United States Patent

Russell

[15] **3,666,857**

[45] **May 30, 1972**

[54] AQUEOUS COMPOSITIONS CONTAINING LANOLIN OIL

[72] Inventor: **Kenneth L. Russell**, Nutley, N.J.

[73] Assignee: **Malmstrom Chemical Corp.**, Linden, N.J.

[22] Filed: **Mar. 4, 1966**

[21] Appl. No.: **531,756**

Related U.S. Application Data

[63] Continuation of Ser. No. 178,221, Mar. 1, 1962, abandoned.

[52] **U.S. Cl.** **424/240**, 424/70

[51] **Int. Cl.** **A61k 7/08**

[58] **Field of Search** ..... 167/63 L; 424/240

[56] **References Cited**

UNITED STATES PATENTS

2,900,307 8/1959 Wei 167/63

OTHER PUBLICATIONS

Conrad et al., Proceedings of Scientific Section, Toilet Goods Assoc., No. 29, pages 14– 18, June 1958

Robinson, Wagner Co., Inc. Products Bulletin No. 37, pages 2– 3, Apr. 2, 1956

Russell et al., Drug and Cosmetic Industry, Vol. 90, No. 3, pages 294– 296, 325, 326, and 328, Mar. 1962

Primary Examiner—Richard L. Huff

Attorney—Eric Y. Munson

[57] **ABSTRACT**

A liquid fraction of lanolin is solubilized in water with a combination of (A) fatty acid soaps and/or synthetic detergents, (B) a polyoxyethylene ether of lanolin alcohol condensed with 16 mols of ethylene oxide, and (C) a higher fatty acid alkanolamide.

7 Claims, No Drawings

AQUEOUS COMPOSITIONS CONTAINING LANOLIN OIL

This application is a continuation of my prior application, Ser. No. 178,221, filed Mar. 1, 1962, now abandoned.

This invention relates to aqueous compositions containing lanolin oil, and concerns particularly aqueous compositions in which lanolin oil is employed as an emollient and conditioner component of the composition, as in the case of shampoos and other detergent products.

Still more particularly, the invention is concerned with the preparation of such compositions in the form of clear, transparent aqueous solutions.

Prior to relatively recent years, aqueous detergent preparations containing an appreciable quantity of lanolin were characterized by opaqueness of the product, owing to the fact that the lanolin was emulsified in the detergent solution, rather than being completely solubilized therein or at least so finely dispersed therein that the product is clear to the naked eye and exhibits a transparency thereto sufficient to look like a solution.

It is known (W.C. Griffin, "Cosmetics, Science and Technology", Interscience Publishers, Inc., New York, Sept. 16, 1957, pages 1027–1028) that non-ionic surfactants of the polyoxyethylene ester, ether, and ether-ester types are efficient solubilizers for oils, in that they form clear, transparent emulsions of liquid oils which are, in effect, oil in water emulsions wherein the product is clear because the particle size of the oil is so small that light is not refracted by the particles. The non-ionic surface active agents commonly used in cosmetic formulations are not, however, efficient solubilizers for lanolin, so that it is necessary to employ relatively high ratios of such non-ionics to lanolin, in order to solubilize the oil sufficiently to form a clear, transparent solution.

It is also known (Conrad, Motiuk and Maso, "Solubilization by Lanolin Derivatives", Proceedings of Scientific Section, Toilet Goods Association, 29, June 14, 1958) that the U.S.P. lanolin and a liquid lanolin may be solubilized by the polyoxyethylene ethers of lanolin alcohol, particularly the ether made by condensing lanolin alcohols with 16 mols of ethylene oxide.

In U.S. Pat. No. 2,900,307, to Ling Wei, dated Aug. 18, 1959, lanolin and fractionated lanolin are described as being rendered water-soluble by combining therewith certain higher fatty acid alkylolamides in ratios of from 1:1 to 100:1. In practise, however, it has been found necessary to employ a ratio of at least 8:1 of alkylolamide to lanolin in order to solubilize the latter. Thus, in each of the illustrative examples set forth in the Wei patent, the ratio of alkylolamide to lanolin is 10:1.

