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DERIVATIVES OF CERTAIN HIGHLY BRANCHED CHAIN ACIDS AS COATING AIDS

William J. Knox, Jr., Rochester, N.Y., and Robert E. Sticker, Lawrence, Kans., assignors to Eastman Kodak Company, Rochester, N.Y., a corporation of New Jersey

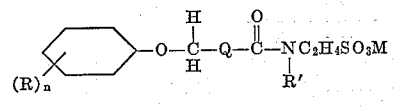
No Drawing. Filed Feb. 7, 1962, Ser. No. 171,560
11 Claims. (Cl. 96—94)

This invention relates to coating aids for gelatin coating solutions particularly for photographic purposes which are certain alkaryloxyalkanoyl taurine salts.

In the application of aqueous gelatin coating compositions, particularly gelatin-silver halide photographic emulsions, to a support such as film base or paper, it is desirable that there be present therein a coating aid to facilitate the application of the layer to the support. This application of gelatin composition can be either as a single layer or in multilayers such as by simultaneous application. The presence of coating aid reduces defects such as repellencies, pencil line streaks, and the like in the coating when dry. In such coating operations, however, the coating aids used often fail to induce adequate rewettability of the layer which usually exhibits surface slickness. In many cases, this surface slickness or slipperiness is especially undesirable in certain photographic products because of difficulties in handling and possible telescoping of the film wound in the form of rolls. Often it has been necessary to use a mixture of two surfactants as the coating aid to impart both desired surface roughness and rewettability of the dry coating. Not only is the mixing of coating aids sometimes bothersome but in addition some of the surfactants proposed for that purpose are naturally occurring materials and may vary in quality or contain impurities undesirable for photographic use.

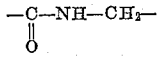
One object of our invention is to provide gelatin coating compositions from which layers having both adequate rewettability after drying and sufficient roughness of surface to facilitate handling may be obtained. Another object of our invention is to provide gelatin coating compositions from which coatings having few if any repellencies, pencil line streaks or other undesirable defects may be obtained. A further object of our invention is to provide coating aids comprising certain synthetically prepared taurines. Other objects of our invention will appear herein.

We have found that taurines which have alkaryloxyalkanoyl substituents show good properties when incorporated in photographic emulsions or other gelatin coating compositions as coating aids. The resulting compositions have provided coatings having good handling characteristics. Coating aids which we have found to be particularly useful in gelatin compositions are those having the formula:



in which

$(R)_n$ is one or two branched alkyl groups of a total of 8 to 16 carbon atoms, n is an integer 1 or 2, Q is a bond or



R′ is alkyl of 1 to 4 carbon atoms or cycloalkyl, and

M is a substituent such as Na, K, Li, NH_4 or alkylammonium to give a water soluble salt.

We have found that when these compounds are incorporated in small amounts in gelatin coating compositions those compositions exhibit good coating properties. This has been found to be true both in coating operations in which a single layer has been applied to a support or in multiple coating operations such as described in U.S. 2,761,991 of T. A. Russell. It was found that any amount of coating aid present in the gelatin composition was effective in giving better coatings but that it is especially convenient to operate within the range of 0.5–15%, based on the weight of the gelatin, of coating aid in the composition and preferably within the range of 1–10%. In the coating aids in accordance with our invention it is preferred that the branched alkyl substituent attached to the benzene ring have 8–16 carbon atoms although included within our invention are the coating aids in which two alkyl substituents may be on the benzene ring, the sum of the carbons of these alkyls being 8–16 carbon atoms. For instance, some of the compounds which have been considered most convenient to use are alkaryl compounds in which the sum of the alkyl substituents of the alkaryl has 8, 10, 12, 15 or the like carbon atoms either one or two alkyls being attached to the benzene ring. The nitrogen of the taurine compound has as a substituent thereon alkyl of 1–4 carbon atoms such as methyl, ethyl or cycloalkyl i.e. cyclohexyl.

Some coating aids for use in gelatin coating compositions in accordance with our invention are:

Na salt of N-p-t.-octylphenoxyacetyl-N-cyclohexyltaurine
Na salt of N-p-t.-octylphenoxyacetyl-N-methyltaurine
Na salt of N-p-t.-octylphenoxyacetylglycyl-N-methyltaurine
Na salt of N-(branched)nonylphenoxyacetyl-N-methyltaurine
Na salt of N-(branched)nonylphenoxyacetyl-N-cyclohexyltaurine
Na salt of N-(branched)dodecylphenoxyacetyl-N-methyltaurine
Na salt of N-(branched)dodecylphenoxyacetyl-N-cyclohexyltaurine
Na salt of N-(branched)dodecylphenoxyacetyl-N-ethyltaurine

The following examples illustrate the preparation of coating aids of the type referred to and their use in preparing film products.

