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ANTISEPTIC RE-CIRCULATING TOILET SUMP COMPOSITIONS

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This application is a continuation-in-part of my co-pending application, Serial No. 700,755 filed December 5, 1957 now Patent No. 2,998,390 issued August 29, 1961. This invention pertains to the disposal of human wastes in vehicles such as aircraft, busses, trailers, boats, and the like, having chemical toilets where it is either dangerous, impractical or not possible to jettison the waste material which must consequently be stored for convenient periodic disposition. Specifically, the object of this invention is to provide a fluid composition for the sump of a chemical toilet which will serve to abate odor, destroy bacteria, and which can be used to flush residual waste matter from the sides of the toilet bowl or horn and simultaneously to provide a method of rapidly producing large volumes of foam stable during flushing operations.

The commonly used facility for human waste disposal consists of a toilet seat, a bowl or horn, and a receptacle or tank beneath for the collection and storage of the deposits. It is conventional to charge the sump of such a toilet with a suitable amount of fluid for odor and bacterial control, but at the present time there is usually no provision for washing the waste matter from the bowl. This often causes objectionable appearance and odor, and if the sump itself is visible, it also usually has a displeasing appearance.

Conventional sump fluid compositions vary widely, but they are almost always water solutions or emulsions containing phenols or cresols, caustic soda or lime, chlorinated hydrocarbons, pine oil disinfectants, quaternary ammonium salts or soaps, covering or neutralizing aromatics, and/or other materials selected from a wide variety of available germicides, perfumes and emulsifying agents. All of these compositions become progressively more highly colored as wastes are added to them and are consequently not aesthetically suitable for re-circulating flushing purposes.

An improved mechanical type of toilet recently developed makes use of an electrical pump to remove a portion of the sump fluid which is filtered and pumped around the toilet bowl horn flushing residual waste matter into the sump. This development has rendered the appearance, stability, odor and germicidal qualities of the sump fluid even more important than in the past.

THE COMPOSITION IN GENERAL

As will be elaborated later on in more detail, this invention contemplates disinfectant sump compositions containing a foam agent which permits the emulsification of air in the liquid during flushing operations to give the fluid a clean, nearly white, soapy appearance. The fluid is left with a blanket of foam that not only improves its appearance but also assists in abatement of odors. The foaming agent may also have germicidal properties and act as a germicide in the composition.

This quality of the contemplated sump compositions goes a long way towards solving the many problems inherent in obtaining a satisfactory usable sump composition.

Nonetheless, this feature alone does not overcome all of the objectionable features of conventional sump compositions that so greatly limit their usefulness.

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Another object of this invention is to produce a sump composition containing a compatible mixture of several cooperating germicides for effective sterilization of waste material; the contemplated germicides are to include an antiseptic to attack bacteria and other micro organisms and one or a plurality of cooperating disinfectants for control of disease germs and the like.

Another object of this invention is to provide the sump composition with suitable odor masking and reodorizing agents.

Another object of this invention is to provide a disinfectant sump composition having the above ingredients that is stable and active even after dilution with large amounts of waste material.

The instability of quaternary ammonium compounds and formaldehyde as germicides in this invention, the additional foaming agent (if used) and the dye (if used) in storage and during dilution with waste material is a major problem. In particular, the formaldehyde can attack the quaternary ammonium compounds, dye and foaming agent causing precipitation of these constituents and rendering the composition impotent and unsightly after a period of storage or use. The desirability of using together the complementary germicides, formaldehyde and quaternary ammonium compounds, and the need to incorporate a foaming agent and a dye in the sump composition outweigh the consideration of omitting the formaldehyde if some way can be found to make the ingredients compatible and still effective. The inventor has discovered compositions which will meet these ends.

To further mask the appearance of the sump fluid, it has been found desirable to add dyes to the sump composition to impart a pleasing color to the sump fluid, masking the brown, red or amber colors thereof. In the past, dyes have been found to be either undesirable in appearance or unstable in the formaldehyde-quaternary ammonium compound-acid environment. The present invention also contemplates a suitable dye which is both compatible with the other ingredients of the compositions and still pleasing in appearance even when diluted with waste material.

Metal corrosion in mechanical toilets is also a drawback to conventional sump compositions. Due to the desirability of an acidic solution in maintaining a stable composition, corrosion in excessive amounts is the result. The corrosion is further accelerated by inherent corrosive qualities of the waste material. This invention also contemplates a sump composition which contains a corrosion inhibitor which is both compatible with the other ingredients of the composition and effective even when the composition has been diluted with large amounts of corrosive waste material.

