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3,002,883

DISINFECTANT COMPOSITIONS

Frederick J. Butt, Midland, and Clarence L. Moyle, Clare, Mich., assignors to The Dow Chemical Company, Midland, Mich., a corporation of Delaware
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This invention relates to the production of liquid concentrate compositions which are suitable for dilution with water for use. It is more specifically concerned with concentrate compositions of phenyl-substituted phenols.

At the present time there is a growing need for detergent-disinfectant or detergent-germicide compositions for commercial, industrial and general use which have a broad spectrum of antimicrobial activity and which have wide detergent action. Because of facility in many areas of application, there is a further demand that such detergent-germicide composition be a liquid concentrate which is readily diluted for use. Germicidal phenols of the type having a phenyl substitution with or without halogen substitution have a broad spectrum of antimicrobial activity, very low human toxicity, and maintain their activity in hard water and in the presence of organic contaminants, and furthermore, do not have the high corrosive properties possessed by germicides having labile chlorine. Such properties have made phenyl-substituted phenols of interest as a potential component in detergent-germicide compositions. However, phenyl-substituted phenols have found limited use in detergent-germicide compositions since surface active agents having suitable detergent properties have frequently been found to inactivate the phenols thereby resulting in loss of germicidal effectiveness. Many other detergents have been found to be non-compatible with the phenols thereby failing to form stable homogeneous compositions. Thus, non-ionic type surfactant and certain of the phosphates may effectively solubilize the phenols but decrease or remove the germicidal properties of the phenols. Others such as cationic type of surfactant are non-compatible and fail to form a concentrate composition. Still others such as most anionic and certain phosphate surfactants fail to form stable concentrate compositions or the compositions formed therewith form a precipitate on dilution with water. The problem of stable, homogeneous compositions is aggravated in built detergent disinfectant compositions where addition of builders to detergent disinfectant composition causes immediate precipitation of the disinfectant. This is of great consequence in the detergent industry since the alkaline builders greatly enhance or extend the detergent powers. Thus, there is a need for a stable homogeneous detergent disinfectant composition and further one which will retain the disinfectant properties when modified by the addition of builders.

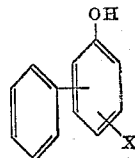
It has been discovered according to the present invention, that an aqueous stable, liquid concentrate detergent-disinfectant composition comprising a phenyl-substituted phenol which is normally sparingly soluble in water may be obtained without inactivating the phenol with respect to its germicidal properties by incorporating in the composition a water-soluble alkyl diphenyl ether sulfonate as an anionic dispersing agent whereby the phenol or phenols are maintained in solution by the presence of the anionic dispersing agent.

This invention is particularly concerned with stable aqueous, liquid concentrate compositions comprising a phenyl-substituted phenol and water-soluble alkyl diphenyl ether sulfonate. These concentrate compositions

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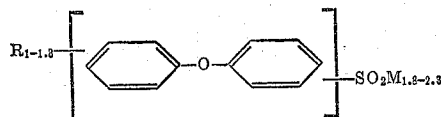
are clear and stable liquids which further form clear and stable solutions on dilution with water while maintaining the germicidal property of the phenyl-substituted phenols. These compositions are also stable to temperature changes, thus resisting precipitation of the phenolic material when subjected to freezing. Furthermore, these concentrate compositions require no added organic solvent to maintain the phenol in solution. It has further been found that when builders are added to said compositions, the resulting built detergent-disinfectant compositions are stable and retain their disinfectant and detergent properties. Thus, a detergent-disinfectant composition having the combination of properties of being a liquid, retaining germicidal effectiveness and maintaining homogeneity on subjection to freezing, on dilution with water, and on addition of builders is provided by this invention.

By the expression "phenyl-substituted phenol" or "phenylphenol" as herein employed is meant a monophenylphenol which may also have a chlorine substitution and may be represented by the formula



In this and succeeding formulas, X is hydrogen or a chloro radical. Particularly suitable are o-phenylphenol, 2-chloro-4-phenylphenol, 4-chloro-2-phenylphenol, 6-chloro-2-phenylphenol or mixtures thereof. The water-soluble salts of the phenyl-substituted phenols may also be employed when the detergent-disinfectant composition is to be in the alkaline pH range. Suitable water-soluble salts include alkali metal salts such as sodium and potassium, and ammonium salts.

