United States Patent

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[54] **PHOTOGRAPHIC COATING FORMULATION**
7 Claims, No Drawings

[52] **U.S. Cl.** **96/114.5,** 96/94, 106/125
[51] **Int. Cl.** **G03c 1/38**
[50] **Field of Search** 96/84, 114.5, 94; 106/125

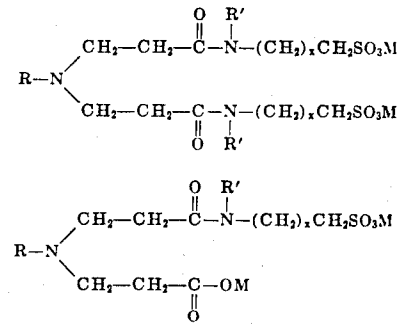
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ABSTRACT: Novel amphoteric surfactants having the formulas:



wherein R is an aliphatic hydrocarbon radical having from eight to 20 carbon atoms, R′ is selected from the group consisting of hydrogen, and alkyl radicals having from one to four carbon atoms, X is an integer of one–five and M is hydrogen, ammonium, nitrogen containing bases such as diethanolamine, triethanolamine, triethylamine, morpholine and piperidine, an alkali metal, and are used as wetting agents in gelatin coating compositions and photographic gelatin silver halide emulsions.

PHOTOGRAPHIC COATING FORMULATION

This invention relates to photography, and more particularly, to a new class of coating aids or wetting agents useful in coating and washing photographic material such as film or paper.

The use of wetting agents in the coating of photographic materials is well known. A number of such wetting agents have been recommended and used in conjunction with emulsions, dye washes, rinses, noncurling (NC) layers and antihalation layers. These wetting agents include saponin which is derived from vegetable sources and a considerable number of synthetic surfactants of the anionic, cationic and nonionic type. These known surfactants have been found not to be entirely satisfactory because of the formation of repellency spots, mottle, streakiness and/or air bells in the coating. Especially harmful is the formation of air bells which are not present in the original film but appear only during or after development.

Therefore, it is an object of this invention to provide a surfactant for use in photography that is free of the drawbacks of the prior art wetting agents.

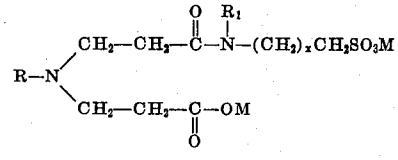
A further object of this invention is to provide a wetting agent for use in photography which has superior coating qualities and does not produce air bells in the coating.

Still another object of the instant invention is to provide a coating agent for use in photography which has antistatic properties.

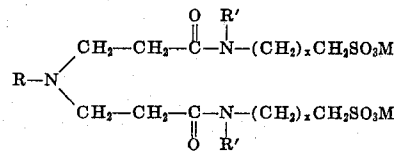
Other objects will be apparent from the following description.

The objects and advantages of this invention are accomplished by incorporating in a photographic coating composition an amphoteric surfactant which is a β-alanine derivative and characterized by one of the following formulas;

FORMULA 1



FORMULA 2



In these formulas, R represents an aliphatic hydrocarbon chain of from eight to 20 carbon atoms such as octyl, nonyl, decyl, undecyl, dodecyl, tridecyl, tetradecyl, pentadecyl, hexadecyl, heptadecyl and octadecyl; R′ represents hydrogen or an alkyl radical containing from one to four carbon atoms such as methyl, ethyl, propyl or butyl; *x* is an integer of from one to five and M represents hydrogen, ammonium, nitrogen containing bases such as diethanolamine, triethanolamine, triethylamine, morpholine or piperidine, an alkali metal.

These compounds have the additional advantage of permitting "wet-on-wet" coatings which means that they have the ability to facilitate the spreading of a colloidal layer over a previously coated emulsion layer which is chilled, but still wet, thus eliminating an additional coating pass and an extra drying operation.

The coating compositions in which the novel surfactant may be incorporated may be any of the coating compositions used in the photographic art; for example, those used for the preparation of photographic emulsions, gelatin surface coatings, NC layers, dye washes, rinses, antihalation layers, dyes and antistatic layers.

