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[21] Appl. No.	610,477	3,125,487	3/1964	Hutchinson	424/170 X
[22] Filed	Jan. 20, 1967	3,288,824	11/1966	Mahler et al.	424/365 X
[45] Patented	Nov. 30, 1971	3,335,053	8/1967	Weitzel	424/365 X
[73] Assignee	Wyandotte Chemicals Corporation Wyandotte, Mich.	3,341,465	9/1967	Kaufman et al.	424/365 X
		3,036,118	5/1962	Jackson et al.	260/615 X

FOREIGN PATENTS

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[54] FLOATING BATH OIL COMPOSITION 8 Claims, No Drawings	
[52] U.S. Cl.	424/365
[51] Int. Cl.	A61k 7/00
[50] Field of Search	424/78, 170, 341, 365

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[56]	References Cited	
	UNITED STATES PATENTS	
2,674,619	4/1954	Lundsted
		260/485

ABSTRACT: A floating bath oil composition is prepared from an emollient oil, a surfactant compound insoluble in the emollient oil and an ester solvent for the surfactant compound. The composition forms a continuous, stable film on the bath water surface and has rinsibility properties.

FLOATING BATH OIL COMPOSITION

This invention relates to a spreading bath oil. In a more particular aspect, this invention relates to a floating bath oil in the form of a clear nonemulsified composition.

The practice of anointing the body with oils is known to have been practiced by the Egyptians. Thus, the first bath oil is probably more than 5,000 years old. Cosmetic products for the bath, other than soap, have, however, only achieved real popularity in the United States in recent years. True bath oils, which are the subject of this invention, did not appear on the American market until about 8 years ago, and have steadily gained in importance ever since.

Bath oils can be divided into the following types: Spreading bath oils; dispersible bath oils; highly perfumed bath oils; soluble bath oils; foaming bath oils; and germicidal bath oils. The first three can be considered true bath oils, while the latter three are aqueous preparations containing solubilized oil.

Spreading bath oils, or the floating type of bath oil, have won exceptional popularity because they not only may be formulated to give a pleasant scent to the bath, but may also help prevent skin dryness through spreading a thin layer of oil over the body. The oil itself also has an emollient effect on the skin of the bather.

A typical floating bath oil formula consists primarily of an oil, or a mixture of oils, a scenting agent that is compatible with these particular oils, and a suitable spreading agent that will aid in the formation of a nonmolecular layer of oil on the surface of the bath water.

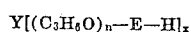
Many floating bath oils have certain objectionable features due to their lack of good spreading. For example, when added to the bath water, they often tend to form unattractive spots on the surface, instead of spreading evenly on the water. Besides presenting a disagreeable appearance, the oil reaches the body in patches, instead of in a continuous film. Moreover, when the tub is emptied, an oily scum is usually left behind, which can be removed only by scouring.

The primary esthetic requirement in a bath oil, per se, is that it be a sparkling clear solution. Therefore, solubility of the surfactant or spreading agent in the emollient oil, or other bath oil components, and the related spreadability of the mixture are extremely important. It has been noted that some prior art bath oil compositions are not clear. This is particularly so at higher than room temperatures. Further, these compositions spread slowly at elevated temperatures and upon cooling, as the bath temperature drops, exhibit a tendency to break up in unsightly droplets.

It is, therefore, a specific object of this invention to provide a novel bath oil composition which spreads rapidly and evenly in warm water. Another object is to provide a bath oil composition which will form a continuous film and thereby coat the body in an even manner.

A further object of this invention is to provide a bath oil composition which will not form unattractive spots on the bath water surface. A still further object is to provide a bath oil composition which will not create a greasy impression on the skin. Still another object of this invention is to provide a composition exhibiting excellent and enhanced rinsibility, thereby insuring that an oily scum will not be left behind when the tub is emptied.

