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DISPERSION OF GELATIN

George S. Babcock, Rochester, N. Y., assignor to Eastman Kodak Company, Rochester, N. Y., a corporation of New Jersey

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This invention relates to the dispersion of gelatin in solution, and more particularly to the provision of a new type of gelatin solution adapted for the subbing or coating of cellulose derivative film base or similar materials.

This application is in part a continuation of my co-pending application Serial No. 27,408, filed June 19, 1935.

As is well known, a photographic film comprises a film base or support composed of a cellulose derivative material such as cellulose nitrate or cellulose acetate, and having superimposed thereon a layer of a light-sensitive gelatino-silver halide emulsion. In order properly to prepare the surface of the cellulose derivative material for permanent adhesion of the emulsion coating, it is necessary to coat the cellulose derivative material with a layer of gelatin. The solutions employed for this purpose comprise gelatin dissolved in a mixture of water and certain organic solvents, such as acetone. Such solutions also contain a dispersing agent for the gelatin, such as acetic acid, it having been found necessary to lower the pH of the solution by means of acid before the gelatin can properly be dispersed.

The use of acetic acid or other acid components, even though used in small amounts of the order of 1 or 2% of the solution, have been costly from the standpoint of sacrifice in the quality of the finished film, because by lowering the pH of the solution applied to the base, the speed of emulsion is lowered, sometimes with accompanying changes in the so-called "gamma" and fog characteristics thereof. In addition, gelatin subbing solutions of this type are unstable after standing for a short period of time and become cloudy or opalescent in appearance due to the reluctance of the gelatin to remain completely dissolved in solutions containing relatively high percentages of organic solvents. A typical prior art subbing solution of this character is one having the following composition:

	Percent by weight
Gelatin.....	1.2
Acetic acid.....	1.2
Water.....	3.0
Acetone.....	70.0
Ethyl alcohol (95%).....	24.6

This invention has as its principal object to provide a novel means of dispersing gelatin in a solution or colloidal suspension thereof. Another object is to provide a clear gelatin solution in which the gelatin is substantially completely dispersed without the use of the amount of acids or

other chemicals heretofore considered necessary. A further object is to provide such a solution which will not become cloudy or opalescent even after prolonged standing. A still further object is to provide a stable gelatin subbing solution adapted for use in coating or subbing a photographic film support to provide a proper surface thereon for emulsion coating. Another object is to provide a means for eliminating fog and reduction in speed of an emulsion when applied to a gel subbed support due to ingredients present in the gel sub layer. Other objects will appear hereinafter.

These objects are accomplished by the following invention which, in its broader aspects, comprises the use of the material sold under the registered trade-mark, "Methyl Cellosolve" (the mono-methyl ether of ethylene glycol) as a substitute for part or all of the organic solvents, such as acetone or alcohol, heretofore employed in subbing solutions of the character herein described. I have found, contrary to what would be expected from the results obtained with the use of solvents such as acetone and alcohol, that the presence of "Methyl Cellosolve" in such a solution makes possible substantially complete dispersion of the gelatin, and that such solutions will not become cloudy or opalescent even after standing for long periods of time. I have also found that the amount of acid dispersing agent heretofore considered necessary can be either eliminated entirely, or reduced by approximately 60% and still obtain a completely satisfactory degree of dispersion of the gelatin.

In the following examples and description, I have set forth several of the preferred embodiments of my invention, but they are included merely for purposes of illustration and not as a limitation thereof.