The compositions have been provided with an acid for buffering the solution to a pH approximating 5 which will inhibit precipitation of the active ingredients prior to and during dilution with waste material and at the same time remain almost completely noncorrosive to the metals in the toilet structure including such light metals as aluminum and magnesium-aluminum alloys so prevalently used in airplane structures.

There has previously been no easy way to ascertain whether the sump fluid in a sump has received a charge of sump composition or to identify the particular sump composition charged. Either chemical analysis or compatible dye identification has been necessary. The latter method proves unsatisfactory when the fluid is discolored by waste material and the former obviously is too long and complicated to be of practical use. The presence of foam can reveal that a foaming agent has been added but goes no further in identification of the sump composition in the sump fluids. A normally colorless fluorescent

indicator dye which is compatible with a masking dye is provided so that the presence of the sump composition may be detected by exposure to an ultra-violet light source.

It is to be noted that the sump compositions as outlined above will economically and effectively handle large amounts of waste material and are satisfactorily usable in both recirculating and sump type toilets.

METHOD OF USE FOR RE-CIRCULATION TYPE TOILETS

The re-circulating pump obviously produces a greater than atmospheric pressure on a fluid that passes through it. It follows therefore that any air entrained by or dissolved in the liquid is also subjected to this pressure. When the fluid emerges from the pump, this pressure is quickly relieved. This sudden relief causes the entrained or dissolved air to expand rapidly thus producing large volumes of foam. It is necessary that large volumes of foam be rapidly produced upon ejection into the toilet bowl or horn so that a non-objectionable appearance is maintained at least for the period of the flushing operation. In aircraft, as will be obvious, the output of the pump must be sufficient to maintain proper circulation of the fluid at all cabin conditions as well as at sea level. Present standard operating equipment maintains a pumping pressure of about 12 p.s.i. which is normally sufficient to cause adequate foaming for the purposes contemplated, without the necessity of adding bleed air to assist in the foaming.

However, bleed air may be used additionally whenever desired by addition of same to the intake of the pump or by entrapment during the flushing cycle.

QUATERNARY AMINES

Several quaternary amines have been found suitable for the purposes of this invention. The foremost of these are the 1-(2-hydroxyethyl)-2-n-alkyl-1-benzyl-2-imidazolinium chlorides. Preferably a mixture of these quaternary amines is used wherein the n-alkyl groups are C_{11} , C_{13} , C_{15} , and C_{17} hydrocarbon chains. A commercial preparation of National Aluminate Corporation, Chicago, Illinois, known as "Nalquat G-8-11" (one of the aforementioned imidazolinium chlorides in which the n-alkyl group is derived from stripped coco fatty acids. Coco fatty acids contain the following acids approximately: 3 to 9% oleic, 1 to 3% linoleic, 1/2 to 1 1/2% capric, 45 to 60% lauric, 17 to 27% myristic, 7 to 15% palmitic, and 1 to 5% stearic) is such a mixture sixty percent in isopropanol solution.

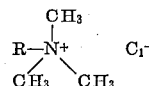
Similarly, "Nalquat G-8-12" (one of the aforementioned imidazolinium chlorides in which the n-alkyl group is derived from approximately 39 to 54% linoleic and 46 to 61% oleic acids) and "Nalquat B-8-13" (one of the aforementioned imidazolinium chlorides in which the n-alkyl group is derived from oleic acid) may also be used although "Nalquat G-8-11" is preferable due to its stability in the solution.

"Nalquat G-8-12" is a mixture sixty percent in isopropanol solution of the above quaternary imidazolinium salts in which the n-alkyl groups are heptadecenyl or heptadecadienyl hydrocarbon chains.

"Nalquat G-8-13" is a sixty percent in isopropanol solution of the above quaternary imidazolinium salt in which the n-alkyl group is the heptadecenyl hydrocarbon chain.

"Soya trimethyl ammonium chloride" marketed 50% in isopropanol solution as "Arquad S-50" by Armour & Company, Chicago, Illinois, is another preferred quaternary amine. It may be used alone in the composition or more preferably to supplement another quaternary amine such as the previously described "Nalquat G-8-11" for more thorough germicidal action, since no one germicide is equally effective against all types of bac-

teria. This is actually a mixture of amines having the formula



where R is mainly heptadecenyl and heptadecadienyl hydrocarbon chains and also with some heptadecatrienyl and saturated chains such as heptadecyl as R because the amines are derived from soya oil fatty acids.