These and the other objects of this invention are accomplished by providing a floating bath oil composition which comprises: (1) an emollient oil, (2) a surfactant compound insoluble in said emollient oil and having the formula



where Y is the residue of an organic compound containing x active hydrogen atoms; E is a polyoxyalkylene chain wherein the oxygen/carbon atom ratio is at least 0.5, and E constitutes from about 3 to about 18 percent, by weight, of the surfactant compound; x is an integer from 1 through 4; and n is an integer such that the molecular weight of the compound, exclusive of E , is from about 1,500 to 3,000 when x is 1, 3,000 to 6,000 when x is 2, 3,500 to 9,000 when x is 3, and 4,000 to 12,000

when x is 4, and (3) an ester solvent for said surfactant compound. The significance of each component of the above composition will be discussed in detail hereinafter.

Emollient oils are well-known compounds in the art and are generally defined by their physical properties to distinguish them from fats and waxes. Thus, these properties are:

1. They are liquid, have low volatility at room temperature, and are insoluble in water.

2. They feel oily or greasy when rubbed between the fingers.

3. They spread easily on the skin and leave a water-repellent or hydrophobic film on the surface.

4. They can be emulsified with water in the presence of a suitable emulsifier to form a system in which the water is continuous or is dispersed as small droplets.

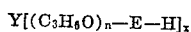
5. The oils have valuable solvent properties and can thus be used as vehicles, either in the emulsified or the unemulsified state, to bring organic compounds with remedial or other desirable effects into contact with the skin.

6. The oils are described as emollients and their action on the skin is termed emollience. This action was until recently understood to mean skin-softening and lubricating, nourishing and conditioning, but is now understood to involve the prevention or relief of skin dryness by conservation of the water content of the skin, thus maintaining the suppleness of normal skin under adverse conditions such as cold and low atmospheric humidity, or restoring resilience to skin which has become inflexible as a result of drying. The water-insoluble, water-repellent substances now under discussion are all understood to function as emollients by forming continuous occluded films when rubbed on the skin, thus reducing the rate of loss of moisture from the skin. Emollient action is thus something more than lubrication of the skin arising from oiliness or greasiness.

It is to be especially noted as fundamental to the practice of this invention, that the surfactant component of the floating bath oil composition is not soluble in the emollient oil. Thus, while, as stated in property (5) above, emollient oils have solvent properties for many organic compounds, those emollient oils useful in the practice of this invention are not solvents for the surfactant compound. Therefore, wherever the term "emollient oil" is used herein, it is used in this context. Examples of such emollient oils are mineral oil, hydrocarbon oils, while oil, olive oil, peanut oil, silicone oils and the like. It is to be understood that mixtures of these oils may also be used in the practice of this invention. Mineral oil is the preferred emollient oil.

The esters useful in the practice of this invention are those esters in which the surfactant component described above is soluble. These esters may be naturally occurring compounds of animal or vegetable origin, or they may be prepared synthetically from a C_1-C_3 short chain alcohol, and the alkylene oxide adducts thereof, and a C_8-C_{22} long chain fatty acid. Alternatively the ester may be prepared from a C_8-C_{22} long chain alcohol, and the alkylene oxide adducts thereof, and a C_1-C_3 short chain acid. It is also within the contemplation of this invention that one of the above esters may be reacted with an alkylene oxide to prepare alkylene oxide adducts of these esters. The sole criterion is that the surfactant described above must be soluble in the ester compound utilized. Such compounds are very well known in the art and are commercially available. It is also to be noted that certain esters themselves possess emollient properties. Because they do possess this property, they are not, however, to be confused with the emollient oils of this invention, discussed above. Another advantageous property is that many esters aid in reducing the greasy feeling of the emollient oils. Examples of these esters are methyl palmitate, methyl myristate, dibutyl adipate, butyl sebacate, isopropyl laurate, isopropyl palmitate, isopropyl myristate, ethylene glycol dipelargonate, propylene glycol dipelargonate, polypropylene glycol oleate, and the like compounds. Isopropyl myristate and palmitate are especially preferred for use in preparing the bath oil composition.

The surfactant compound described above and having the generic formula



is the component which causes the floating bath oil composition of this invention to be novel and to possess such unexpected and outstanding properties. As mentioned above, the use of surfactants as spreading agents in bath oil preparations is old in the art. Further, compounds of the type defined by the above generic formula are well known in the art. Such compounds and their method of preparation are described in detail in U.S. Pat. No. 2,674,619. However, it has been discovered that the use of the specific surfactants as defined by the limiting values placed upon Y , n , E and x in the above generic formula is necessary in preparing the floating bath oil composition of this invention. Accordingly, it is critical that the surfactant have the specified composition.