Example 1

A clear gelatin solution adapted for the subbing of cellulose nitrate, cellulose acetate, cellulose acetate propionate and other cellulose derivative film supports is prepared by mixing the following ingredients in the indicated proportions:

	Percent by weight
Gelatin.....	1.35
Acetic acid.....	0.56
Water.....	2.00
"Methyl Cellosolve".....	5.00
Methyl alcohol.....	15.00
Acetone.....	76.09

Example 2

Other examples of gelatin subbing solutions prepared in accordance with my invention may have the following compositions:

	Percent by weight
5 Gelatin.....	1.35
Acetic acid.....	0.56
Water.....	2.00
10 "Methyl Cellosolve".....	10.00
Methyl alcohol.....	10.00
Acetone.....	76.09

Example 3

	Percent by weight
15 Gelatin.....	1.35
Acetic acid.....	0.56
Water.....	2.00
"Methyl Cellosolve".....	20.00
20 Acetone.....	76.09

Example 4

	Percent by weight
Gelatin.....	1.35
Acetic acid.....	0.56
25 Water.....	4.00
"Methyl Cellosolve".....	30.00
Acetone.....	64.09

Example 5

	Percent by weight
30 Gelatin.....	1.35
Acetic acid.....	0.56
Water.....	4.00
"Methyl Cellosolve".....	40.00
35 Acetone.....	54.09

Example 6

A clear gelatin solution is prepared by mixing the following ingredients in the indicated proportions:

	Percent by weight
Gelatin.....	0.67
Water.....	10.00
45 Acetone.....	20.00
"Methyl Cellosolve".....	69.06
Acetic acid.....	0.27

Such a solution is particularly suitable for subbing of cellulose nitrate film support to prepare it for emulsion coating.

Example 7

A solution particularly adapted for the subbing of cellulose acetate film support has the composition indicated below:

	Percent by weight
Gelatin.....	1.2
60 Water.....	5.8
Alcohol.....	26.0
"Methyl Cellosolve".....	66.5
Acetic acid.....	0.5

Example 8

A solution particularly adapted for the subbing of cellulose acetate-propionate film support has the composition indicated below:

	Percent by weight
70 Gelatin.....	1.2
Water.....	2.0
Alcohol.....	26.0
"Methyl Cellosolve".....	70.3
75 Acetic acid.....	0.5

The application of the above compositions to a film support may be carried out by any well-known subbing or coating technique. One of such methods is to run the support through an immersion type hopper containing the solution. Another standard method is by bead application. With some types of support such as those composed of cellulose acetate propionate, for example, it has been found advantageous to slightly roughen the surface of the material prior to the application of the subbing solution. This may conveniently be carried out by wetting the surface of the support with an appropriate solvent and passing it over a roll having a very slightly etched surface. The roughening thus produced is, of course, microscopic in character and does not in any way adversely effect the optical properties of the film, merely providing a slightly toothed surface to which the gelatin of the subbing solution will cling with a higher degree of adherence than would otherwise be the case.

Although I have referred in the above examples to subbing solutions containing definite percentages of "Methyl Cellosolve," it is to be understood that the proportions of this ingredient may vary widely for different formulas and that the additional solvents employed will be selected upon the basis of the particular type of cellulose derivative film support to which the solution is to be applied. I may, for example, employ as little as 5% of "Methyl Cellosolve" and still obtain a highly satisfactory gel-subbing solution which will not deteriorate or become opalescent, even on long standing. On the other hand, I may employ as much as 95% or more and obtain satisfactory results. In general, the greater the proportion of this ingredient, the greater is the stability of the resulting solution.

Likewise, the amount of gelatin employed in a given composition may vary, depending upon the conditions of application and the specific results desired. In general, I prefer to use not less than about 5% and up to about 2%, or slightly more, for most practical purposes. Also, in general accordance with my invention, I prefer to keep the acid or other dispersing agent down to a minimum in order to obtain a substantially neutral solution. A satisfactory range of acetic acid, for example, is from 0.27% to 0.5%. Inasmuch as the specific proportions of the various ingredients to be used in a given subbing solution are more or less within the skill and experience of the operator, no hard and fast rule as to the proportions of the various ingredients which will cover every case can be laid down. As a matter of fact, my invention is based on the broad discovery that the use of "Methyl Cellosolve," even in small amounts, in subbing solutions give such solutions heretofore unknown and unexpected properties. I have found that the proportion of other organic solvents such as acetone and alcohol may be reduced by the substitution of "Methyl Cellosolve" and that my solutions are far superior, in keeping qualities and in resistance to the development of opalescence, to any subbing solutions heretofore employed. In addition, such solutions are quite unexpectedly satisfactory from the standpoint of producing flexible (non-brittle) film support, a result which is difficult of attainment when using subbing solutions containing only relatively strong subbing solvents.