The wetting agents of this invention may be used in amounts ranging from 0.02 to 0.5 percent by volume based on the total volume of the coating solution. Based on the amounts of gelatin used, the alanine derivatives used in accordance with my invention are added to the liquid photographic emulsions or other aqueous gelatin dispersions or solutions in proportions ranging from 1–40 grams per kilogram of dry gelatin emulsion. Expressed in terms of coated areas, the effective amounts range from 0.1 to 5 milligrams per square foot of coated area.

The process used in coating the solutions containing the novel wetting agent of this invention may be any one of the known processes used in this art; such as, air knife, applicator pan, extrusion or dip pan coating. In the coating operations with the surfactants of this invention, both paper and film supports may be utilized. Suitable film supports include cellulose esters such as cellulose acetate, cellulose propionate, cellulose acetate-butyrate; vinyl esters such as the copolymers of vinyl acetate and vinyl chloride, polystyrene, polycarbonate and the polymers derived from terephthalates.

The β-alanines derivatives of my invention, when used alone, have satisfactory photographic properties; they do not cause fog or loss of speed and prevent the formation of air bells. These wetting agents are especially useful in permitting coating operations at very high speeds.

The novel wetting agents of this invention may be used as a coating aid, either alone or in conjunction with any known surfactant or wetting agent; such as, the taurides, protein condensation products of fatty acids, saponin or polyoxyethylene derivatives of long-chain fatty acids and alcohols.

We have found that compounds of the above type may be produced by the reaction of a primary amine having the requisite number of carbon atoms, with an ester of acrylic acid such as methyl acrylate. As disclosed in U.S. Pat. No. 2,468,012 the amino propionate ester thus produced may be hydrolyzed to the free acid or may be converted to any of the salts mentioned above.

The condensation between the amine and the acrylic ester proceeds readily without any catalyst, the amine itself being sufficiently basic to catalyze the reaction. The condensation is desirably carried out at a temperature of 25° to 30° C., and since the reaction is exothermic it is usually necessary to cool the reaction mixture to maintain the desired temperature range.

The hydrolysis of the condensation product may be carried out by adding this material slowly to a slight excess of a boiling solution of aqueous alkali and maintaining the resulting mixture at the boiling point with vigorous stirring until a clear solution is formed.

The present invention will now be described by reference to the following specific examples. Such examples are presented for purposes of illustration only, and the present invention is in no way to be deemed as limited thereto.

EXAMPLE I

To a solution for the preparation of an NC layer containing 10 percent gelatin and a small amount of a dye was added 0.075 percent of the N-ethylsulfomethyl carbomate derivative of N-pentadecyl-β-iminopropionate. The NC layer was coated at a speed of 75 feet per minute. The coating was found to be very uniform and free of repellency spots, mottle and streakiness.

EXAMPLE II

To a silver halide emulsion containing gelatin as the carrier material was added 0.2 percent of the N-methylsulfoethyl carbomate derivative of N-lauryl-β-iminopropionate. The emulsion was coated at a speed of 120 feet per minute and chilled; a 4 percent gelatin solution containing 0.2 percent of the same N-dodecyl-β-aminodipropionate was coated over this chilled

emulsion in the same pass and at the same web speed of 120 feet per minute. The coating was found to be very uniform and free of repellency spots, mottle and streakiness. The photographic properties were excellent, and neither fog nor loss of speed was observed.

A similar emulsion containing saponin as the sole surfactant could not be coated at a speed of more than 60 feet per minute because skipping occurred. Wet-on-wet coating proved impossible.

EXAMPLE III

To one liter of a silver halide emulsion containing 50 grams of gelatin as the carrier material was added one gram of the N-methylsulfoethyl carbamate derivative of N-lauryl-β-iminopropionate (sodium-N-lauryl-β-aminopropionate). This emulsion was coated at a speed of 120 feet per minute. The coating was found to be very uniform and free of repellency spots, mottle and streakiness. A sample of the film was developed in a black and white developer having the following formula:

Metol	grams	1.5
Sodium sulfite, anhydrous	do	45.0
Sodium bisulfite	do	1.0
Hydroquinone	do	3.0
Sodium carbonate, monohydrated	do	6.0
Potassium bromide	do	0.8
Water to make	liter	1.0

The developed film was then fixed, washed and dried. It was free of air bells whereas an emulsion coated with saponin as the surfactant showed considerable air bells.

