

May 28, 1974

KARL-WILHELM SCHRANZ ET AL 3,813,246
PROCESS FOR THE REOXIDATION OF PHOTOGRAPHIC
BLEACHING AND BLEACH-FIX BATHS

Filed March 17, 1972

4 Sheets-Sheet 1

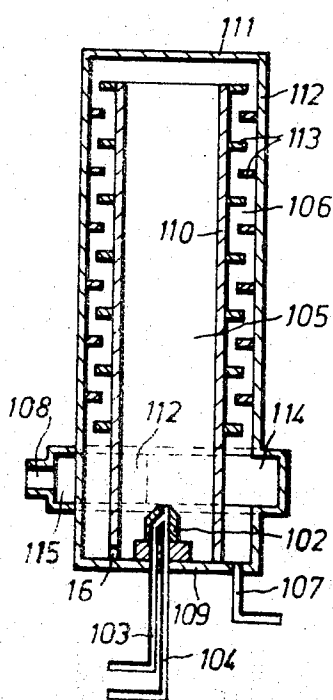


FIG. 1

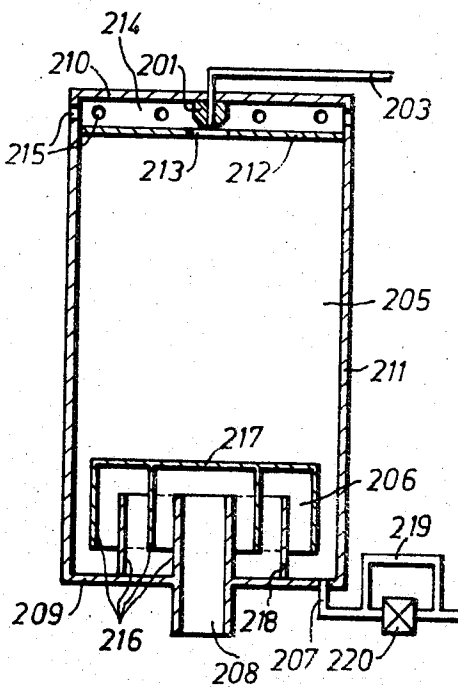


FIG. 2

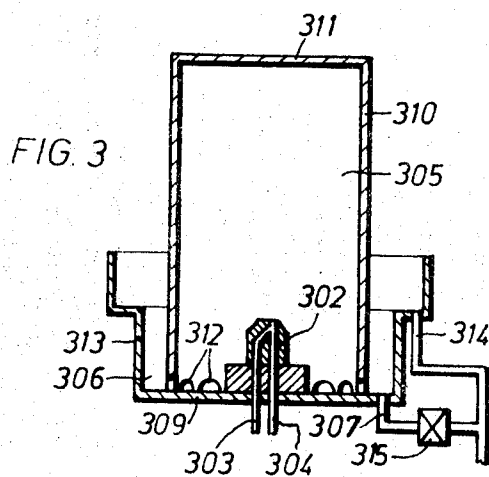


FIG. 3

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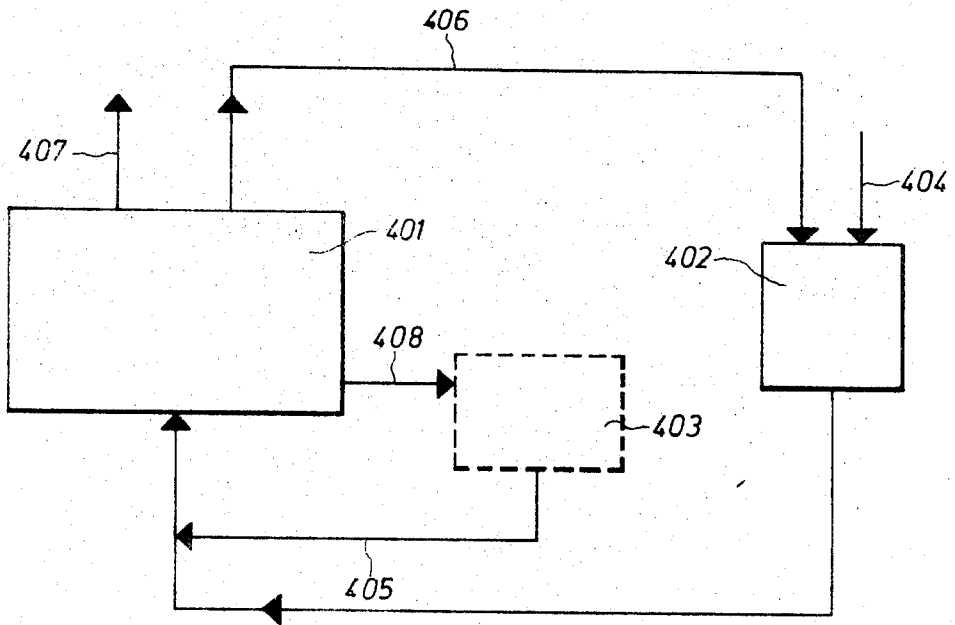


