

UNITED STATES PATENT OFFICE

2,190,645

SPREADING AGENT FOR PHOTOGRAPHIC EMULSIONS

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No Drawing. Application December 29, 1937
Serial No. 182,303

7 Claims. (Cl. 95-9)

This invention relates to wetting or spreading agents for gelatin solutions and more particularly to wetting or spreading agents for use in photographic gelatin and emulsions.

When photographic gelatin emulsions or solutions are coated in multiple coatings on supports such as cellulose nitrate or cellulose acetate, it is desirable for the gelatin solution or emulsion to wet the previously coated emulsion or gelatin layer on which it is coated, so that a uniform coating is obtained. The only material previously found to be satisfactory for this purpose is saponin. This material has the disadvantages that it is expensive, in quantity it must be obtained abroad, and it is a wood extract whose quality varies considerably and whose active ingredient is unknown. It is therefore desirable to use other substances, preferably pure organic compounds of known structure, as spreading agents for gelatin or emulsions.

It is therefore an object of the present invention to provide a novel wetting or spreading agent for photographic gelatin solutions or emulsions. A further object is to provide a wetting or spreading agent which is a chemical compound whose constitution is definitely known and which may therefore be purified, and which may be cheaply obtained in this country.

These objects are accomplished by incorporating in the gelatin or gelatin emulsions a small amount of a chemical which may be (1) an ester formed either from a polyhydroxy alcohol having less than six hydroxyl groups and a higher fatty acid having from eight to fifteen carbon atoms, or from a monobasic hydroxy acid and a higher fatty alcohol having from eight to fifteen carbon atoms, or (2) an ether which may be formed from a polyhydroxy alcohol, having less than six hydroxyl groups and a higher fatty alcohol having from eight to fifteen carbon atoms.

The esters which may be used for this purpose are those formed by the reaction of a polyhydroxy alcohol having less than six hydroxyl groups such as glycerol, glycol or glucose with a higher fatty acid having from eight to fifteen carbon atoms such as capric, undecylic, lauric, or myristic acid, or an ester formed from a mono-basic hydroxy acid such as glycollic, lactic, hydracrylic, or glyceric acid and a higher fatty alcohol having from eight to fifteen carbon atoms, such as decyl, undecyl, lauryl, or capryl alcohol. The esters of the first type formed in this way are compounds such as glycol mono laurate, glycerol monocaprate, glycerol monoundecylate, glycerol monolaurate, glycerol monomyristate, and glucose monolaurate. The esters of the second type are compounds such as decyl glycolate, lauryl glycolate, undecyl glycerate, or capryl glycerate, etc. The ethers are formed by reaction of the sodium salt of a higher fatty alcohol (eight to fifteen

carbon atoms) with a chloroalcohol. For example, alphasglyceryl lauryl ether is formed from sodium laurylate and alpha glycerol mono chlorohydrin.

The esters may be formed, for example, as follows: (J. C. S. 1935—T. P. Hilditch & J. G. Riggs, 1774-8).

Preparation of glycerol α -monolaurate

Twenty grams of lauric acid are mixed with an equal weight of phenol and 74 grams of glycerol in a round-bottom flask which is fitted with a cork carrying both an inlet tube well below the surface of the liquid and an exit tube connected to a reflux condenser. The flask is heated in an oil bath to about 180° C., meanwhile passing a gentle current of dry CO₂ through the reaction mixture for agitation and dehydration.

After one hour the phenol is removed by steam distillation and the non-volatile material dissolved in ether and washed free from unchanged fatty acid with aqueous potassium carbonate, followed by water to remove the soap. The mixed glycerols are then freed from ether and the residue crystallized from a small amount of ether. The yield of glycerol-monolaurate, white crystals melting at 62° C., is about 27.4 grams, which is 90% of the theoretical value.

The ethers may be formed, for example, as follows:

Preparation of glycerol α -monolauryl ether

In a 3-liter, round-bottom, 3-neck flask equipped with a dropping funnel, reflux condenser, and mechanical stirrer, is placed 365 grams (1.95 moles) of lauryl alcohol and about 1500 cc. of ligroin (B. P. 90°-120°). The solution is heated to refluxing and 37.2 grams (1.6 moles) of sodium is added in small pieces. Vigorous stirring is maintained to keep the sodium finely divided. When all of the sodium has reacted, 195 grams (2.1 moles) of glycerol monochlorohydrin is added slowly through the dropping funnel. Heating is continued for about two hours, after which the reaction mixture is filtered to remove crystallized salt. The ligroin is distilled off and the following fractions, under reduced pressure, are taken:

	Grams
(A) B. P. 120-185°/7 min.-----	255
(Mostly lauryl alcohol.)	
(B) B. P. 185-209°/7 min.-----	92
(Mostly at 199°-209°/7 min.)	
(C) B. P. 209-245°/7 min.-----	34
(Glycerol α -monolauryl ether.)	
Residue -----	95
Salt-----	100
Total recovery-----	576

These compounds are dissolved in a suitable

solvent which does not adversely affect the gelatin or the sensitivity of the emulsion made from it and are then added to the gelatin solution or emulsion. A suitable solvent for this purpose is ethyl alcohol.

