Patented May 16, 1939

2,158,219

UNITED STATES PATENT OFFICE

2,158,219

METHOD OF PRODUCING PHOTOGRAPHIC EMULSIONS

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No Drawing. Application January 15, 1937, Serial No. 120,783. In Germany January 20, 1936

5 Claims. (Cl. 95—7)

My invention relates to photographic emulsions and more especially to the production of peptisation emulsions, in which a sediment of silver halide precipitated from a solution and sufficiently freed from the adhering solution, for instance by decanting and washing, is mixed with a binder, for instance with a gelatine solution and allowed to age.

The production of such peptisation emulsions offers great advantages, since the time-consuming and little reliable washing process can be dispensed with and since these emulsions possess a higher sensitivity than emulsions of the same order of grain size produced according to customary methods. Preferably, when emulsifying the silver halide sediment subsequently mixed with the binder, so-called peptisation agents, for instance ammonia, are added, but hitherto a comparatively too large proportion of such agent such as ammonia was required.

I have now found that in the emulsification of the silver halide sediment in a gelatine solution as well as in the ageing of the mixture of silver halide sediment and gelatine the absolute quantity of the peptisation agent such as a solution of ammonia is unimportant, the concentration of the agent such as ammonia being alone decisive. Consequently it appears inadvisable to carry through emulsification and ageing in the whole of the quantity of gelatine solution, since this would only require an unnecessarily large quantity of ammonia injurious to peptisation emulsions to obtain the ammonia concentration which is necessary and most favorable for emulsification and ageing. I have found that it is far better to emulsify and also to age the light-sensitive sediment at first only in a small part, less than one half or one fourth or one sixth, and in some case in as little as one tenth or one thirtieth or one fiftieth of the total emulsifying liquid, since I am thus enabled to effect a great saving in ammonia.

Therefore, in accordance with the present invention, I mix the silver halide sediment, which has been sufficiently freed from the adhering solution, with less than one third and preferably less than one fifth or one tenth of the total quantity of the emulsifying solution (aqueous gelatine solution) and allow it to age therein, while adding, gradually or as a whole, the larger part of the emulsifying solution only after emulsification of the sediment in the first part and after it has already been allowed to more or less age therein.

Before adding the balance of the emulsification solution, which may be added in instalments, I prefer expelling the ammonia, by evaporation or otherwise, sufficiently to ensure stability of the photographic emulsions and for this purpose I heat the first mixture of the sediment and the smaller part of the emulsifying solution under continuous stirring in a low layer, air being caused to sweep the surface of the liquid or to rise in the liquid etc., in order to facilitate the escape of the ammonia.

In the operation of my invention I may for instance proceed as follows:

Into a 10% solution of potassium bromide and potassium iodide, wherein the proportion of KBr to KJ is 25:1 and which contains 0.5% gelatine, a 10% silver nitrate solution is entered under continuous stirring in such manner that a slight excess of bromine, for instance 1%, remains over, so that the proportion of silver nitrate solution to potassium halide solution is about 100:73. After the silver halide has settled down as a sediment, the supernatant liquid is decanted and the silver halide sediment washed. Now one sixth of the 10% gelatine solution heated to 50° C. used in the preparation of the emulsion is mixed with so much of a 25% ammonia solution that the gelatine solution contains 5% ammonia. This solution is now poured under continuous stirring over the silver halide sediment. The mixture is kept thirty minutes at 50° C. and is then poured in a shallow tray, the height of the liquid being preferably not above 5 cms. The mixture is now kept another hour at about 50° C. under continuous stirring, whereby it is substantially freed from the ammonia. Now the remaining five sixths of the gelatine solution, to which no ammonia had been added, are mixed with the preliminary emulsion, so that this concentrated emulsion is now reduced to the normal concentration of about 7%. This normal emulsion may now still be allowed to age until it has reached the maximum sensitivity and may then be poured.

Obviously the proportions and temperatures mentioned in this example may be varied within wide limits, provided only that the silver halide sediment, precipitated from a silver solution and sufficiently freed from the adhering solution, is mixed first with a comparatively small part of the whole of the emulsification liquid, the emulsifying agent such as ammonia being added to the mixture and the sediment is emulsified and if desired, also allowed to age in this part, while the rest of the emulsifying solution is added only

after dispersion and after the emulsification agent such as ammonia has been sufficiently expelled, for instance by evaporation.

Various changes may be made in the details disclosed in the foregoing specification without departing from the invention or sacrificing the advantages thereof.

I claim:

1. In the process of producing photographic emulsions the steps of emulsifying a wet silver halide sediment in less than one third of the total quantity of the water and of the gelatine required to produce a photographic emulsion, ready to be poured, which contains said silver halide, allowing the silver halide to age therein in the presence of an aging agent and thereafter mixing the ripened product with the rest of the water and gelatine.

2. The process of claim 1, in which, after the silver halide sediment has been emulsified and aged in the presence of an aging agent, said agent is rendered at least partially inactive.

3. The process of claim 1, in which, after the silver halide sediment has been emulsified and aged in the presence of ammonia in a concentration in which ammonia acts as an emulsifying and aging agent, the main part of the ammonia is removed by evaporation.

4. The process of claim 1, in which, after another part of the required quantity of water and gelatine has been added, the whole is allowed to age further.

5. The process which comprises precipitating a silver salt solution with an alkali bromide solution in the presence of a small quantity of an alkali iodide and of gelatine, allowing the silver halide produced in the reaction to settle, decanting the supernatant liquid, washing the sediment, emulsifying the silver halide sediment in less than one third of the total quantity of the water and of the gelatine required to produce a photographic emulsion ready to be poured, which contains said silver halide, allowing the silver halide to age therein in the presence of an emulsifying and aging agent and thereafter mixing the ripened product with the rest of the water and gelatine.

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