



Resin coagulation grain wash process

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In the preparation of photographic silver halide emulsions, a preliminary step is preparation of photosensitive silver halide by the double decomposition reaction between a soluble silver salt, such as silver nitrate, and a soluble halide salt such as ammonium halide or an alkali metal halide, usually the potassium halides. As the insoluble silver halide is formed, it is necessary to control formation of silver halide grains by means of a peptizer which regulates grain growth and dispersion of the silver halide grains in the emulsion. Characteristics of grain growth and grain dispersion during preparation of photographic emulsions will have critical influence on the basic sensitometric and photographic properties of the emulsion such as photographic speed, contrast, graininess, and acutance. Gelatin has been the most generally used peptizer for photographic emulsions and while it remains a useful peptizer, there are certain disadvantages inherent in gelatin, for example, gelatin is subject to attack by bacteria and fungus which sometimes damage photographic emulsions. Also, since gelatins are derived from animal sources, control of uniformity of gelatin compositions used for peptizers has been a serious problem.

A class of synthetic peptizers has recently been described in Belgian Patent 727,604 which permit easier and more uniform control of composition and exhibit relative immunity from decay. Certain members of this class, however, have presented some difficulties in the coagulation and washing procedures which are an important part of the preparation of photographic silver halide emulsions. Particular difficulty has been experienced with synthetic polymeric peptizers containing anionic sulfoxy groups. It has been necessary up until now to employ organic or inorganic substances for coagulation which subsequently contaminate the system and then to shred and wash the coagulum for long periods of time, a procedure contributing substantially to the cost of the finished product.

A process has now been discovered which overcomes the difficulties of silver halide emulsions containing peptizers having anionic sulfoxy groups (i. e. groups containing sulfur atoms such as, for example, sulfates, sulfonates and the like) of the type disclosed in Belgian Patent 727,604. Silver halide emulsions precipitated in such peptizers can be readily coagulation washed by adding a small amount of gelatin and adjusting the pH of the silver halide dispersion to less than

5.0. The best results for obtaining the coagulum are achieved when the pH adjustment is preceded by the addition of gelatin to the silver halide dispersion and dilution with water.

In another and related aspect, it has been found that the addition of these synthetic peptizers to silver halide emulsions in which the grains are peptized in bone gelatin permits these silver halide grains to be coagulation washed by adjusting the pH to less than 5.0.

The optimum concentration of these peptizing agents in the precipitation medium will depend upon several factors such as the selected temperature of the precipitation medium, the selected pH of the medium, silver and halide concentrations in the medium, etc. Generally, optimum concentrations of the peptizer will be low, usually from about 0.5% to about 3% by weight, and operable concentrations can range from about 0.25% to about 5% by weight, based on total weight of the aqueous medium.

Silver halide emulsions prepared using these peptizers can contain other hydrophilic colloids added for the purpose of a film-forming binder or other purposes. Such emulsions can be sensitized chemically with any suitable chemical sensitizers or with combinations thereof, and can be spectrally sensitized, either by panchromatic sensitizing as for black-and-white negative emulsions or sensitized to selected wavelengths for specialty products such as color films and the like. These emulsions can be prepared for use in x-ray recording elements, gamma-ray recording elements, electron beam recording elements, for use in various color recording films and color printing elements, for use in conventional black-and-white photography and the like. These emulsions can also be prepared using various grain growth restrainers for making fine grain emulsions, using any of various procedures for grain ripening and the like. Generally these emulsions can be prepared and finished using any of the addenda, treatments or procedures that are using in preparing and finishing other silver halide photographic emulsions.

Example 1

A fine grain silver bromiodide emulsion (3.5 mole % iodide) is prepared by adding the aqueous halide and silver salt solutions to a kettle containing an aqueous solution of 25 grams of (1:6) 3-thiapentylacrylate-3-acryloxypropane-1-sulfonic acid sodium salt per silver mole at a pH of 6.4. Upon completion of the precipitation, 60 grams of bone gelatin per silver mole is added and then the total solution is diluted to equal volume with distilled water. When the pH of the dispersion is lowered to

4.5 with dilute aqueous sulfuric acid and the temperature lowered to 20°C, a precipitate is formed. The aqueous supernatant is removed by decantation. Distilled water is added to the coagulum and the pH readjusted to its original value causing reformation of the silver halide dispersion.

Example 2

A fine grain silver bromide emulsion, referred to as nuclear track emulsion, is prepared as described in U.S. Patent 3,025,162, column 3. Prior to coagulating the emulsion, the dispersion is diluted to an equal volume by adding distilled water and to the diluted solution is added a 20% solution of the polymer described in Example 1 above.

The pH of the dispersion is then adjusted to 4.0 with dilute sulfuric acid and a clear precipitate is formed. The precipitate is readily redispersed by adding distilled water and readjusting the pH to 6.0.

Patent applications embodying subject matter disclosed in this report may be pending in one or more countries.

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