The terms "water-soluble alkyl diphenyl ether sulfonate" or "water-soluble alkyl diphenyl oxide sulfonate" as herein employed are sulfonated alkyl diphenyl ether products containing alkyl substituents in amounts corresponding to from 1 to 1.3 alkyl substituents each corresponding to an alkyl radical containing from 9 to 15 carbon atoms and from 1.8 to 2.3 sulfonate groups per diphenyl ether nucleus, and may be represented by the formula



In this and succeeding formulae, R is alkyl containing from 9 to 15 carbon atoms and M is a water-soluble cation. The alkyl diphenyl ether sulfonates may be prepared as described in U.S. Patent No. 2,854,447. By "water-soluble cation" as herein employed is meant alkali metal, such as sodium and potassium, or ammonium. Suitable water-soluble alkyl diphenyl ether sulfonates to be employed in the practice of the present invention include commercially available sodium salt of disulfonated dodecyldiphenyl oxide.

A "built detergent-disinfectant" composition as herein employed is meant a detergent-disinfectant composition comprising a phenylphenol and a sulfonated alkyl diphenyl ether as above defined to which has been added alkaline builders. By "alkaline builders" is meant alkaline salts which are normally employed in detergent com-

positions to extend the detergent action. Builders include phosphates, particularly tetraprophosphates such as tetrasodium or tetrapotassium pyrophosphate, tripolyphosphates such as pentasodium tripolyphosphate, soda ash, sodium metasilicate, borax and sodium bicarbonate. When builders are employed in the detergent-disinfectant compositions they are usually added in an amount equal to the weight of the alkyl diphenyl ether sulfonate employed although two to threefold excesses of the builder may be employed.

For the preparation of these concentrate compositions, it has been found that the ratio of the alkyl diphenyl ether sulfonate to the phenol is important. Concentrate compositions may be prepared when the relative amounts are from 1 to 20 parts by weight of alkyl diphenyl ether sulfonate to 1 part by weight of the phenyl-substituted phenol. The preferred ratios depend on whether or not the detergent-disinfectant composition contains builders. When the detergent-disinfectant composition does not contain builders, the preferred ratio of alkyl diphenyl ether sulfonate to phenol is from 2.5:1 to 20:1. When the detergent-disinfectant composition contains builders or alkali, the preferred ratio of sulfonate to phenol is 1:1 to 4:1. These ratios are based on the weight of the phenyl-substituted phenol calculated as the free phenol and weight of water-soluble sulfonate.

These concentrate detergent-disinfectant compositions containing components in the ratios above set forth, may contain the germicidal phenyl-substituted phenol in a concentration of from 1 to 12 percent by weight of the total aqueous composition, although in certain compositions a higher concentration of the phenyl-substituted phenol may be employed. These compositions may then be diluted to produce germicidal treating compositions as clear homogeneous solutions containing as low as 0.0025 percent by weight of the phenyl-substituted phenol without destroying the germicidal effectiveness of the phenyl-substituted phenol. The compositions may be prepared by mixing the components together.

In a preferred method for preparing the detergent-disinfectant composition, the phenyl-substituted phenol is intimately mixed with an aqueous solution of a water-soluble alkyl diphenyl ether sulfonate. The mixing is generally carried out at room temperature with stirring until a homogeneous solution is obtained. The resulting solution may then be diluted with water, if necessary, to produce the desired composition.

The salt of the phenol is employed when a composition having relatively high alkaline pH is desired. The performed salt may be employed or a solution of alkali or ammonia may be added to the solution prepared as described above.

When the composition is a built detergent disinfectant composition, builders are added to the phenylphenol-alkyl diphenyl ether sulfonate composition prepared as above described and stirred until a homogeneous composition is obtained. Additional water may be added, if desired.