The principal object of the present invention is to provide aqueous compositions which contain lanolin oil as an essential component thereof and are in the form of clear, transparent solutions.

Another object of the invention is to provide aqueous compositions of the character set forth, which will be of particular advantage in the preparation of hair shampoos, hand lotions and other cosmetic and pharmaceutical products.

Still other objects, and the advantages of, the invention will be apparent to those skilled in the art from the following more detailed description.

Briefly stated, the objects and advantages of the invention may be achieved by combining with lanolin oil, a non-ionic surface active agent consisting essentially of a polyoxyethylene ether of lanolin alcohols, and a higher fatty acid alkanolamide, and then combining this mixture with an aqueous solution of an organic synthetic detergent, or a fatty acid soap, or suitable admixtures thereof.

The lanolin oil used in accordance with the invention is a water-insoluble liquid fraction of lanolin composed for the most part of liquid lanolin esters. Typical of such lanolin oils, for example, is the liquid fraction of lanolin obtained from wool grease by solvent fractionation and crystallization according to the process set forth in U.S. Pat. No. 2,758,125 to Sunde. A lanolin oil of this type is commercially available from Malmstrom Chemical Corporation under the tradename "Lantrol". Other lanolin oils suitable for use in the practise of the invention are available commercially from other suppliers under the trademarks "Lanogene", "Fluilan" and "Viscolan".

The non-ionic surface active agent preferably employed as one of the components of the solubilizing agent for the lanolin oil in accordance with the invention is a polyoxyethylene ether of lanolin alcohols condensed with 16 mols of ethylene oxide. Typical of such an ether is the product marketed under the trademark "Nimcolan S" by Malmstrom Chemical Corporation.

The alkanolamide employed as the other component of the solubilizing agent for the lanolin oil in accordance with the invention may be any of a number of well-known alkanolamides of higher fatty acids, preferably the diethenolamide of coconut fatty acid. Typical of suitable alkanolamides are those marketed by Clintwood Chemical Company under the trade designations "Sandrol Superamide 100C" and by others.

It has been found that mixtures of a lanolin alcohol polyoxyethylene oxide ether, particularly of the preferred type above-mentioned, with a higher fatty acid alkanolamide, provide surprisingly effective results when employed as solubilizer for lanolin oil in the preparation of clear, transparent aqueous solutions containing lanolin oil. Moreover, it has been found that although the aforementioned components of the solubilizing mixture may be used in a relatively wide range of concentrations for the preparation of such aqueous products, these ingredients in admixture with one another lend themselves to use in concentrations, relative to the concentration of the lanolin oil, such as to effect a synergistic solubilization of the lanolin oil. Thus, markedly lower relative amounts of these ingredients serve to produce clear, transparent aqueous solutions containing lanolin oil.

The clarity and transparency of these solutions are an "as is" characteristic thereof, i.e., as produced without requiring filtering of the solutions. Hence, the terms "clear" and "transparent" as used herein with reference to the solutions embodying the present invention, refer to and mean the aforesaid characteristic of the solutions as initially produced, without requiring them to be filtered to render them clear and transparent.

The herein described solubilizing mixtures of the polyoxyethylene ether of lanolin alcohols with the higher fatty acid alkanolamide may be employed in relative proportions as hereinafter more particularly set forth, to form clear, transparent aqueous compositions containing from approximately 1 to 3 percent by weight of lanolin oil and from about 5 to 30 percent, preferably from about 8 to 24 percent, by weight, of a water-soluble detergent, such as a fatty acid soap, or a suitable ionic or non-ionic synthetic detergent, or suitable mixtures thereof.

Thus, in the case of aqueous solutions containing from about 8 to 24 percent triethanolamine lauryl sulfate as detergent and from 1 to 3 percent lanolin oil, clear, transparent solutions may be obtained by utilizing a mixture of the polyoxyethylene ether of lanolin alcohols with the coconut fatty acid diethanolamide in amounts such that the former is present in a ratio of from about 1:1 to 3:1 relative to the amount of lanolin oil, and the latter is present in a ratio of from about ½:1 to about 7:1 relative to the amount of lanolin oil, the above-mentioned synergistic effect disappearing, however, when the ratio of the polyoxyethylene ether of lanolin alcohols to the lanolin oil is about 1:1 or lower.