Initially, it should be pointed out that the surfactant compounds utilized in the prior art floating bath oils are oil-loving compounds. Thus, they are predominantly hydrophobic in nature and are oil soluble. It was very surprising and unexpected to find that the surfactant compounds of this invention, which are not oil soluble, would produce such enhanced and outstanding properties. This is even more surprising when one considers that with the narrow limits described above, the surfactant compounds of this invention are neither oil soluble nor water soluble. They are, thus, a very small unique class of surfactant compounds which one would not even expect to be of value in a floating bath oil composition. It is postulated that their outstanding contribution to the floating bath oil composition is due to a concentration of their surfactant and spreading properties at the interface of the bath water and the emollient oil.

The surfactant compound of this invention may be prepared according to conventional oxyalkylation procedures well known in the surfactant art and as described in U.S. Pat. No. 2,674,619. Thus, an organic reactive hydrogen compound and an anhydrous alkaline hydroxide catalyst are placed in a reaction flask equipped with a mechanical stirrer, reflux condenser, thermometer, and alkylene oxide feed inlet. The flask is purged with nitrogen to remove air, heated to about 120° C. with stirring, and the alkylene oxide introduced through the feed inlet until the reaction product possess the calculated molecular weight. Propylene oxide and ethylene oxide may be reacted sequentially in that order or the hydrophobic propylene oxide adduct may be recovered and later reacted with ethylene oxide in the manner described above. It is within the contemplation of this invention, but not preferred, to substitute for propylene oxide a higher alkylene oxide, such as butylene oxide or a mixture of alkylene oxides, possessing equivalent hydrophobic characteristics.

The limiting values placed upon Y , n , E and x in the above generic formula are determinative of the surfactant compounds which may be utilized in the practice of this invention. Y is an organic active hydrogen compound. The terminology 'organic active hydrogen compound' is well known in the art and is defined and exemplified in numerous patents, such as U.S. Pat. No. 2,674,619. Since x in the generic formula is an integer from 1 through 4, typical examples of Y are methanol, ethanol, ethylene glycol, propylene glycol, glycerol, trimethylolpropane, pentaerythritol, methylamine, propylamine, ethylene diamine, propanoic acid, and the like organic reactive hydrogen compounds.

The term " n " in the generic formula is an integer such that the molecular weight of the surfactant compound, exclusive of E , is from about 1,500 to 3,000, preferably 2,000 to 2,500 when x is 1; 3,000 to 6,000, preferably 3,750 to 4,250, when x is 2; 3,500 to 9,000, preferably 4,500 to 6,000, when x is 3; and 4,000 to 12,000, preferably 5,000 to 8,000, when x is 4. By the term "molecular weight of the surfactant compound, exclusive of E ," is meant the hydrophobic portion of the surfactant molecule. Thus, we have a critical molecular-weight range for this hydrophobic portion of the surfactant molecule. The preferred range is no more than 3,000 molecular-weight units for any given value for x . The preferred range is thus

seen to be dependent upon the functionality of Y . The narrow molecular-weight ranges are critical and necessary for the preparation of the surfactant compound of this invention. Below the given range, the surfactant compound does not have sufficient hydrophobic character, and thus does not contribute adequate surface activity to the floating bath oil composition. Such compounds do not promote rapid spreading of the bath oil on the water. They also do not prevent the formation of droplets and oily patches upon addition to, and cooling of, the bath water. When the molecular weight of this portion of the surfactant molecule is too high, it favors poor spreading and promotes adherence of the oil to the bathtub wall. An examination of the examples below will clearly indicate the necessity of operating within the molecular-weight limits as expressed above.

The term " E " in the generic formula represents a polyoxyalkylene chain wherein the oxygen/carbon atom ratio is at least 0.5, and E constitutes from about 3 to 18 percent, by weight, preferably 10 to 15 percent, of the surfactant compound. This, again, is a relatively narrow range which is critical in defining the percentage of oxyalkylene groups necessary to produce the surfactant of this invention. Below about 3 percent E the compound is but only poorly surface active and possesses insufficient spreading power. This is felt to be due to insufficient hydrophilic character. At E values above about 18 percent, by weight, of the surfactant compound, the hydrophilic nature of the compound is too great. This is evidenced by poor solubility, cloudiness or insolubility of the surfactant in the esters described above. There is also a tendency to the formation of oil-water emulsions. Furthermore, at high E values, the bath oil composition, which might have been in an initially well-spread film on the bath water surface, breaks up in the form of extremely unesthetic oily patches before the end of the normal bath time. A ring may also form around the tub sides.