Fig. 4

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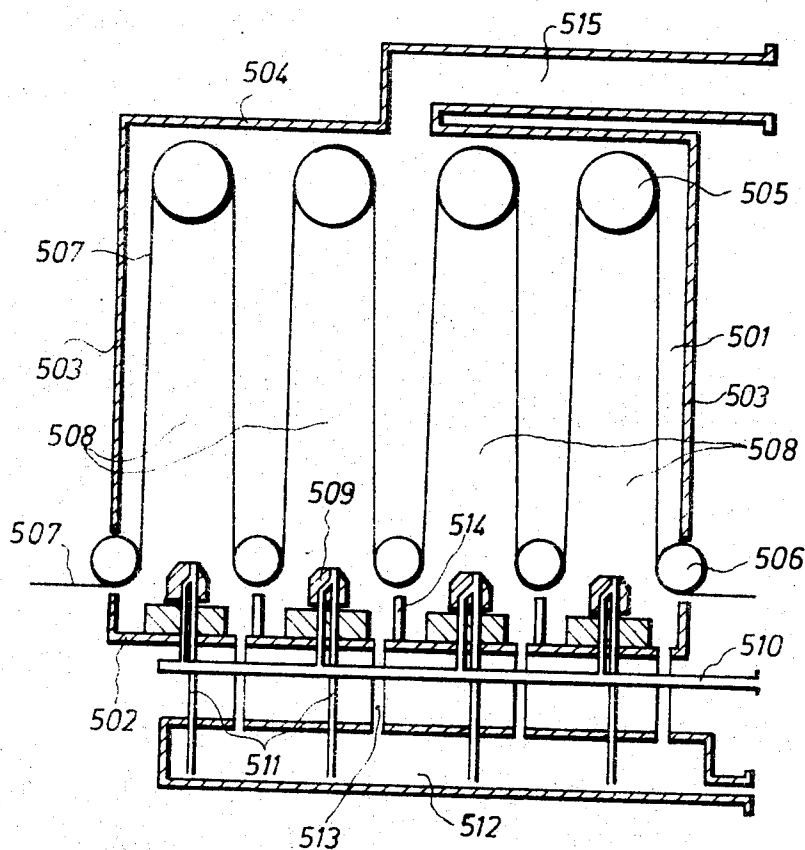