In producing the aqueous compositions according to the invention, the preferred procedure is to form a mixture of lanolin oil with the above-mentioned solubilizing agents, and separately, an aqueous solution of the selected detergent, the aforesaid mixture and the detergent solution each being heated to about 75° to 80° C., whereupon the oil mixture is added to the detergent solution with stirring at the elevated temperature above-mentioned.

In order to demonstrate the beneficial effects of the combination of the above-mentioned ingredients as solubilizers for lanolin oil, there is set forth in Table I, below, the solubilization results when utilizing the non-ionic surfactant (polyox-

yethylene ether of lanolin alcohols condensed with 16 mols of ethylene oxide) and the higher fatty acid alkanolamide (coconut fatty acid diethanolamide), separately, as the solubilizer for 1.0 percent lanolin oil in aqueous solutions containing varying concentrations of an anionic detergent (triethanolamine lauryl sulfate).

TABLE I.—SOLUBILIZATION RESULTS
1.0% Lanolin Oil in Triethanolamine Lauryl Sulfate

Materials	Percent Triethanolamine lauryl sulfate		
	8.0	16.0	24.0
Ratio-nonionic to lanolin oil.	4.0:1, clear---- 3.5:1, cloudy...	3.5:1, clear---- 3.0:1, cloudy ..	3.5:1, clear. 3.0:1, hazy.
Ratio-diethanolamide to lanolin oil.	8.0:1, clear---- 7.0:1, cloudy---	8.0:1, clear---- 7.0:1, cloudy..	9.0:1, clear. 8.0:1, cloudy.

In contrast to the results obtained with the individual ingredients of the solubilizer, as set forth in Table I, there are set forth in Table II, below, the results obtained when utilizing mixtures of the above-mentioned ingredients as solubilizers for the lanolin oil in accordance with the invention.

TABLE II - SOLUBILIZATION RESULTS

1.0% Lanolin Oil in Triethanolamine Lauryl Sulfate

Mixtures:	% Triethanolamine Lauryl Sulfate 8.0	16.0	24.0
Non-ionic surfactant (A)Lanolin Oil Diethanolamide	3.0) 1.0)clear 0.5) 2.0)	3.0) 1.0)clear 0.5)) 2.0)	3.0) 1.0)clear 0.5) 2.0)
(B)Ditto above	1.0)clear 2.0) 2.0)	1.0)clear 3.0) 2.0)	1.0)clear 3.0) 2.0)
(C)Ditto above	1.0)cloudy 1.0) 1.0)	1.0)cloudy 2.0) 1.0)	1.0)cloudy 2.0) 1.0)
(D)Ditto above	1.0)clear 6.5–7.0)	1.0)clear 5.5–6.0)	1.0)clear 7.0-7.5)

In all instances set forth in Table II, as well as in Table I, the amount of the detergent solution of triethanolamine lauryl sulfate used was 100.0 parts, less the 1.0 part of the lanolin oil and less the parts of non-ionic surfactant and the diethanolamide used, as the case may be, yielding 100.0 parts of the final composition containing 1.0 part of lanolin oil.

In all instances, also, the detergent solution was heated to 75°–80 C., the mixture of lanolin oil and solubilizing material was heated to the same temperature, and then the oil mixture was added to the detergent solution with stirring at the elevated temperature.