The above compositions may be modified as is known in the art without departing from the spirit thereof. Thus, diluents or solvents such as polyglycol ethers particularly propylene glycol monomethyl ether and alcohols such as isopropyl alcohol may be added. However, such diluents or solvents are not necessary according to the present invention. When compositions are to be prepared containing solvents, modifying agents, etc. such additaments may be added at any point in the manufacture according to the practice followed by those skilled in the art. If the additament is a solvent, it is conveniently employed by dissolving the phenylphenol in said solvent and adding the solution to the aqueous solution of alkyl diphenyl ether sulfonate.

The following examples illustrate the invention but are not to be construed as limiting.

EXAMPLE 1

Concentrate compositions having the following compositions were prepared:

Parts by weight

5	Composition I:	
	Component—	
	Sodium salt of disulfonated	
	dodecyldiphenyl oxide-----	2.0
10	o-Phenylphenol -----	0.5
	Water -----	97.5
	Composition II:	
	Component—	
	Sodium salt of disulfonated	
15	dodecyldiphenyl oxide-----	1.5
	o-Phenylphenol -----	0.5
	Water -----	98.0

In separate operations, each of the compositions were dispersed in microbiological culturing media to produce varying concentrations of the compositions, the modified culturing media poured into separate Petri dishes and the dishes streaked with species of bacteria or fungi and the inoculated dishes then incubated at 30° C. for 3 days. At the end of this period, the dishes were observed for microbial growth. The results with the concentrations at which inhibition of growth was observed are set forth in Table I.

Table I

30		Concentration of o-Phenylphenol at Which Inhibition of Growth Was Observed					
	Compo-	<i>Staphylo-</i>	<i>Salmo-</i>	<i>Aero-</i>	<i>Erwinia</i>	<i>Asper-</i>	<i>Penecil-</i>
	sition	<i>coccus</i>	<i>nella</i>	<i>bacter</i>	<i>caroto-</i>	<i>gillus</i>	<i>lium</i>
		<i>aureus</i>	<i>typhosa</i>	<i>aerogenes</i>	<i>vora</i>	<i>terreus</i>	<i>dig-</i>
35							<i>itatum</i>
	I-----	0.0025	0.025	0.025	0.01	0.0075	0.005
	II-----	0.0025	0.01	0.025	0.01	0.0075	0.005

EXAMPLE 2

A concentrate composition was prepared by mixing together the following:

		Grams
50	50 percent aqueous solution of the sodium salt of disulfonated dodecyldiphenyl oxide-----	60.0
45	2-chloro-4-phenylphenol -----	1.5
	Water -----	38.5

The resulting composition was a clear solution.

Two grams of the concentrate composition prepared as above described was dispersed in 1 liter of water to produce a clear dispersion having a pH of 7.

EXAMPLE 3

A concentrate composition was prepared by mixing together the following:

		Grams
60	Disodium nonyldiphenyl oxide disulfonate-----	20.0
	4-chloro-2-phenylphenol -----	1.0
	Water -----	45.5

The resulting concentrate composition was a clear solution.

Two grams of the concentrate composition was dispersed in 1 liter of water to produce a clear solution.

EXAMPLE 4

A concentration was prepared by mixing together the following:

		Weight percent
70	Disodium dodecyldiphenyl oxide disulfonate-----	41.35
	o-Phenylphenol -----	11.3
	Water -----	47.35

The resulting composition was a clear solution.

Two parts by weight of the above composition was dispersed in 100 parts by weight of water to produce a

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solution of tenfold dilution. The resulting solution was clear and homogeneous with no precipitation of the o-phenylphenol.

EXAMPLE 5

A built detergent-disinfectant concentrate having the following composition was prepared:

	Weight percent
Disodium dodecylphenyl oxide disulfonate-----	10
Dowicide 32 ¹ -----	5
Tetrapotassium pyrophosphate-----	10
Water -----	75

¹Mixture of 4- and 6-chloro-2-phenyl-phenol, product of The Dow Chemical Company.