Fig. 5

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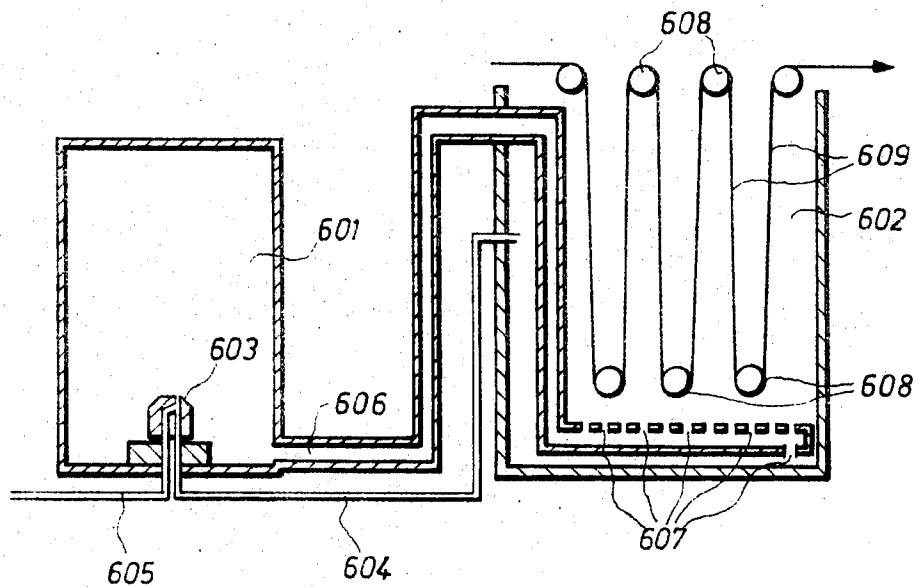


Fig. 6

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PROCESS FOR THE REOXIDATION OF PHOTOGRAPHIC BLEACHING AND BLEACH-FIX BATHS

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10 Claims

ABSTRACT OF THE DISCLOSURE

Photographic bleaching and bleach-fixing baths are reoxidized by spraying them through a nozzle in the presence of oxygen into an oxidation chamber. The oxygen is provided either in the form of air or pure oxygen, which may be intermixed at the nozzle or contacted in the chamber atmosphere. The gases and liquid constituents of the sprayed baths are separated by directing them through baffled passageways or bubbling through a liquid. The baffled passageways may be disposed concentrically about the oxidation chamber or in its bottom. The nozzles for liquid and gas atomization may be directed in the same or opposite directions or in separate or combined nozzles. A liquid for bubbling separation may be concentrically disposed in a jacket about the bottom of the oxidation chamber into which the liquid and gaseous constituents are forced by excess pressure. The liquid is discharged from an overflow near the top of the jacket and the gaseous constituents are separated by bubbling through the jacket. Reoxidation may be performed directly within the bleach or bleach-fixing chamber and may be performed at the same time as the baths are sprayed upon the silver halide emulsion layers of an exposed photographic material. Silver salts are removed from the baths before they are sprayed, and the photographic material may form part of the wall of the chamber.

CROSS-REFERENCE TO RELATED APPLICATION

None.

BACKGROUND OF THE INVENTION

The invention relates to a process for the reoxidation of photographic bleaching and bleach fix baths, in which process the said baths are sprayed in the presence of oxygen.

It is known to spray photographic baths on to photographic materials which are to be processed by means of spray nozzle systems. The main advantage of this process is that the bath is applied uniformly and only to that side of the material which carries the photographic layer with vigorous agitation so that the time required for the individual processing steps is very short. It is also known that when a photographic developer bath is applied by spraying in the presence of oxygen the bath is very liable to undergo oxidative changes which may impair the results of the process. It is therefore generally necessary to add antioxidants to a bath which is to be sprayed in order to counteract this effect or to apply the spray with the substantial exclusion of oxygen.

It has not previously been known that the oxidative change which inevitably occurs when spraying photographic baths can be utilized for the photographic process.

When processing color photographic material based on silver halide, the photographic material must be treated

either with a bleaching bath and a fixing bath or with a bleach fix bath after the color development in order to remove the image silver and any silver halide still present after development. This treatment converts the silver image into a silver salt by the oxidizing properties of the bleaching agent, and the silver salt is converted into a readily watersoluble form by the fixing agent. In order that the bleaching agent may be used again for the photographic process it must be reoxidized. When bleach-fix baths are used, reoxidation is preceded by electrolytic or chemical desilvering of the bath. Reoxidation of the bleach-fix bath combined with desilvering is gaining a position of increasing importance due to increasingly stringent legal requirements to prevent pollution of effluent water and to the increasing cost of silver. The oxidation process may be initiated in known manner by chemical compounds such as bromine, potassium persulfate or potassium bromate or by blowing air into the bleaching bath or bleach-fix bath. Most chemical oxidizing agents, however, lead to an accumulation of salts in the baths which may cause the bleaching process to be considerably impaired.