By comparing the data set forth in Table I with those set forth in Table II, it will be noted that whereas in the case of an 8.0 percent solution of the detergent, 4.0 parts of the non-ionic surfactant were required to solubilize 1.0 part of lanolin oil in 95.0 parts of the 8.0 percent detergent solution, and 8.0 parts of the diethanolamide were required to solubilize 1.0 part of lanolin oil in 91.0 parts of the 8.0 percent detergent solutions, only 3.0 parts of the non-ionic surfactant and only 0.5 parts of the diethanolamide, when these two are used in admixture with one another, are required to solubilize 1.0 part of lanolin oil in 95.5 parts of the 8.0 percent detergent solution. Similarly, only 2.0 parts of the non-ionic surfactant admixed with only 2.0 parts of the diethanolamide are required to solubilize 1.0 part of lanolin oil in 96.0 parts of the 8.0 percent detergent solution. Comparably striking differences in the solubilization results will be noted when comparing the data in Table I with those of Table II in respect of the 16 and 24 percent detergent solutions.

In most instances set forth in Table II, the solubilization took place readily as the mixture of lanolin oil, non-ionic and diethanolamide, heated to 75°–85° C., was added to the detergent solution heated to the same temperature. In the case of combinations made with 1.0 part of the non-ionic surfactant to 1.0 part of lanolin oil, holding the mixture of lanolin oil, non-ionic and diethanolamide with the detergent solution at a temperature of **75°–80°** C., for a period of up to 30 minutes may be required in order to obtain a clear, transparent solution. Indeed, in such instances, maintaining the mixture at the temperature mentioned for periods longer than 30 minutes permits the use of a smaller quantity of the diethanolamide than those shown in Table II for obtaining clear solutions.

As will be observed from the data in Table II, when ratios of 3:1 and2:1 of the non-ionic surfactant to lanolin oil are used, the amount of diethanolamide required for solubilization is substantially less than would be calculated if the solubility effects of the non-ionic and diethanolamide were merely additive. This synergistic solubilization of the lanolin oil is exemplified in the compositions of series (A) and (B) in Table II. This synergistic effect disappears, apparently, when the ratio of non-ionic to lanolin oil is 1:1.

Using mixtures of the non-ionic and diethanolamide, larger quantities of lanolin oil were readily solubilized. For example, the following two compositions containing 3.0 percent lanolin oil were clear immediately after combining the ingredients by the above-mentioned procedure:

Triethanolamine Lauryl Sulfate	16.0	**16.0**
Water	57.0	**58.0**
Non-ionic (lanolin alcohols with 16 mols ethylene oxide)	9.0	**10.5**
Lanolin Oil	3.0	**3.0**
Coconut Fatty Acid Diethanolamide	15.0	**12.0**
Total:	100.0	**100.0**

Solubilization of lanolin oil in aqueous solutions of detergents other than the one referred to in Tables I and II, when utilizing mixtures of the non-ionic and diethanolamide as solubilizers, resulting in clear, transparent solutions, is illustrated by the two following examples of compositions prepared in the manner above set forth:

Sodium Lauryl Polyoxyethylene Ether Sulfate	1 12.0	**2** —
Triethanolamine Dodecyl Benzene Sulfonate	—	12.4
Water	79.0	**78.1**
Non-ionic (lanolin alcohols with 16 mols ethylene oxide)	2.0	**2.5**
Lanolin oil	1.0	**1.0**
Coconut Fatty Acid Diethanolamide	6.0	**6.0**
Total:	100.0	**100.0**

Solubilization of lanolin oil in concentrations of 1.0 percent in aqueous solutions of mixtures of anionic synthetic detergents and a fatty acid soap, and utilizing mixtures of the non-ionic surfactant with the diethanolamide as solubilizer, is illustrated by the following example:

Part I	Parts
Sodium lauryl sulfate	9.0
Triethanolamine dodecyl benzene sulfonate	8.0
Potassium Oleate	2.5
Water	72.0
Part II	
Non-ionic (lanolin alcohols with 16 mols ethylene oxide)	2.5
Lanolin oil	1.0
Coconut fatty acid diethanolamide	5.0
Total:	100.0

Part I was heated to 78° C. and Part II was added to Part I with stirring. The resulting product was hazy immediately after combining them, but became sparkling clear during 5 minutes additional heating on a steam bath at 75°–80° C.

As above indicated, the aqueous compositions containing the lanolin oil, non-ionic surfactant and coconut fatty acid diethanolamide, may be utilized in the preparation of clear aqueous hair shampoos, hand lotions and other cosmetic products. Table III, below, sets forth the data with respect to several clear, aqueous hair shampoos.

