoric acid compounds, sodium triphosphate, trisodium phosphate, alkali metal salts of ethylene bis (iminodiacetic) acid and other conventional water soluble alkaline metal salts. The resulting products find use in the household and in the textile, cosmetic, paper, plastic and leather industries.

The products of the instant invention may be produced in the form of flakes, grains, hollow beads, ribbons or chips, which products will have a substantially reduced tendency to dust over the original untreated material.

The specific examples have been restricted to a method of producing non-dusting detergents in which the phenol-ethylene oxide condensate is incorporated with the normally dusting synthetic solid organic detergent by spraying the condensate onto the detergent. However, as indicated earlier herein, other methods of achieving the above objective may be employed.

For example, one very practical method of producing non-dusting detergents is to incorporate the phenol-ethylene oxide condensate in a solution or slurry of the detergent and then spray dry the mixture to form a granular product. Thus, a granular non-dusting detergent may be produced by preparing a 30%–50% slurry of the following mixture:

	Parts by weight
Mixture of dodecyl and tridecyl benzene sodium sulfonates	100
Sodium sulfate (anhydrous basis)	100
Magnesium sulfate (anhydrous basis)	2
Condensate of 10 moles of ethylene oxide with 1 mole of phenol	10

and then spray drying the resulting composition under the following conditions:

Temperature of feed stock	°C.	54
Inlet gas temperature	°C.	232
Outlet gas temperature	°C.	121
Spray wheel type		Small labyrinth type
Speed of spray wheel	R. P. M.	3,000

Another method of producing a substantially non-dusting product is to add the phenol-ethylene oxide condensate to a solution or slurry of the normally-dusting, solid synthetic organic detergent and then dry the product on an evaporative surface, for example, on a drum drier, to produce the detergent in the form of flakes. Thus, a non-dusting detergent in the form of flakes may be obtained by preparing a 30% to 50% slurry of the following mixture:

	Parts by weight
Mixture of dodecyl benzene and tridecyl benzene sodium sulfonates	100
Sodium sulfate (anhydrous)	60
Sodium carbonate (anhydrous)	40
Hydrated magnesium sulfate	4
Condensate of 16 moles of ethylene oxide with 1 mole of phenol	10

and then drum drying the above composition under the following conditions:

Temperature of suspension fed to drier	70° C.
Drum spacing (aperture between rolls)	0.021 inch.
Roll temperature	121° C.
Speed of rolls	4 R. P. M.
Steam pressure	30–32 lbs./in.^{2}.
Flake thickness of dried product	0.018–0.022 inch.
Moisture in dried product	2.8%.
Apparent specific gravity	0.3586.

The dust values given earlier herein as M. A. S. (microampere seconds) were determined in the following manner.

A light source was adjusted so that the light coming through the test chamber gave an output of 100 microamperes from a photoelectric cell. A sample of the detergent was dropped into the test chamber and the dust cloud reduced the light received by the cell with a corresponding amperage output decrease. The microampere reading from the cell during the test was plotted and the area between this plot and the plot that would be obtained if no dust were present (a straight line at 100 microamperes) was a measure of the dustiness. Therefore, a high M. A. S. value indicates a dusty product.

It is to be understood that the invention is not to be limited by the specific examples hereinabove set forth as they are illustrative only. The invention is limited solely by the appended claims.

What I claim is:

1. The method of preparing a substantially non-dusting detergent, having a M. A. S. dust value of less than 1000, which comprises spray drying an aqueous composition containing a normally-dusting, solid synthetic anionic organic detergent of the class consisting of sulfates and sulfonates and about 1.25% to about 12.5% by weight, based on total solids content, of a condensate of from 1 to 25 moles of ethylene oxide with 1 mole of phenol.

2. The method of preparing a substantially non-dusting detergent, having a M. A. S. dust value of less than 1000, which comprises spray drying an aqueous composition containing a normally-dusting, solid synthetic anionic organic detergent of the class consisting of sulfates and sulfonates and about 1.25% to about 12.5% by weight, based on the total solids content, of a condensate of from 6 to 16 moles of ethylene oxide with 1 mole of phenol.

3. The method of producing a substantially non-dusting detergent having a M. A. S. dust value of less than 1000, which comprises spray drying an aqueous composition containing a water soluble salt of a higher alkyl aryl sulfonic acid and about 2.5% to about 7.0% by weight, based on total solids content, of a condensate of from 6 to 16 moles of ethylene oxide with 1 mole of phenol.