The method of injecting air into the bleaching bath or bleach-fix bath, on the other hand, requires the use of large storage tanks because the rate of oxidation is low due to the comparatively small phase interface between the gaseous atmospheric oxygen and the bleaching bath and consequently a long time is required for reconstituting the bleaching bath or bleach-fix bath.

It is an object of this invention to provide a simple and economical process for the reoxidation of used photographic bleaching and bleach-fix baths whereby the baths are rapidly made available again for use without being impaired by the accumulation of foreign salts.

SUMMARY OF THE INVENTION

It has now been found that used bleaching and bleach-fix baths can be reoxidized by spraying in the presence of oxygen.

This invention therefore relates to a process for the reoxidation of photographic bleaching and bleach-fix baths which is characterized in that the photographic baths are sprayed in the presence of oxygen in an oxidation chamber and that the spray mist is subsequently separated into gaseous and liquid constituents in a separator. The invention also relates to various apparatus for carrying out this process.

The baths which are to be reoxidized are finely sprayed or atomized by means of spray nozzle systems in an oxidation chamber, the oxygen required for reoxidation being supplied in the form of pure oxygen or air. The apparatus according to the invention are provided with separators which enable the spray mist formed in the oxidation chamber to be separated into gaseous constituents (effluent air) and liquid constituents (bleaching or bleach-fix baths) and which ensure that the effluent air is not discharged until it is free from spray. The effluent, from which oxygen has been partly or completely removed, may then be either introduced into another reaction chamber, e.g. to serve as protective gas for photographic processes which are sensitive to oxidation, or reused for the reoxidation process in the spray chamber, but the oxygen used up in the process of reoxidation is advantageously replaced by the addition of fresh air or oxygen.

BRIEF DESCRIPTION OF THE DRAWING

Further details and advantages of the invention will be clear from the claims in conjunction with the description of practical examples which are explained in detail with reference to the figures of the drawing in which:

FIG. 1 shows an apparatus for the reoxidation of bleaching and bleach-fix baths by the process according to

the invention, in which the separator surrounds the oxidation chamber;

FIG. 2 shows another apparatus for the process according to the invention, in which the separator is situated immediately below the oxidation chamber; and

FIG. 3 shows another apparatus for the process according to the invention, in which ventilation of the oxidation chamber is effected by way of a bubbling vessel.

FIG. 4 shows schematically the recirculating air process in which the air leaving the oxidation chamber is partly used again for spraying the bath, and the oxygen used up in the oxidation reaction is supplied by admixture of oxygen or fresh air.

FIG. 5 shows an apparatus for carrying out simultaneously the reoxidation of bleaching or bleach-fix baths and the bleaching or bleach-fixing, both by spraying the bath in the presence of oxygen in the same chamber.

FIG. 6 shows an apparatus according to the invention consisting of an oxidation chamber and a bubbling vessel which functions as a separator and constitutes the processing tank for the bleaching or bleach-fixing process.

DESCRIPTION OF THE PREFERRED EMBODIMENTS

FIG. 1 shows a longitudinal section through an apparatus for the reoxidation of bleaching and bleach-fix baths. The oxidation chamber 105 is formed by an upright cylindrical tube 110 (wall of oxidation chamber) which is open at the top and rigidly connected to a horizontal base plate 109 at the bottom through which the two component nozzle 102 extends axially and opens into the oxidation chamber. The oxidation chamber 105 is surrounded coaxially by a wider tube 112 (wall of housing) which is closed at the top by the cover plate 111 and is also rigidly connected with the baseplate 109 and which together with the cover plate 111 forms the housing of the separator 106. The atomized spray composed of bleaching or bleach-fix bath supplied through feed tube 103 and air supplied through feed tube 104 is discharged at the top of the two-component nozzle 102 to enter the oxidation chamber 105 where reoxidation of the bleaching agent substantially takes place. Most of the atomized bleaching or bleach fix bath is deposited on the wall of the oxidation chamber 110 and on the cover plate 111 of the separator housing. The remainder of liquid is removed from the air by entering the separator 106 which forms a jacket surrounding the oxidation chamber 105, where it is deposited on the annular separator plates 113 which are attached alternately to the external surface of the wall of the oxidation chamber 110 and the internal surface of the wall of the housing 112. The air freed from spray then enters the annular extension 114 at the level of the outlet connection 108 on the side diametrically opposite to the said outlet connection 108. This annular extension 114 surrounds the oxidation chamber 105 and on the side of the outlet connection 108 it is continuous with the semi-circular ring channel 115 which lies on the outside of the wall of the housing 112. On that side, the wall of the housing 112 does not have an annular extension in its width but is partially continuous to form baffle 112 so that no bath liquid dripping from the separator plates 113 can be blown into the outlet connection 108. The bath liquid deposited as described above is removed from the apparatus via the discharge pipe 107. Bath liquid which has been deposited inside the oxidation chamber enters the discharge pipe 107 through the aperture 116.