TABLE III

	SHAMPOO			
	A	B	C	D
Triethanolamine lauryl Sulfate	12.0	12.0	12.0	12.0
Triethanolamine oleate	5.0	5.0	5.0	5.0
Propylene glycol	5.0	5.0	5.0	5.0
Water	71.0	70.5	69.0	69.0
Lanolin oil	1.0	1.0	1.0	1.0
Lanolin alcohols condensed with 16 mols of ethylene oxide	3.0	2.5	2.0	3.0
Coconut fatty acid 1:1 diethanolamide	3.0	4.0	6.0	5.0
Total:	100.0	100.0	100.0	100.0

The anionic detergent, soap and propylene glycol were dissolved in the water and this solution heated to 75°–80° C. The other ingredients were combined and heated to the same temperature, then poured into the aqueous solution while stirring. Compositions A and D were clear very shortly after combining the ingredients; composition B required 5 minutes additional heating at 75°–80° C.; and composition C required 15 minutes at this temperature.

Although the data in Table III, above, indicate that the synergistic effect of the non-ionic and the diethanolamide is not exerted, it is believed this is due to the presence of the propylene glycol, and that in the absence of the latter the synergistic effect would be exerted.

The product of the composition listed in Column D of Table III was subjected to rabbit-eye irritation tests by the Draize technique. Nine rabbits were used, three with no rinsing after instillation of the product, three with rinsing after 2 seconds, three after 4 seconds. In all nine rabbits zero readings for irritation of the cornea or iris were obtained. Low readings for irritation of the conjunctivae were obtained on the three rabbits' eyes which were not rinsed; these readings became zero after 4 days. Zero readings of conjunctivae irritation were obtained on the other six rabbits.

The product of composition D listed in Table III was also tested for shampoo results by the so-called half-head shampoo test. In such test, the hair is wetted, parted in the middle, and then the hair on one side of the part is shampooed with the product under test, and the hair on the other side of the part is shampooed with another shampoo for comparison. Equal quantities of the product under test and of the reference product are used, and a trained beauty operator conducts the tests, making observations with respect to the foaming properties, feel of the hair during and after shampooing and rinsing, and combing properties of the hair on each side of the parted hair after the shampooing is completed. Utilizing this test, the product of composition D (with an addition of 0.4 percent, by weight, of a suitable perfume) was compared with several commercially available shampoo products, containing 1.0 lanolin oil and one or the other of the above-mentioned solubilizing ingredients. The tests showed that the product of composition D foamed excellently and had unusually good effects on the combing properties of the hair following the shampoo. On hair damaged by bleaching or dyeing, the easier combing properties and manageability of the hair were outstandingly better when shampooed with the product of composition D than when shampooed with the commercial shampoo products tested.

As hereinbefore indicated, the preferred type of alkanolamide for use in preparing the compositions of the invention is coconut fatty acid diethanolamide. More specifically, as in the compositions shown in Table III, the diethanolamide is a 1:1 coconut fatty acid-diethanolamine condensate of high (minimum 90 percent) amide content. Thus, with this amide, a clear, transparent product is obtained immediately after combining the following ingredients, in the proportions indicated, at 75° to 80° C. in the manner above set forth:

Triethanolamine lauryl sulfate	8.0
Water	87.0
Polyoxyethylene ether (lanolin alcohols with 16 mols ethylene oxide)	2.0
Lanolin oil	1.0
Amide	2.0
Total:	100.0

Experiments have shown that different lots of raw materials may produce some variation in results. For example, one lot of the non-ionic may solubilize the lanolin oil in a given detergent solution when present at a ratio of 3.0 parts to 1.0 part of lanolin oil, while another lot may require a slightly higher ratio. Similar variations occur using different lots of the fatty acid diethanolamide. Likewise, using given lots of the non-ionic and the diethanolamide, one lot of lanolin oil may be slightly easier to solubilize than another. Generally the differences in the materials are small, and their effect on solubilization can be compensated for by variation of the period of heating at 75°–80° C. The raw materials from different suppliers may also vary somewhat in their ability to solubilize, or in the case of lanolin oil, to be solubilized. However, for a given finished product, the formulator may readily determine and choose a final composition and conditions so that these variations will not be significant.