As may be noted from the foregoing examples, photographic compositions comprising the compounds of interest herein, provide excellent results with regard to a lack of air bells, repellency spots, mottle and streakiness.

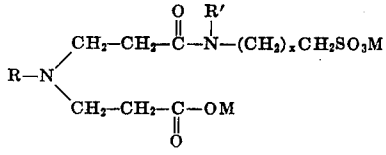
EXAMPLE IV

One mol of N-lauryl-β-aminodipropionic acid was reacted with 2 mols of phosgene to form the dichloro formate derivative thereof. The resulting product was then added simultaneously with aqueous sodium hydride to an aqueous solution of N-methyl taurine in a manner such that the pH was maintained between 10 and 11. The resulting solution was then stripped under vacuum to remove water, and the residue containing the N-methyl sulfomethyl derivative of N-lauryl-β-imino dipropionate was filtered.

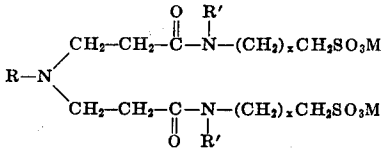
Many modifications will be apparent to persons skilled in the art. Therefore, we do not intend to be limited in the patent granted except as necessitated by the appended claims.

What is claimed is:

1. A coating solution for use in coating a photographic base comprising gelatin as the colloidal carrier material and containing as a wetting agent in an amount effective to produce wetting characteristics, a compound selected from the group consisting of the compounds having the following general formulas:



and

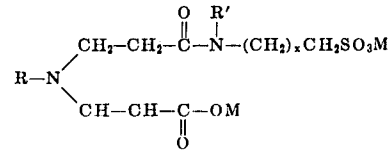


wherein R is an aliphatic hydrocarbon radical having from eight to 20 carbon atoms, R′ is selected from the group consisting of hydrogen, and alkyl radicals having from one to four carbon atoms and M is selected from hydrogen, ammonium, alkali metal and organic amine cations, and x is an integer of one to five.

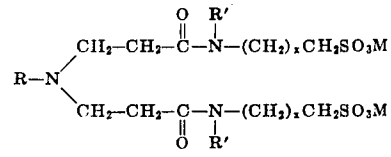
2. A coating solution as recited in claim 1 wherein said wetting agent is the N-methyl sulfoethyl carbamate derivative of N-lauryl-β-iminodipropionate.

3. A coating solution as recited in claim 1 wherein the wetting agent is present in an amount ranging from 0.02 percent to 0.5 percent by weight based on the volume of the solution.

4. A liquid photographic gelatin silver halide emulsion containing in an amount effective to produce wetting characteristics a wetting agent selected from the group consisting of the compounds having the following general formulas;



and

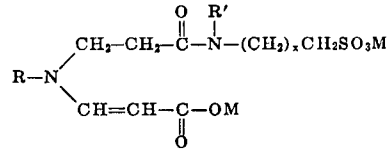


wherein R is an aliphatic hydrocarbon radical having from eight to 20 carbon atoms; M is selected from the group consisting of hydrogen, an alkali metal, ammonium and an organic amine cation; and x is an integer of one to five.

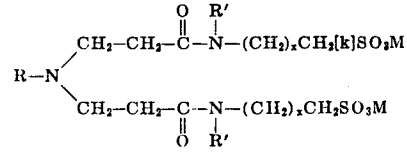
5. A photographic silver halide emulsion according to claim 8 and wherein said wetting agent is the N-methyl sulfoethyl carbamate derivative of N-lauryl-β-iminodipropionate.

6. A liquid photographic silver halide emulsion as recited in claim 4 wherein the amount of the wetting agent present in the emulsion is from 0.02 percent to 0.5 percent by weight based on the volume of the emulsion to be coated.

7. A photographic element carrying on a suitable base a layer comprising gelatin as the colloidal carrier material said layer containing in an amount effective to produce wetting characteristics a β-alanine derivative selected from the group consisting of those compounds having the following general formulas;



and



wherein R is an aliphatic hydrocarbon radical having from eight to 20 carbon atoms; M is selected from the group consisting of hydrogen, alkali metal, ammonium and an organic amine cation; and x is an integer of one to five.