Another embodiment of an apparatus for carrying out the process according to the invention is illustrated in FIG. 2. The oxidation chamber 205 consists of a cylindrical tube 211 (wall of oxidation chamber) provided with a baseplate 209 and cover plate 210. The bath liquid supplied through the inlet 203 is atomized into the oxidation chamber 205 vertically from above through the one-component nozzle 201. In the plane of the outlet aper-

ture of the spray nozzle 201 there is a partition 212 which has an aperture 213 in the region of the spray nozzle 201. This aperture provides for automatic access of air from the chamber 214 formed between cover plate 210 and partition 212 into the oxidation chamber 205 when bath liquid is atomized into the oxidation chamber. The higher the rate of discharge of the bath from the spray nozzle 201, the more vigorously is the spray mixed with air. Chamber 214 communicates with the outside air through a plurality of apertures 215.

When the bath liquid has been sprayed into the oxidation chamber, the spray is separated into gaseous and liquid constituents by means of the separator 206 which in this case is arranged in the lower part of the apparatus. It is composed of several pipe elements 216 of different diameters which are boxed inside one another and which are attached alternately to the baseplate 209 and the circular plate 217. The air outlet connection 208 is arranged axially at the centre of the baseplate. The air is sufficiently freed from moisture by repeated deflection so that it is free from spray when it leaves the outlet connection 208.

The deposited bath liquid collects on the baseplate 209 of the oxidation chamber 205 and can be discharged through a discharge pipe 207. The inner zones of the separator 206 communicate with the discharge pipe 207 through apertures 218. A knee bend trap 219 is connected to the discharge pipe 207 to enable a sump to be formed and thereby prevent air from escaping through the discharge pipe 207 or through the apertures 218. The liquid collected at the bottom of the oxidation chamber 205 can be completely discharged by opening the shut-off valve 220 when required, for example when work is finished. To prevent any siphon effects in the knee bend trap 219, the upper horizontal portion of pipe and the adjacent vertical portion are made wider than the rest.

FIG. 3 shows another apparatus for carrying out the process according to the invention. The oxidation chamber 305 consists in this case of the cylindrical pipe 310 which is rigidly connected to the baseplate 309 and closed at the top by the cover plate 311 so that there is no outlet at the top. As in the case of FIG. 1, the atomized spray from the two-component nozzle 302 is formed from the bath liquid supplied through the feed tube 303 and air supplied through the feed tube 304. The spray is reflected from the cover plate 311 of the oxidation chamber 305 and partly deposited as liquid. Subsequently, the ascending spray from nozzle 302 and the return flow of air together produce a vigorous turbulence which is very effective for the reoxidation process of the bleaching agent. Numerous apertures 312 are provided at the lower end of the wall of the oxidation chamber to enable air to escape. The lower part of the oxidation chamber 305 is surrounded by an annular jacket 313 in the form of a vessel which is filled with bath liquid to the top of the overflow pipe 314 when the apparatus is in operation. Inside the oxidation chamber 305, the excess pressure produced maintains the liquid level at the height of the apertures 312. The spray mist formed in the oxidation chamber 305 then escapes to the outside through the apertures 312 and on bubbling through the liquid which acts as separator 306 in the annular jacket vessel 313 it is separated into its constituents so that only air free from spray can escape to the outside. The jacket 313 is widened at the top to prevent spilling over the top by spraying or foaming. The discharge pipe 307 with shut-off valve 315 serves to empty the apparatus when operation is finished. When the apparatus is put into operation after it has been emptied, the process must be begun with a small supply of air until sufficient liquid has collected in the jacket 313 to separate the discharged spray.