FOAMING AGENTS

The quaternary amines employed in the composition which are discussed above also are significant foaming agents in a water solution. However, in general they are insufficient alone in the small quantities required for germicidal action to produce the desired foaming effect in a sump composition for re-circulating toilets. Besides being too weak initially for this purpose, aging and dilution with waste material further emasculates their foaming capacity.

However, it has been found that a certain concentration of a mixture of several quaternary amines in amounts necessary for proper germicidal action impart sufficient foaming action to the composition without the necessity of adding supplemental foaming agents. The amines previously identified by the trade names "Nalquat G-8-11" and "Arquad S-50" added together in appropriate quantities to the composition comprise this mixture.

It has been found that addition of a mixture of 1-(2-hydroxyethyl)-2-heptadecenyl-2-imidazoline and 1-(2-hydroxyethyl)-2-heptadecadienyl-2-imidazoline in appropriate proportion to the compositions will impart the desirable foaming characteristics for use in a re-circulating toilet regardless of the quaternary amine or amines employed. Preferably, a mixture of these two amines in sixty percent weight concentration in isopropanol marketed as "Nalcamine G-12" (one of the aforementioned imidazolines in which the alkyl group is derived from approximately 39 to 54% linoleic and 46 to 61% oleic acids) by National Aluminate Corporation, Chicago, Illinois is used in the composition. The amount of foaming agent must be carefully proportioned to give sufficient foam throughout the dilution cycle yet to avoid too much foam formation during circulation and flushing.

STABILIZERS

Acetic and lactic acids have been discovered to be advantageous and very useful as stabilizers for the composition, preventing the gradual loss of foaming ability upon dilution by waste material and aging of the composition. The acid further prevents reaction of the formaldehyde with the quaternary amines, a serious latent instability of the composition. Lactic acid is preferred due to its complete lack of obtrusive odor and extremely slight corrosiveness.

Cashmere in sufficient quantities also adds to the stability of the composition.

CORROSION INHIBITOR

Phosphate ion added to the composition, preferably as a basic salt, such as di-sodium phosphate dedecahydrate has been found to inhibit corrosion of aluminum alloys and steel and to form phosphate protective coatings on the surfaces thereof even after dilution of the concentrate with large quantities of waste material. Thus any corrosiveness of the stabilizing acid of the composition or the waste material is offset.

DYES

Although not a critical factor, it has been found advantageous to the appearance of the foaming composition to add a compatible organic dye thereto. Two dyes have been found particularly suitable, namely, Methyl Violet

and Alizarin Blue Sapphire G both of which are well known in the art.

INDICATOR DYES

The composition may contain a colorless indicator dye visible only under ultraviolet light for purposes of identification of the sump composition when in use in a re-circulating toilet. Presence of the dye indicates presence of a charge of sump composition in the sump fluid. Beta Methyl Umbelliferone and Uranine (soluble fluorescein) have been found satisfactory and compatible in the composition. The former dye fluoresces white under ultraviolet light and the latter yellowish white.

MASKING AGENT

To mask or cover up the unpleasant sump odors an aromatic material such as musk is added to the composition. The cashmere in the composition also serves this purpose.

For use in aircraft, it is essential that the weight of the sanitary facilities be minimized. The volume of the sump fluid must be as small as possible and extra tanks such as separate reservoirs for flushing liquid must be eliminated.

SUMMARY

In accordance with this invention, the sump acts as both waste storage and flushing fluid reservoir and sump fluid serves to control waste odor, bacteria and appearance, as well as to operate acceptably for flushing purposes. In the proposed disinfectant sump compositions, the combination of agents for odor, bacterial control and for producing emulsified gaseous or air foam for control of the appearance makes adequate flushing and sanitation practical with a minimum of equipment and waste.

Example I

A preferred formulation for the flushable toilet composition is:

To 150 ml. of water add and dissolve:

Nalquat G-8-12	g--	50
Acetic acid	ml--	50
Formaldehyde 40%	ml--	150
Oil of cashmere	ml--	4
Oil of musk	ml--	2
Nalcamine G-12	g--	5

This charge is a little less than one pint formulated for addition to three gallons of sump water in an aircraft flushable toilet and is capable of dilution to at least twelve gallons with waste material without losing its foaming, germicidal and odor masking properties.