The preparation of the floating bath oil composition of this invention may be accomplished by a simple, safe and convenient procedure. The emollient oil, surfactant compound and ester solvent for the surfactant compound are placed in a container and gently mixed until the product is clear and homogenous. An excellent bath oil composition may be prepared which comprises from about 30-70 percent by weight emollient oil, preferably 40-60 percent; 0.2-2.0 percent by weight surfactant compound, preferably 0.5-1.5 percent; and 25-65 percent by weight ester solvent, preferably 30-50 percent. Of course, more surfactant compound may be used, but it has been found that no significant advantage is obtained by exceeding the above range.

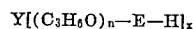
Other ingredients may be added to the bath oil composition for esthetic and other reasons. Thus, the addition of antioxidants, essential oils or perfume, and a coloring agent may be desired. Oil-soluble perfumes such as jasmine, spice and floral bouquet are often added in concentrations of from about 2 to 5 percent by weight, of bath oil. Oil-soluble dyes, government approved F. D. & C. and D. & C. dyes such as D. & C. Yellow No. 11, are often added in trace amounts for coloring purposes.

The invention is further illustrated, but not limited by the following examples in which the parts and percentages given are by weight.

EXAMPLE I

A floating bath oil composition was prepared by adding 60 grams of refined mineral oil, 1.0 gram of the surfactant compound described below, 22 grams of isopropyl myristate, 13 grams of isopropyl palmitate, and 4.0 grams of jasmine (Fleuroma No. 1,203) perfume to a 250 ml. beaker and gently mixing for about 5 minutes.

The surfactant compound employed had the generic formula



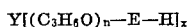
wherein:

Y = propylene glycol
 n = 32-35 (molecular weight of hydrophobe approximately 4,000)
 E = polyoxyethylene chain having a molecular weight of about 15 percent by weight of the total compound
 x = 2

The bath oil composition was clear and homogeneous. It spread rapidly and easily to form a continuous film on the surface of warm water having a temperature of 110° to 150° F. When the water cooled to room temperature, upon standing, the continuous bath oil film did not break up. Rinsibility was excellent.

EXAMPLES 2-17

A series of floating bath oil compositions of this invention are prepared according to the procedure of example 1. All ingredients, amounts, and the like are the same as Example 1 except that the surfactant having the generic formula



is varied as follows in Table I.

TABLE I

Ex.	Y	x	E, approx. percent polyoxyethylene by weight of total compound	Molecular weight of hydrophobe ¹
2	Propylene glycol	2	15	3,250
3	do	2	3	2,750
4	do	2	25	4,000
5	do	2	15	2,250
6	do	2	13	1,750
7	do	2	25	2,750
8	Trimethylolpropane	3	12	4,500
9	Glycerol	3	18	5,250
10	Pentaerythritol	4	10	5,750
11	Ethylene diamine	4	15	5,000
12	do	4	25	5,000
13	do	4	15	2,750
14	Methanol	1	15	3,000
15	Propylene glycol	2	10	4,000
16	do	2	18	4,000
17	Ethylene diamine	4	15	7,000

¹ Molecular weight of hydrophobe is molecular weight of surfactant compound exclusive of E.

Example 13-poor spreading power, droplets form on water surface, and rinsibility is poor.
 Example 14-a good bath oil.
 Example 15-an excellent bath oil.
 Example 16-an excellent bath oil.
 Example 17-an excellent bath oil.