Similarly, the amount of water present in the

gel subbing solutions herein described may vary within rather wide limits. I may, for example, employ anywhere from about 2% to about 94% or 95% of water, depending upon the character and amounts of the other ingredients of the composition.

Gelatin solutions prepared in accordance with the procedure outlined above are adapted for application to an untreated cellulose derivative film support or to one which has been previously subbed with a solution of another cellulose derivative. For example, a gelatin solution prepared as indicated in Example 6 and containing suitable proportions of the various ingredients may be applied directly to a cellulose nitrate film support or to a cellulose acetate film support which has been previously subbed or coated with a thin layer of cellulose nitrate, and similarly for other types of subbed or unsubbed cellulose derivative film supports.

As previously indicated, the compositions herein described have exceptional keeping qualities. I have found, for example, that the gelatin solutions of my invention have, in some instances, almost double the life of the ordinary prior art solutions of this type, while in other instances the solution may be kept for a practically indefinite time without the development of cloudiness or opalescence.

Another outstanding feature of my invention is the fact that by the use of "Methyl Cellosolve" as an ingredient of the subbing formula, the amount of acid or other dispersing agent required to keep the gelatin in solution is markedly reduced and in some cases practically no acid is required. In other words, the use of "Methyl Cellosolve" makes possible the production of a practical subbing solution of almost complete neutrality. This has an important effect upon the completed photographic film in that it cuts down the amount of acid present in the material to which the emulsion is applied, and this in turn reduces the possibility of the undesirable reduction in speed of the emulsion with accompanying changes in both the gamma and fog characteristics which occurs when the film base contains any appreciable amount of acid.

A still further advantage of the gelatin solution of my invention is that film supports subbed with such compositions retain nearly their natural flexibility and do not develop brittleness after the subbing operation to the same extent as film support subbed with the ordinary types of gel subbing solutions not containing "Methyl Cellosolve".

What I claim is:

1. A stable, substantially neutral gelatin solution suitable for subbing a cellulose derivative film support, which comprises .5%-2% by weight of gelatin, a weakly acidic dispersing agent for the gelatin in an amount sufficient to bring it into solution, and a solvent comprising 5-95% of the monomethyl ether of ethylene glycol, the balance being water.
2. A stable, substantially neutral gelatin solution suitable for subbing a cellulose derivative film support, which comprises .5%-2% by weight of gelatin, a weakly acidic dispersing agent for the gelatin in an amount sufficient to bring it into solution, and a solvent comprising 5%-95% of the monomethyl ether of ethylene glycol, the balance being a mixture of water and an organic solvent for the cellulose derivative.
3. A stable, substantially neutral gelatin solution suitable for subbing a cellulose derivative film

support, which comprises .5%-2% by weight of gelatin, a weakly acidic dispersing agent for the gelatin in an amount sufficient to bring it into solution, and a solvent comprising 5%-95% of the monomethyl ether of ethylene glycol, the balance being a mixture of alcohol and water.

4. A stable, substantially neutral gelatin solution suitable for subbing a cellulose derivative film support, which comprises .5%-2% by weight of gelatin, a weakly acidic dispersing agent for the gelatin in an amount sufficient to bring it into solution, and a solvent comprising 5%-95% of the monomethyl ether of ethylene glycol, the balance being a mixture of acetone and water.

5. A stable, substantially neutral gelatin solution suitable for subbing a cellulose derivative film support, which comprises .5%-2% by weight of gelatin, a weakly acidic dispersing agent for the gelatin in an amount sufficient to bring it into solution, and a solvent comprising 5%-95% of the monomethyl ether of ethylene glycol, the balance being a mixture of acetone, methyl alcohol and water.