FIG. 4 shows a flow diagram of the recirculation air process. In the spraying device 401 including separator the spray mist is separated into liquid 408 and gaseous

constituents 407 and 406. Part of the effluent air 406 is to be reused and the remaining part 407 is discharged out of the circuit. Oxygen or fresh air 404 is admixed to the recirculated air 406 which is partly depleted of oxygen. The mixture is pumped by the compressor 402 again into the spraying device, thereby carrying the bleaching or bleach-fixing bath to be reoxidized 405. The latter is supplied from the collecting vessel 403 wherein the liquid 408 is collected. The bleaching or bleach-fixing process may be carried out within the spraying device 401 by spraying or alternatively by immersion in the bleach or bleach-fix bath collected in the collecting vessel.

FIG. 5 shows a longitudinal section through an apparatus in which the bleaching or bleach-fixing and the spraying are carried out in the same chamber. The chamber 501 is formed by the rectangular base plate 502, the side walls 503 and the cover plate 504. Within the chamber 501 are situated several upper deflecting rollers 505 and lower deflecting rollers 506. A photographic web 507 with a silver halide emulsion layer to be bleached which is moved through the chamber at a constant speed is deflected several times by the deflecting rollers thus forming several segment chambers 508 within the chamber 501. Several two component nozzles 509 extend through the base plate and open into each of the segment chambers 508. Oxygen or air is supplied by feed tube 510 and the bath to be reoxidized and applied to the silver halide emulsion layer of the photographic web 507 is supplied by feed tubes 511. The latter feed tubes are dipping to the bottom of a collecting vessel 512 which collects via the discharge pipes 513 the bath liquid deposited from the spray mist within the chamber 501. Underneath the lower deflecting rollers 506 the various segment chambers 508 are separated from each other by partial dividing walls 514. The moist spraying gas from which most of the liquid has been separated within the chamber 501 is removed through the outlet connection 515 and can be conducted through a separator for removing the remainder of liquid and/or can be refed wholly or partially into the chamber 501. If the front and rear walls of the apparatus (not seen in FIG. 5) have a distance that equals the width of the photographic web, then each of the segment chambers 508 can be considered as a separate spraying and application chamber which is limited by the upper deflecting roller 505, the photographic web 507 the front and rear wall (not seen), two partial dividing walls 514 and part of the base plate 502. In this case nearly no intermixture occurs between different segment chambers 508 and it would be possible to have in different segment chambers photographic processing baths of different composition.

FIG. 6 shows a longitudinal section through an apparatus according to the invention which consists of an oxidation chamber 601 and a bubbling vessel 602 which is simultaneously used as processing tank. A two component nozzle 603 extends through the bottom of the oxidation chamber 601 and atomizes bleaching or bleach-fixing bath supplied by the processing tank (bubbling vessel) 602 through feed pipe 604. The gas for spraying (oxygen or air) is supplied by feed pipe 605. The spraying mist is partly separated into gaseous and liquid constituents and leaves the oxidation chamber 601 through the outlet pipe 606 which reaches to the bottom of the processing tank 602 where it has numerous apertures 607 to allow the gaseous constituents to bubble through the bath liquid in the processing tank. Several upper and lower deflecting rollers 608 provide means for moving a photographic web 609 to be processed through the bleaching or bleach-fixing bath in the processing tank.

The process according to the invention will now be further explained by the example which follows.