Observations were made of the sump composition in actual use in an aircraft re-circulating toilet mock-up during six days of operation. The sump fluid was found to be only mildly corrosive to metal and non-injurious to neoprene.

Bacteriological testing of the fluid after six days of operation showed no bacterial growth. Appearance and odor of the re-circulating fluid at the end of the test was satisfactory.

When pumped through the filter from the sump, foam is generated in the centrifugal pump and if insufficient amount is produced by cavitation in the pump or if the fluid does not contain enough dissolved gas for adequate foaming, bleed air is admitted into the suction line of the pump. The pressure drop across the pump has been found to be the most significant factor in the amount of foam produced and may be varied accordingly to regulate to some degree the amount of foam produced. The foamed liquid from the pump circulated around the bowl or horn has a white or very light creamy soapy appearance.

The quaternary ammonium salt in the composition must be at least 20 g. to be effective. Any increase in concentration over 70 g. does not appear to add to the germicidal effect of the composition.

The formaldehyde concentration may be varied from 20 ml. at which concentration it is faintly discernable, to about 200 ml. Musk concentration should not be over 3 ml. as it imparts a slightly unpleasant odor to the composition above this concentration. Oil of cashmere may be used in a quantity of 1 ml. to quantities as high as 6 ml. with satisfactory results. Its desirability is mainly due to its increase of foam stability and also to its reodorizing effect on the fluid.

Nalquat G-8-12 may vary from 20 g. to 70 g. depending upon the amount of foaming capacity desirable (this would vary with the type of toilet).

Acetic acid concentrations over 70 ml. are undesirable due to the increasingly unpleasant sour odor imparted to the sump fluid. For proper stability of the composition the amount should not fall below 20 ml.

The pint concentrate for the three gallon sump charge in the toilet, suitable for dilution to twelve gallons with waste matter should accordingly contain approximately:

Water	ml--	20-250
Nalquat G-8-12	g--	20-70
Acetic acid	g--	20-70
Formaldehyde 40%	ml--	20-200
Oil of cashmere	ml--	1-6
Oil of musk	ml--	0-3
Nalcamine G-12	g--	2-10

Example II

To 175 ml. of water add and dissolve:

Nalquat G-8-12	g--	50
Lactic acid 44%	ml--	25
Formaldehyde 40%	ml--	100
Oil of cashmere	ml--	2
Oil of musk	ml--	2
Nalcamine G-12	g--	5
Uranine	g--	0.10

This pint charge is formulated for addition to three gallons of sump water in a re-circulating toilet and is capable of dilution to at least twelve gallons with waste material.

This formulation varies from that of Example I only by the substitution of lactic acid for acetic acid as stabilizer and a decrease in the amount of cashmere and formaldehyde. Uranine, a colorless fluorescing substance was also added for identification purposes.

The charge was tested in a mock-up aircraft toilet by a week of actual use after dilution of the concentrate with three gallons of water. Users recorded the odor to be mild to imperceptible and the foamy appearance to be satisfactory.

Bacteria culture tests of samples of the sump fluids were all negative, all samples considered sterile. Samples were made directly of the flushing stream and the spray therefrom.

The sump fluid when viewed under ultraviolet light fluoresced strongly.

The essential purpose of the lactic acid being to make the sump fluid acidic, the quantity may vary in the composition from 10 to 50 ml. of 44% water solution of lactic acid. Corrosive action about the same as for tap water was experienced during a two week test of the concentrate and the used sump fluid on various metals used in toilet construction.

Example III

To 175 ml. of water add and dissolve:

Nalquat G-8-12	g--	50
Lactic acid 44%	ml--	25
Formaldehyde 40%	ml--	50
Oil of cashmere	ml--	4
Oil of musk	ml--	2
Nalcamine G-12	g--	5
Alizarin Blue Sapphire G (0.5% H ₂ O solution)	ml--	18
Beta methyl umbelliferone (1% methanol solution)	ml--	9

This formulation is identical to that of Example II except for variation of the proportion of formaldehyde and cashmere to give a more pleasing odor to the sump fluid. The germicidal properties of this charge of sump composition are still adequate to keep the sump fluids sterile in extended operation.

Beta methyl umbelliferone has been substituted for uranine as a fluorescing dye and Alizarin Blue Sapphire G dye has been added to give the sump fluid a blue cast for more pleasing appearance for flushing aluminum toilet bowls found in commercial aircraft.