4. The method of producing a substantially non-dusting detergent having a M. A. S. dust value of less than 1000, which comprises drying on an evaporative surface an aqueous composition containing a water soluble salt of a higher alkyl aryl sulfonic acid and about 0.5% to about 5% by weight, based on total solids content, of a condensate of from 1 to 25 moles of ethylene oxide with 1 mole of phenol.

5. The method of preparing a substantially non-dusting detergent, having a M. A. S. dust value of less than 1000, which comprises drum drying an aqueous composition containing a water soluble salt of a higher alkyl aryl sulfonic acid and about 0.5% to about 5% by weight, based on total solids content, of a condensate of about 6 to 16 moles of ethylene oxide with 1 mole of phenol.

6. The method of preparing a substantially non-dusting detergent, having a M. A. S. dust value of less than 1000, which comprises producing an aqueous composition containing a water soluble salt of a higher alkyl aryl sulfonic acid and about 0.5% to about 12.5% by weight, based on total solids content, of a condensate of from 1 to 25 moles of ethylene oxide with 1 mole of phenol, and then converting said mixture to particle form with substantial drying.

7. The method of preparing a substantially non-dusting detergent, having a M. A. S. dust value of less than 1000, which comprises spraying onto the particles of a normally-dusting, solid synthetic anionic organic detergent of the class consisting of sulfates and sulfonates about 0.5% to about 5% by weight of a condensate of from 1 to 25 moles of ethylene oxide with 1 mole of phenol.

8. The method of preparing a substantially non-dusting detergent, having a M. A. S. dust value of less than 1000, which comprises spraying onto the particles of a water soluble salt of a higher alkyl aryl sulfonic acid about 1.25% to about 2.0% by weight of a condensate of from 6 to 16 moles of ethylene oxide with 1 mole of phenol.

9. A detergent composition in particle form comprising a normally-dusting, solid synthetic anionic organic detergent of the class consisting of sulfates and sulfonates and a sufficient amount of a phenol-ethylene oxide condensate to reduce the M. A. S. dust value of said detergent to less than 1000, said phenol-ethylene oxide condensate containing from 1 to 25 moles of ethylene oxide per mole of phenol.

10. A substantially non-dusting detergent composition in particle form having at least one short dimension comprising a normally-dusting, solid synthetic anionic organic detergent of the class consisting of sulfates and sulfonates and about 0.5% to about 5% by weight of a condensate of from 1 to 25 moles of ethylene oxide with 1 mole of phenol as a dedusting agent therein and sufficient to reduce the M. A. S. dust value of said detergent to less than 1000.

11. A substantially non-dusting detergent composition in particle form having at least one short dimension comprising a water soluble salt of a higher alkyl aryl sulfonic acid and about 0.5% to about 5% by weight of a condensate of from 6 to 16 moles of ethylene oxide with 1 mole of phenol as a dedusting agent therein and sufficient to reduce the M. A. S. dust value of said detergent to less than 1000.

12. A substantially non-dusting detergent composition in the form of spray dried particles comprising a water soluble salt of a higher alkyl aryl sulfonic acid and about 0.5% to about 5% by weight of a condensate of from 6 to 16 moles of ethylene oxide with 1 mole of phenol as a dedusting agent therein and sufficient to reduce the M. A. S. dust value of said detergent to less than 1000.

13. A substantially non-dusting detergent composition in the form of flakes comprising a water soluble salt of a higher alkyl aryl sulfonic acid and about 0.5% to about 5% by weight of a condensate of from 6 to 16 moles of ethylene oxide with 1 mole of phenol as a dedusting agent therein and sufficient to reduce the M. A. S. dust value of said detergent to less than 1000.

14. The method of preparing a substantially non-dusting detergent, having a M. A. S. dust value of less than 1000, which comprises producing an aqueous composition containing a normally-dusting, solid synthetic anionic organic detergent of the class consisting of sulfates and sulfonates and a sufficient amount of a condensate of from 1 to 25 moles of ethylene oxide with 1 mole of phenol to render said detergent substantially non-dusting, and then converting said mixture to particle form with substantial drying.

15. The detergent composition of claim 11 wherein the water-soluble salt of a higher alkyl aryl sulfonic acid is sodium dodecylbenzene sulfonate.

References Cited in the file of this patent

UNITED STATES PATENTS

1,970,578	Schoeller	Aug. 21, 1934
2,423,450	Heald	July 8, 1947
2,489,955	Colgate	Nov. 29, 1949