The above composition was a clear, stable, liquid composition and formed clear and stable solutions on dilution with water.

The above composition was diluted in tap water and the phenol coefficient determined by the official AOAC method as set forth in "Official Methods of Analysis of the Association of Official Agricultural Chemists," published by the Association of Official Agricultural Chemists, Eighth Edition, 1955, pp. 87-91.

The phenol coefficients were found to be as follows:

Organism:	Phenol coefficient
<i>Staphylococcus aureus</i> -----	4.7
<i>Salmonella typhosa</i> -----	7.5

EXAMPLE 6

The following detergent-disinfectant compositions were prepared with addition of water to complete the composition:

Composition A:	Percent by weight in water
o-Phenylphenol -----	5
Disodium dodecylphenyl oxide disulfonate-----	25
Composition B:	
o-Phenylphenol -----	5
Disodium dodecylphenyl oxide disulfonate-----	15
Composition C:	
o-Phenylphenol -----	5
Disodium dodecylphenyl oxide disulfonate-----	25
Sodium hydroxide-----	1
Composition D:	
o-Phenylphenol -----	5
Disodium dodecylphenyl oxide disulfonate-----	5
Sodium hydroxide-----	1

¹Amount sufficient to form a salt with 75 percent of the o-phenylphenol.

Composition E:	
o-Phenylphenol -----	5
Disodium dodecylphenyl oxide disulfonate-----	10
Sodium hydroxide-----	2

²Amount sufficient to form a salt with 50 percent of the o-phenylphenol.

The above compositions were all clear, homogeneous solutions. These solutions were stable on cooling to 0° C. with no precipitation of the o-phenylphenol.

EXAMPLE 7

Various built detergent-disinfectant compositions were prepared and the compositions examined for clarity and stability against precipitation of solids or separation of a second phase on cooling to freezing temperatures.

Composition	Percent by Weight in Water			Appearance
	Sulfonate ¹	Dowicide 32	Tetra-potassium pyrophosphate	
1-----	3	3	3	Clear, stable.
2-----	3	3	6	Do.
3-----	3	3	9	Do.

¹Sodium salt of disulfonated dodecylphenyl oxide.

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EXAMPLE 8

Various built detergent-disinfectant compositions in which part of the phenolic germicide was present as its sodium salt were prepared and the compositions examined for clarity and stability against precipitation of solids or separation of a second phase.

Composition	Percent by Weight in Water			
	Sulfonate ¹	Dowicide 32	Tetra-potassium pyrophosphate	1 Normal NaOH
4-----	4.5	3	8	9
5-----	6	3	8	6

¹Sodium salt of disulfonated dodecylphenyl oxide.

The above compositions were clear solutions and remained clear on being subjected to freezing temperatures.

EXAMPLE 9

A built detergent-disinfectant concentrate having the following composition was prepared:

	Weight percent
Disodium dodecylphenyl oxide disulfonate-----	6
Dowicide 32-----	3
Tetrapotassium pyrophosphate-----	6
Water -----	8.5

The above composition was a clear, stable, liquid composition which further formed clear, stable solutions on dilutions with water.

The above composition was diluted with water and the phenol coefficient determined by the official AOAC method. The phenol coefficients were found to be as follows:

Organism:	Phenol coefficient
<i>Staphylococcus aureus</i> -----	3.0
<i>Salmonella typhosa</i> -----	4.5

EXAMPLE 10

Various modified built detergent-disinfectant compositions were prepared wherein the modifying additament was an organic solvent and the phenol coefficient determined. All compositions were homogeneous and stable.

Composition	Percent by Weight in Water				Phenol Coefficient Against <i>Staphylococcus aureus</i>
	Sulfonate ¹	Dowicide 32	Tetra-potassium pyrophosphate	Solvent	
8-----	3	3	8	A	2.0
9-----	3	3	8	B	2.5

¹Sodium salt of disulfonated dodecylphenyl oxide.
A=17.5 percent dipropylene glycol monomethyl ether.
B=15.5 percent isopropyl alcohol.