Example 1

354 parts (0.359 mole) of a 23.8% aqueous solution of the sodium salt of N-cyclohexyltaurine was evaporated in vacuo on a steam bath to ⅓–½ the original volume so as to remove any cyclohexylamine which might be present. The mass was then diluted with water to 354 parts and 200 parts of acetone were added. Added simultaneously to the stirred solution at 60° C. while maintaining the pH at 10–13, was 107 parts of p-t.-octylphenoxyacetyl chloride and 15.2 parts of sodium hydroxide in 25% aqueous solution. After completion of the addition, the mass was held at 60° C. for ½ hour. The acetone and some water were boiled off on a steam bath and the solution was cooled to room temperature. 2000 parts of ether and 100 parts of sodium chloride were added. After thorough stirring and standing, an ether layer separated. The lower aqueous layer was removed and extracted with 1000 parts of ether. The

two ethereal extracts were combined, dried over sodium sulfate, and filtered. The ether was mostly evaporated off at room temperature; further evaporation was then carried out at 80° C. for several days followed by evaporation in vacuo at 55° C. The light tan product thus obtained, N - p - t. - octylphenoxyacetyl - N - cyclohexyltaurine, sodium salt, was useful as a coating aid in aqueous gelatin compositions.

Example 2

1690 parts (4.2 moles) of a 40% solution in water of the sodium salt of N-methyltaurine was evaporated in vacuo on a steam bath to approximately ½ the original volume thereby removing any methylamine present. The mass was diluted to 1690 parts with water and was further diluted with 6000 parts of 1:2 acetone:water. 1185 parts (4.2 moles) of p-t.-octylphenoxyacetyl chloride and 168 parts of 25% sodium hydroxide solution were added simultaneously to the the stirred solution at 60° C. while maintaining a pH of 10–13. The addition having been completed, the reaction mass was stirred for one hour at 60° C. removing the acetone and some water. The solution was slightly turbid but cleared upon dilution to 25% concentration and cooling to room temperature. The material, N-p-t.-octylphenoxyacetyl-N-methyltaurine, sodium salt, was found to impart good coating properties to gelatin coating compositions in which it is incorporated as will be indicated below.

Example 3

In a coating experiment in which a single coating of an aqueous solution of gelatin was applied to cellulose acetate film base N-p-t.-octylphenoxyacetyl-N-methyltaurine, sodium salt (OPT), was used as the coating aid at a concentration of 0.22 gram per pound of gelatin solution. The following results were obtained as compared with a control in which no coating aid was used:

Coating aid:	Repellencies (25 ft.)
Control	13
OPT	2

Example 4

In a coating run similar to that described in Example 3 except that N-pentadecylphenoxyacetyl-N-methyltaurine, sodium salt (PPT), was used as a coating aid, the following results (as compared with a control in which no coating aid was used) were obtained:

Coating Aid	Repellencies (25 ft.)	Mottle	Longitudinal Streaks
Control	6	Sl	Sl.
PPT	3	Sl	Sl.

Example 5

In a coating run similar to that described in Example 3 in which N-p-t.-octylphenoxyacetylglycyl - N - methyltaurine, sodium salt (OPGMT), was used as the coating aid; the following results were obtained:

Coating Aid	Repellencies (25 ft.)	Mottle	Longitudinal Streaks
Control	2	Med	Sl.
OPGMT	0	Sl to Med	Sl.

Example 6

In a coating experiment in which both a photographic emulsion layer and a clear gelatin overcoating were applied to paper by a multiple hopper coating technique as described in U.S. Patent No. 2,761,991 using N-p-t.-octylphenoxyacetyl-N-methyltaurine, sodium salt (OPT), compared to a standard in which a mixture of two coating aids was used so as to give both good developer spread and resistance to slipping. The following results were obtained:

Surfactant Concentration		Developer Spread [1]	Coef. of Friction [2]	Repelencies (7.5 sq. ft.)
Wet Emul., g./lb.	Wet Overcoat, g./lb.			
0.13	0.5	4.90	0.507	None
0.25	0.5	6.16	0.517	None
0.5	0.5	8.55	0.517	None
Standard	Standard	5.72	0.517	None

NOTE.—In the above tables repellencies are the number of dull and uncoated spots in the area referred to.

[1] Developer spread is determined as follows:
One cc. of developer solution is delivered in a standard manner to a surface of photographically sensitized paper carrying a clear gelatin overcoat and the area which develops in a period of 30 seconds was measured. This test indicates how effectively an emulsion containing a coating aid will be wet. A developer spread of 4.0 cm.² or more is desirable.

[2] Coefficient of friction is determined by placing the coated side of photographically sensitized paper carrying a clear gelatin overcoat against the uncoated side of a similar sheet. The coefficient of friction is then measured thus determining the slickness of gelatin coatings containing coating aids. Difficulties occur in stacking coated sheets if the coefficient of friction is less than about 0.25.