EXAMPLE 18

Part A

A floating bath oil composition of this invention was compared with two commercial floating bath oil compositions as follows: 150 milligrams of oil-soluble dye, Brilliant Blue BMA, was added as a visual aid, to 100 grams of Example 1 and each of two commercial compositions. One drop of each of these three bath oil compositions was added simultaneously to three 1-liter beakers, each containing 950 ml. of water heated to about 140° F. The time of initial complete dispersion of the bath oil on the water surface was measured as an indication of spreading power. As another physical indication of surface activity and spreading power, the presence of a continuous film, patches, droplets, and rings of bath oil on or around the perimeter of the bath water surface, was noted at various time intervals as the water cooled on standing. The results of this test were as follows in Table II.

TABLE II

Bath oil composition	Example 1	Commercial 1	Commercial 2
Initial complete dispersion	2.5 sec.	5 sec.	30 sec.
2 minutes	Continuous film	Film starting break-up	Patches.
30 minutes	do	Patches	Droplets.
1 hour	do	Droplets	Droplets near sides.
3 hours	do	Droplets near sides	Ring.
16 hours	Continuous film on 2/3 of water surface.	Ring	Do.

Part B

The rinsibility of the above compositions was determined by pouring the contents from the beakers and wiping their inner surfaces with a clean paper towel. The discoloration on the paper towel from the dyed bath oil composition remaining on the beaker surfaces was noted. Example 1, the floating bath oil composition of this invention, showed excellent rinsibility as evidenced by only slight discoloration on the paper towel. Commercial floating bath oil preparations 1 and 2 evidenced poor rinsibility by the large degree of discoloration which appeared on the paper towel.

EXAMPLE 19

Part A of example 18 was repeated with the water temperature at 118° F. and using the same three floating bath oil compositions. The results of this test were as follows in Table III.

TABLE III

Bath oil composition	Example 1	Commercial 1	Commercial 2
Initial complete dispersion	7 sec.	120 sec.	300 sec.
10 minutes	Continuous film	Patches	Droplets.
3 hours	do	Droplets	Droplets near sides of beaker.

EXAMPLE 20

The procedure of Part A of example 18 was repeated using three successful floating bath oil compositions of this invention. The compositions utilized were those prepared according to examples 1, 2 and 11. Comparative results, which appear in Table IV, were obtained with the water temperature heated to 124° and 115° F.

EXAMPLES 21-25

A series of floating bath oil compositions of this invention are prepared according to the procedure of example 1. All ingredients, amounts, and the like are the same as example 1 except as follows:

Example 21—the ester solvent is propylene glycol dipelargonate.

The bath oil composition of Examples 2-17 will have the following properties as compared to Example 1, i.e.:

Example 2-a good bath oil in all respects but does not spread as rapidly as example 1.

Example 3-marginal spreading power and rinsibility.

Example 4-does not present a clear appearance.

Example 5-poor spreading power, droplets form on water surface, and rinsibility is poor.

Example 6-poor spreading power, droplets forming rings, and poor rinsibility.

Example 7-does not present a clear appearance.

Example 8-a good bath oil.

Example 9-a good bath oil.

Example 10-an excellent bath oil.

Example 11-an excellent bath oil.

Example 12-does not present a clear appearance.

TABLE IV

Bath oil composition	Example 1		Example 2		Example 11	
Initial water temperature.....	124° F.....	115° F.....	124° F.....	115° F.....	124° F.....	115° F.....
Initial complete dispersion.....	2 sec.....	25 sec.....	35 Sec.....	65 sec.....	30 sec.....	40 sec.....
1 hour.....	Continuous film.....	Continuous film.....	Continuous film.....	Continuous film.....	Continuous film.....	Continuous film.....
2 hours.....	do.....	do.....	do.....	do.....	do.....	Do.....
3 hours.....	do.....	do.....	do.....	do.....	do.....	Do.....

Example 22—the ester solvent comprises 25 grams of polypropylene glycol oleate and 10 grams of isopropyl myristate.

Example 23—the emollient oil is olive oil.

Example 24—0.5 gram of the surfactant compound is used.

Example 25—2.0 grams of the surfactant compound is used.

The bath oil compositions are clear and homogeneous. They spread rapidly and easily to form a continuous film on the surface of warm water having a temperature of 110° to 150° F. and rinsibility is excellent.