The esters or ethers used as spreading agents according to my invention may be added to the gelatin or emulsion in varying amounts. A suitable concentration is one part of spreading agent to 100,000 parts of gelatin solution or photographic emulsion containing each about 5% gelatin. The amount used may vary, however, from one part of spreading agent in 10,000 parts of gelatin solution or photographic emulsion to one part of spreading agent in 400,000 parts of gelatin solution or photographic emulsion containing each about 5% gelatin. In each case the gelatin content of the gelatin solution or photographic emulsion may vary from about 3% to about 10% gelatin.

The following example which is by way of illustration only will illustrate one method of forming a gelatin layer according to my invention:

To 1000 grams of gelatino-silver bromide emulsion there is added a solution of 10 cc. of ethyl alcohol containing .01 gram of glycerol monolaurate. This solution is thoroughly mixed and is coated on a hydrolyzed cellulose acetate support layer, which has been coated in the usual way with subbing layers, for example, of cellulose nitrate and gelatin. The emulsion containing the spreading agent is found to readily wet and adhere to the subbing layers of the coated support and after partial drying is susceptible of receiving further emulsion layers.

It is to be understood that the examples and ranges of proportions referred to in the above specification are illustrative only and that my invention is to be taken as limited only by the scope of the appended claims.

I claim:

1. A photographic film comprising a support having thereon a subbing layer containing gelatin, and over said subbing layer a gelatin emulsion layer containing as a spreading agent a compound selected from the group consisting of mono-esters formed from a polyhydroxy alcohol containing less than six hydroxyl groups and a higher fatty acid containing from eight to fifteen carbon atoms, esters formed from a monobasic hydroxy acid and a higher fatty alcohol containing from eight to fifteen carbon atoms, and mono-ethers formed from a polyhydroxy alcohol containing less than six hydroxyl groups and a higher fatty alcohol containing from eight to fifteen carbon atoms.

2. A photographic film comprising a support having thereon a subbing layer containing gelatin, and over said subbing layer a gelatin emulsion layer containing as a spreading agent a mono-ester formed from a polyhydroxy alcohol containing less than six hydroxyl groups and a

higher fatty acid containing from eight to fifteen carbon atoms.

3. A photographic film comprising a support having thereon a subbing layer containing gelatin, and over said subbing layer a gelatin emulsion layer containing as a spreading agent an ester formed from a monobasic hydroxy acid and a higher fatty alcohol containing from eight to fifteen carbon atoms.

4. A photographic film comprising a support having thereon a subbing layer containing gelatin, and over said subbing layer a gelatin emulsion layer containing as a spreading agent a mono-ether formed from a polyhydroxy alcohol containing less than six hydroxyl groups and a higher fatty alcohol containing from eight to fifteen carbon atoms.

5. A photographic element comprising a support having thereon a subbing layer containing gelatin and over said subbing layer a gelatin layer containing as a spreading agent a compound selected from the group consisting of mono-esters formed from a polyhydroxy alcohol containing less than six hydroxyl groups and a higher fatty acid containing from eight to fifteen carbon atoms, esters formed from a monobasic hydroxy acid and a higher fatty alcohol containing from eight to fifteen carbon atoms, and mono-ethers formed from a polyhydroxy alcohol containing less than six hydroxyl groups and a higher fatty alcohol containing from eight to fifteen carbon atoms.

6. A photographic element comprising a support having thereon a subbing layer containing gelatin and cellulose nitrate and over said subbing layer a gelatin layer containing as a spreading agent a compound selected from the group consisting of mono-esters formed from a polyhydroxy alcohol containing less than six hydroxyl groups and a higher fatty acid containing from eight to fifteen carbon atoms, esters formed from a monobasic hydroxy acid and a higher fatty alcohol containing from eight to fifteen carbon atoms, and mono-ethers formed from a polyhydroxy alcohol containing less than six hydroxyl groups and a higher fatty alcohol containing from eight to fifteen carbon atoms.

7. A photographic film comprising a support having coated thereon a plurality of emulsion layers each containing as a spreading agent a compound selected from the group consisting of mono-esters formed from a polyhydroxy alcohol containing less than six hydroxyl groups and a higher fatty acid containing from eight to fifteen carbon atoms, esters formed from a monobasic hydroxy acid and a higher fatty alcohol containing from eight to fifteen carbon atoms, and mono-ethers formed from a polyhydroxy alcohol containing less than six hydroxyl groups and a higher fatty alcohol containing from eight to fifteen carbon atoms.

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