Example 7

In a coating experiment similar to that described in the preceding example, N-p-t.-octylphenoxyacetyl - N - cyclohexyltaurine, sodium salt (OPCT), was used as the coating aid. The following results were obtained:

Surfactant Concentration		Developer Spread, cm.²	Coef. of Friction	Repellencies (7.5 sq. ft.)
Wet Emul., g./lb.	Wet Overcoat, g./lb.			
0.13	0.5	18.20	0.660	None
0.25	0.5	15.20	0.650	None
0.5	0.5	16.60	0.627	None
Standard	Standard	8.04	0.715	None

Example 8

In a coating experiment similar to that described in Example 3, N-p-t.-octylphenoxyacetyl-N-cyclohexyltaurine, sodium salt (OPCT), was used as the coating aid. The following results were obtained:

Coating Aid	Repellencies (25 ft.)	Mottle	Longitudinal Streaks
Control	12	Sl	Med.
OPCT	0	Med	Sl.

Example 9

In a coating experiment similar to that described in the preceding example, N-2,4-di(t-amyl)phenoxyacetyl-N-cyclohexyltaurine, sodium salt (ACT), was used as the coating aid. The following results were obtained:

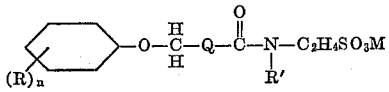
Coating Aid	Repellencies (25 ft.)	Mottle	Longitudinal Streaks
Control	12	Sl	Med.
ACT	0	Med	Sl.

The occurrence of pencil line streaks is a persistent defect in coatings achieved by the simultaneous application of two gelatin layers to a film base or paper. The ability of a coating aid to reduce the incidence of pencil line streaks is indicated when 1 ml. of a photographic emulsion containing the coating aid has a spread of about 5 cm.² when coated in a standard manner on a soap-cleaned, horizontal surface of a highly polished stainless steel plate.

We claim:

1. A composition of matter comprising gelatin con-

taining a coating aid corresponding to the following formula:



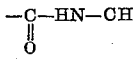
In which

R is branched alkyl,

$(R)_n$ totals 8 to 16 carbon atoms,

n is an integer of 1 to 2,

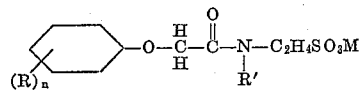
Q is selected from the group consisting of a bond and



R′ is selected from the group consisting of alkyls of 1 to 4 carbon atoms and cycloalkyl,

and M is a substituent selected from the group consisting of Na, K, Li, NH_4 and alkylammonium.

2. A composition of matter comprising gelatin containing coating aid corresponding to the following formula:



In which

R is branched alkyl,

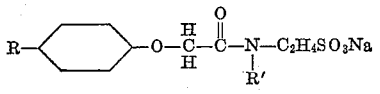
$(R)_n$ totals 8 to 16 carbon atoms,

n is an integer of 1 to 2,

R′ is selected from the group consisting of alkyls of 1 to 4 carbon atoms and cycloalkyl,

and M is a substituent selected from the group consisting of Na, K, Li, NH_4 and alkylammonium.

3. A composition of matter comprising gelatin containing a coating aid corresponding to the following formula:

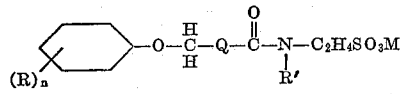


In which

R is branched alkyl of 8 to 16 carbon atoms and

R′ is selected from the group consisting of alkyls of 1 to 4 carbon atoms and cycloalkyl.

4. A gelatin-silver halide photographic emulsion containing a coating aid corresponding to the following formula:



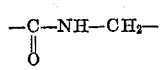
In which

R is branched alkyl,

$(R)_n$ totals 8 to 16 carbon atoms,

n is an integer of 1 to 2,

Q is selected from the group consisting of a bond and



R′ is selected from the group consisting of alkyls of 1 to 4 carbon atoms and cycloalkyl,

and M is a substituent selected from the group consisting of Na, K, Li, NH_4 and alkylammonium.

5. A composition of matter comprising gelatin containing therein as a coating aid the sodium salt of N-p-tert.-octylphenoxyacetyl-N-cyclohexyltaurine.

6. A gelatin-silver halide photographic emulsion containing therein as a coating aid the sodium salt of N-p-tert.-octylphenoxyacetyl-N-cyclohexyltaurine.

7. A composition of matter comprising gelatin containing therein as a coating aid the sodium salt of N-p-tert.-octylphenoxyacetyl-N-methyltaurine.

8. A gelatin-silver halide photographic emulsion containing therein as a coating aid the sodium salt of N-p-tert.-octylphenoxyacetyl-N-methyltaurine.

9. A composition of matter comprising gelatin containing therein as a coating aid the sodium salt of N-pentadecylphenoxyacetyl-N-methyltaurine.

10. A composition of matter comprising gelatin containing therein as a coating aid the sodium salt of N-p-tert.-octylphenoxyacetylglycyl-N-methyltaurine.

11. A composition of matter comprising gelatin containing therein as a coating aid the sodium salt of N-2,4-di(tert.amyl)phenoxyacetyl-N-cyclohexyltaurine.

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