Examples 1, 2, 8, 9, 10, 11, 14, 15, 16, 17, 21, 22, 23, 24 and 25 are representative of the bath oil composition of this invention. An examination of the values appearing in example 1 and Table I for *Y*, *n* (molecular weight of hydrophobe), *x* and *E* of the surfactant compound, indicates that by observing the critical ranges discussed above, excellent floating bath oil compositions can be prepared. Examples 3, 5, 6, 7 and 13 illustrate poor bath oil compositions resulting when a surfactant compound is used which has the hydrophobic portion of the surfactant molecule outside the critical range.

Examples 4, 7 and 12 show that when the critical range for *E* is not observed, a good bath oil composition cannot be produced. Examples 1, 10, 11, 15, 16, 17, 21, 22, 23, 24 and 25 represent excellent floating bath oil compositions prepared according to the preferred conditions discussed above.

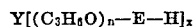
Example 18 indicates the superiority of a floating bath oil composition of this invention as compared to two commercial bath oil preparations. Enhanced speed of complete bath oil dispersion, spreading power and stability of the continuous bath oil film on the water surface are shown in Table II, Part A of example 18. Example 18, Part B shows the excellent and enhanced rinsibility of the bath oil composition of this invention as compared to the two commercial bath oil compositions. Examples 19 and 20 illustrate that these excellent and enhanced properties occur over an ideal temperature range which includes room temperature to about 140° F.

What is claimed is:

1. A clear nonemulsified floating bath oil composition which consists essentially of:

- a. from about 30 to 70 percent by weight of an emollient oil selected from the group consisting of hydrocarbon oils, silicone oils, olive oil, peanut oil and mixtures thereof,
- b. from about 0.2 to 2.0 by weight of a surfactant compound

insoluble in said emollient oil and in water, having the formula:



where *Y* is the residue of an organic compound containing therein *x* active hydrogen atoms and selected from the group consisting of ethanol, methanol, ethylene glycol, propylene glycol, glycerol, trimethylolpropane, pentaerythritol, methylamine, propylamine, ethylenediamine, and propanoic acid; *E* is a polyoxyalkylene chain wherein the oxygen/carbon atom ratio is at least 0.5 and *E* constitutes from about 3 to about 18 percent by weight, of the surfactant compound; *x* is an integer from 1 through 4; and *n* is an integer such that the molecular weight of the compound, exclusive of *E*, is from about 1,500 to 3,000 when *x* is 1, 3,000 to 6,000 when *x* is 2, 3,500 to 9,000 when *x* is 3, and 4,000 to 12,000 when *x* is 4, and

c. from about 25 to 65 percent by weight of an ester solvent for said surfactant compound selected from the group consisting of methyl palmitate, methyl myristate, dibutyl adipate, butyl sebacate, isopropyl laurate, isopropyl palmitate, isopropyl myristate, ethylene glycol dipelargonate, propylene glycol dipelargonate, and propylene glycol oleate.

2. The composition of claim 1 wherein *n* is an integer such that the molecular weight of the compound, exclusive of *E*, is from about 2,000 to 2,500 when *x* is 1, 3,750 to 4,250 when *x* is 2, 4,500 to 6,000 when *x* is 3, and 5,000 to 8,000 when *x* is 4.

3. The composition of claim 1 wherein said emollient oil is mineral oil.

4. The composition of claim 1 wherein said *E* constitutes from about 10 to 15, by weight, of the surfactant compound.

5. The composition of claim 1 wherein said ester solvent is isopropyl myristate.

6. The composition of claim 1 wherein said *Y* is the residue of an organic compound selected from the group consisting of methanol, propylene glycol, glycerol, trimethylolpropane, pentaerythritol, and ethylene diamine.

7. The composition of claim 6 wherein said *Y* is the residue of propylene glycol.

8. The composition of claim 6 wherein said *Y* is the residue of ethylene diamine.

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UNITED STATES PATENT OFFICE
CERTIFICATE OF CORRECTION

Patent No. 3,624,208 Dated November 30, 1971

Inventor(s) Irving R. Schmolka

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

Column 7, line 53 after 2.0 should be added --%-- . Column 8, line 24, after is, first occurrence, should read --1, 3000--; column 8, line 42 after 15 should be added ---%---

Signed and sealed this 9th day of May 1972.

(SEAL)
Attest:

EDWARD M. FLETCHER, JR.
Attesting Officer

ROBERT GOTTSCHALK
Commissioner of Patents