As will be understood by those skilled in the art, suitable amounts of various additive agents may be included in the final compositions embodying the invention. Among these are coloring agents, perfumes, dyes, preservatives, and the like. In this connection, it is noteworthy that the diethanolamide component in the compositions of the invention serve also as foam builders or foam stabilizers in products such as shampoos, and as viscosity adjusting agents in detergent solutions.

What is claimed is:

1. A composition consisting essentially of:

1. water
2. a water-soluble substance selected from the group consisting of fatty acid soaps, synthetic detergents and mixtures thereof,
3. a liquid fraction of lanolin,
4. a non-ionic surfactant consisting essentially of a polyoxyethylene ether of lanolin alcohol condensed with 16 mols of ethylene oxide; and
5. a higher fatty acid alkanolamide:

said surfactant being present in a ratio of from about 1:1 to about 3:1 relative to the amount of said liquid fraction of lanolin, and said alkanolamide being present in a ratio of from about ½:1 to about 7:1 relative to the amount of said liquid fraction of lanolin, whereby to effect synergistic solubilization of said liquid fraction of lanolin said composition being clear and transparent as initially produced and without requiring filtration thereof.

2. The composition of claim **1**, wherein said water-soluble substance is present in a concentration of from about 5 to 30 percent by weight of said solution, and wherein said liquid fraction of lanolin is present in a concentration of from approximately 1 to 3 percent by weight.

3. The composition of claim **1**, wherein said water-soluble substance is present in a concentration of from about 8 to 24 percent by weight of said solution, and wherein said liquid fraction of lanolin is present in a concentration of from approximately 1 to 3 percent by weight.

4. The composition of claim **2**, wherein said alkanolamide is a diethanolamide of coconut oil fatty acid.

5. The composition of claim **3**, wherein said liquid fraction of lanolin is present in a concentration of approximately 1 percent by weight, said non-ionic surfactant is present in a concentration of from approximately 2 to 3 percent by weight, and said alkanolamide is a diethanolamide of coconut oil fatty acid, present in a concentration of from approximately 0.5 to

3 percent by weight, the lower the concentration of said non-ionic surfactant the higher the concentration of said diethanolamide.

6. A composition consisting essentially of:

1. water
2. a water-soluble substance selected from the group consisting of fatty acid soaps, synthetic detergent and mixtures thereof,
3. a liquid fraction of lanolin
4. a non-ionic surfactant consisting essentially of a polyoxyethylene ether of lanolin alcohol condensed with 16 mols of ethylene oxide, and
5. a diethanolamide of coconut oil fatty acid;

said liquid fraction of lanolin being present in a concentration of from 1 to 3% by weight of the composition, said surfactant being present in a ratio of from about 1:1 to about 3:1 relative to the amount of said liquid fraction of lanolin, and said alkanolamide being present in a ratio of from about ½:1 to about 7:1 relative to the amount of said liquid fraction of lanolin, whereby to effect complete solubilization of said liquid fraction of lanolin said composition being clear and transparent as initially produced and without requiring filtration thereof.

7. A liquid composition consisting essentially of:

a. water
b. a liquid fraction of lanolin,
c. a non-ionic surfactant consisting essentially of a polyoxyethylene ether made by condensing lanolin alcohols with 16 mols of ethylene oxide; and
d. a higher fatty acid alkanolamide;

said surfactant and said alkanolamide each being present in ratios relative to the amount of said liquid fraction of lanolin to effect synergistic solubilization of said liquid fraction of lanolin, said ratio of said surfactant to said liquid fraction of lanolin being from about 1:1 to about 3:1, and said ratio of said alkanolamide to said liquid fraction of lanolin being from about ½:1 to about 7:1 said composition being clear and transparent as initially produced and without requiring filtration thereof.

* * * * *