EXAMPLE 11

The following detergent-disinfectant compositions are prepared by intimately mixing together the components.

	Percent by weight in water
Formulation A:	
2-chloro-4-phenylphenol, sodium salt-----	3.0
Disodium dodecylphenyl oxide disulfonate----	9.0
Formulation B:	
o-Phenylphenol, sodium salt-----	3.0
Disodium dodecylphenyl oxide disulfonate----	15.0
70 Formulation C:	
p-Phenylphenol, sodium salt-----	3.0
Disodium dodecylphenyl oxide disulfonate----	4.5
Formulation D:	
o-Phenylphenol -----	0.5
Disodium nonyldiphenyl oxide disulfonate-----	1.5

Formulation E:

2-chloro-4-phenylphenol, ammonium salt.....	0.5
Diammonium dodecyldiphenyloxide disulfonate.....	2.0

Formulation F:

4-chloro-2-phenylphenol, potassium salt.....	0.5
Dipotassium dodecyldiphenyl oxide disulfonate.....	2.0

Formulation G:

o-Phenylphenol	3.0
Dipotassium dodecyldiphenyloxide disulfonate.....	6.0
Tetrapotassium pyrophosphate.....	6.0

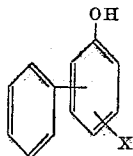
Formulation H:

Disodium dodecyldiphenyloxide disulfonate.....	37.5
o-Phenylphenol	13.3
Isopropyl alcohol.....	8.0

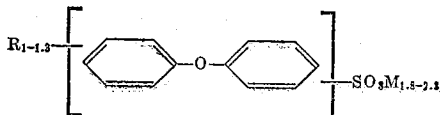
The above formulations are clear solutions which are stable on freezing and give clear solutions on addition to water.

We claim:

1. A liquid concentrate detergent-disinfectant composition comprising an aqueous solution of a phenylphenol having the formula

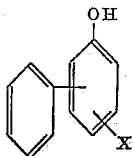


wherein X is selected from the group consisting of hydrogen and chloro and its water-soluble salts, said solution containing at least 1 percent by weight of said phenylphenol maintained in solution by an anionic dispersing agent of the general formula

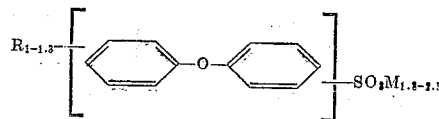


wherein R is an alkyl radical containing from 9 to 15 carbon atoms, inclusive, and M is selected from a group consisting of alkali metal and ammonium.

2. A liquid detergent-disinfectant concentrate composition comprising (1) from 1 to 12 percent by weight of a phenyl substituted phenol having the formula

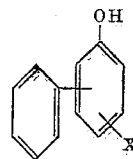


wherein X is selected from the group consisting of hydrogen and chloro and its water-soluble salts, and (2) an anionic dispersing agent of the general formula

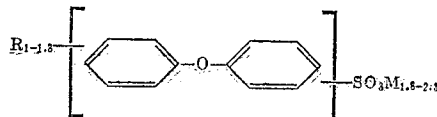


wherein R is an alkyl radical containing from 9 to 15 carbon atoms, inclusive, and M is selected from the group consisting of alkali metal and ammonium, said anionic dispersing agent being present in an amount of from 1 to 20 parts by weight for each part by weight of the phenyl substituted phenol.

3. A built liquid detergent disinfectant composition comprising an aqueous solution of (1) from 1 to 12 percent by weight of a phenyl-substituted phenol having the formula



wherein X is selected from the group consisting of hydrogen and chloro, and its water-soluble salts, and (2) an anionic dispersing agent of the general formula



wherein R is an alkyl radical containing from 9 to 15 carbon atoms, inclusive, and M is selected from the group consisting of alkali metal and ammonium, said anionic dispersing agent being present in amount of from 1 to 20 parts by weight for each part by weight of the phenyl-substituted phenol, and (3) an alkaline builder.

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UNITED STATES PATENTS

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