6. A stable, substantially neutral gelatin solution suitable for subbing a cellulose derivative film support, which comprises .5%-2% by weight of gelatin, a trace of a weak acid, and a solvent comprising 5%-95% of the monomethyl ether of ethylene glycol, the balance being water.

7. A stable, substantially neutral gelatin solution suitable for subbing a cellulose derivative film support, which comprises .5%-2% by weight of gelatin, .27%-56% of acetic acid, and a solvent comprising .5%-95% of the monomethyl ether of ethylene glycol, the balance being water.

8. A stable, substantially neutral gelatin solution suitable for subbing a cellulose derivative film, containing .5%-2% by weight of gelatin, a weakly acidic dispersing agent for the gelatin in an amount sufficient to bring it into solution, and a solvent comprising 66.5%-95% by weight of the monomethyl ether of ethylene glycol, the balance being water.

9. A stable, substantially neutral gelatin solution suitable for subbing a cellulose derivative film containing .5%-2% by weight of gelatin, a weakly acidic dispersing agent for the gelatin in an amount sufficient to bring it into solution, and a solvent comprising 95% by weight of the monomethyl ether of ethylene glycol, and the balance being water.

10. A stable, substantially neutral gelatin solution suitable for subbing cellulose derivative film, containing .5%-2% by weight of gelatin, a weakly acidic dispersing agent for the gelatin in an amount sufficient to bring it into solution, and a solvent comprising 66.5%-95% by weight of the monomethyl ether of ethylene glycol, the balance being water.

11. A stable, substantially neutral gelatin solution suitable for subbing a cellulose derivative film, containing .5%-2% by weight of gelatin, .27%-5% by weight of acetic acid, a solvent comprising 66.5%-95% by weight of monomethyl ether of ethylene glycol, the balance being water.

12. A stable, substantially neutral gelatin solution suitable for subbing cellulose derivative film, containing .5%-2% by weight of gelatin, a weakly acidic dispersing agent for the gelatin in an amount sufficient to bring it into solution, and a solvent comprising 66.5%-95% by weight of the monomethyl ether of ethylene glycol, the balance being a mixture of water and an organic solvent for the cellulose derivative.

13. A stable, substantially neutral gelatin solu-

tion suitable for subbing cellulose derivative film, containing .5%-2% by weight of gelatin, a weakly acidic dispersing agent for the gelatin in an amount sufficient to bring it into solution, and a solvent comprising 66.5%-95% by weight of the monomethyl ether of ethylene glycol, the balance being a mixture of alcohol and water.

14. A stable, substantially neutral gelatin solution suitable for subbing cellulose derivative film, containing .5%-2% by weight of gelatin, a weakly acidic dispersing agent for the gelatin in an amount sufficient to bring it into solution, and a solvent comprising 66.5%-95% by weight of the monomethyl ether of ethylene glycol, the balance being a mixture of acetone and water.

15. A stable, substantially neutral gelatin solution suitable for subbing a cellulose nitrate film having the following approximate composition by weight:

	Percent
Gelatin	0.67
Water	10.0
Acetone	20.0
Monomethyl ether of ethylene glycol	69.06
Acetic acid	0.27

16. A stable, substantially neutral gelatin solution suitable for subbing a cellulose acetate film having the following approximate composition by weight:

	Percent
Gelatin	1.2
Water	5.8
Alcohol	26.0
Monomethyl ether of ethylene glycol	66.5
Acetic acid	0.5

17. A stable, substantially neutral gelatin solution suitable for subbing a cellulose acetate propionate film having the following approximate composition by weight.

	Percent
Gelatin	1.2
Water	2.0
Alcohol	26.0
Monomethyl ether of ethylene glycol	70.3
Acetic acid	0.5

GEORGE S. BABCOCK.