Example IV

To 250 ml. of water add and dissolve:

Nalquat G-8-11	g	50
Arquad S-50	g	55
Lactic acid 44%	ml	25
Formaldehyde 40%	ml	50
Oil of cashmere	ml	4
Oil of musk	ml	2
Methyl Violet	g	0.095
Disodium phosphate dodecahydrate	g	45

The mixture is diluted to approximately a pint charge. Tests in a re-circulating toilet were carried out with addition of two ounces of the concentrate to four gallons of sump water instead of the usual 16 ounce charge of concentrate. After 21 uses of the toilet over the period of one day no breakdown in odor control was observed, indicating bacteriostasis since, when bacterial growth occurs, the odor control rapidly deteriorates. One day duration tests with four ounce charges achieved similar results. Appearance during flushing was indicated as satisfactory by users. While less foam was obtained by this small charge, the fluid coloration by the blue dye supplemented the foam to give an appearance pleasing to the user.

In the usual 16 oz. charge, foaming action is comparable to that found in tests carried at for Examples I-III.

Corrosion tests of the concentrate and sump fluid diluted with waste material on various metals including aluminum alloys and various steels shows much less corrosion occurs than with ordinary tap water.

Accelerated stability tests of the concentrate shows it to be stable for at least 1 and 1/3 years of storage at normal temperatures. Observations show no precipitation or insolubility and good color stability over this period.

In this formulation no additional foaming agent is required due to the foaming ability of Arquad S-50 and Nalquat G-8-11 even at these low concentrations. Although the amounts of these quaternary amines may be increased as desired, they should not be proportionately lower than 20 g. Nalquat G-8-11 or 25 g. Arquad S-50 for adequate bacteriostasis upon dilution with waste material in the sump.

Disodium phosphate may be omitted if desired since the treated sump fluids are only very mildly corrosive without its addition. It may be added up to as much as its solubility in the concentrate.

Due to the potency of this formulation and thus its consequential greater dilutability it is uniquely suitable as an economical all purpose concentrate useable in both the sump type and re-circulating type toilets, particularly when there are shorter periods between cleaning and servicing and thus no great safety margin is required.

It should be noted that the variations in the concentrations of the various ingredients discussed in Examples I and II apply to the concentration of the same ingredients in the remaining examples.

It is to be understood that the foregoing description is by way of illustration, and that changes, omissions, additions, substitutions and/or modifications may be made within the scope of the claims without departing from the spirit of the invention.

I claim:

1. An antiseptic sump composition for use in recirculating toilets for dilution with sump water in a ratio of not less than 1/4 part composition to 24 parts sump water which composition comprises:

- (a) from about 20 to 250 ml. water
 - (b) from about 20 to 70 g. of an isopropanol solution of a quaternary amine, said amine being selected from the group consisting of
 - (1) 60% isopropanol solution of 1-(2-hydroxyethyl) 2-n-alkyl-1 benzyl-2-imidazolinium chloride in which the alkyl group is derived from an acid selected from the group consisting of
 - (A) 100% oleic acid
 - (B) 39 to 54% linoleic acid and 46 to 61% oleic acid mixture
 - (C) stripped coco fatty acids
 - (2) 50% isopropanol solution of soya trimethyl ammonium chloride
 - (c) an acid selected from the group consisting of acetic and lactic in an amount to buffer said composition to a pH of approximately 5
 - (d) from about 20 ml. to 200 ml. of 40% aqueous formaldehyde solution
 - (e) from 0 to a color masking percentage of a masking dye selected from the group consisting of Methyl Violet and Alizarin Blue Sapphire G.
 - (f) from 0 to a foam stabilizing amount during circulation of said sump solution of a foaming agent consisting of about a 60% isopropanol solution containing a mixture of
 - (1) 39 to 54% of 1-(2-hydroxyethyl)-2-heptadecenyl-2-imidazoline and
 - (2) 46 to 61% of 1-(2-hydroxyethyl)-2-heptadecadienyl-2-imidazoline
 - (g) from 0 to an indicating amount of an indicating dye selected from the group consisting of betal methyl umbelliferone and soluble fluorescein
 - (h) from 0 to its solubility point in the concentrated solution of disodium phosphate, and
 - (i) from 0 to an odor masking percentage of a odor masking agent selected from the group consisting of oil of cashmere and oil of musk.
2. The antiseptic sump composition of claim 1 in which:
- (a) 10 to 50 ml. of 44% water solution of said lactic acid is present.

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JULIUS GREENWALD, *Primary Examiner.*