EXAMPLE

An exposed color photographic multilayered material which had previously been treated with a developer was

processed in a bleach-fix bath of the following composition:

	G.
Ethylenediaminetetracetic acid -----	3
5 Na ₂ HPO ₄ -----	20
Na ₃ PO ₄ -----	4
Iron-III-complex of ethylenediaminetetracetic acid, sodium salt -----	30
Na ₂ SO ₃ -----	8
10 Ammoniumthiosulfate -----	120
Na ₂ CO ₃ -----	6
Water up to 1000 ml.	

pH with acetic acid adjusted to 7.7.

During this treatment, the change in Redox potential of the bleach fix bath was continuously determined by means of a platinum electrode against a silver-silver chloride electrode used as reference electrode. The Redox potential of the bleach fix bath was continuously determined by means of a platinum electrode. The Redox potential was -25 mv. at the beginning of the experiment and fell to -112 mv. in the course of processing the photographic material, a reduction in bleaching activity being observed at the same time. The bleach-fix bath was then subjected to electrolysis as generally carried out for desilvering photographic fixing baths. There was then practically no further change in Redox potential.

1st experiment

Part of the desilvered bleach fix bath was sprayed through nozzle 102 in an apparatus according to FIG. 1 simultaneous with a supply of air. The residence time of the liquid in the apparatus was of the order of several seconds. The bleach-fix bath collected from the discharge tube had a Redox potential of -72 mv. after the first passing through the spray process. After a further passing through the spray process, the Redox potential rose to -44 mv. and after a third spraying it was found to be -14 mv. When the bleaching capacity of the reconstituted bath was compared with that of a fresh bleach-fix bath the values were obtained substantially similar.

2d experiment

The other part of the desilvered bleach fixed bath was introduced into a large tank which had a plastics tube provided with small apertures (diameter about 1 mm.) fitted into the base. Air was then injected into the bleach fixed bath through the plastics tube for 120 minutes. The effect on the redox potential was only slight (increase to -106 mv.) so that a method of injecting air by which comparatively large air bubbles are formed must be regarded as practically useless.

3rd experiment

The second experiment was modified in that the plastics tube was replaced by a glass frit G 4 with very small pores and air was again injected. Very fine bubbles of air were observed which permeated the bleach-fix bath almost homogeneously. At the same time, a bulky foam settled on the surface of the bleach fix bath. After 60 minutes of intensive admixture of air the redox potential, which rose in almost linear progression, was -15 mv. When the bleaching capacity of the bath was tested it was found to be comparable to that of a fresh bleach fix bath.

Experiments 1 and 3 show that spraying three times and injecting air for 60 minutes through a glass frit produce practically the same results but the undesirable formation of foam in the latter case renders reoxidation of the bleach fix bath in the processing tank impossible so that additional large storage tanks are required.

When comparing the residence times of the bleach fix bath in the reoxidation chamber (1st experiment) and in the tank (3rd experiment) the results of the spray process are found to be very favorable. Due to the short processing time required for the reconstituted bleach fix bath, the

quantity of bath required in the cycle of processing-desilvering-reoxidation is small so that it becomes possible to use only small apparatus for a cycle including the working up of the bleach fix bath. Moreover, when changing the bath, the costs arising from losses in the bleaching or bleach-fix bath are insignificant owing to the small volume involved.

In the apparatus described in the examples, liquid pumps or compressors commonly known in the art are used for supplying the bleaching or bleach fix bath and air. The choice of devices used depends on the required rate of flow at the given delivery pressure. The choice of material is particularly important in view of the corrosive nature of the baths. For the oxidation chamber and the tubes synthetic materials can be used such as polyvinylchloride, polymethacrylate and polypropylene. The materials used for the liquid pumps must also be highly corrosion-fast. Glass, porcelain or special metal alloys can be used for this purpose, preferably stainless steel such as 18 CrNi 8 (V 2 A, V 4 A) or Hastelloy C, a trademark of Haynes Stellite Co. for a high-temp. nickel-base alloy containing 0.15% (max.) carbon, 13.0–16.0% chromium, 15.0–19.0% molybdenum, 3.5–5.5% tungsten, 4.0–7.0% iron, and the balance nickel. In the case of two-component nozzles (FIG. 1 and FIG. 3) the bath liquid may be delivered to the spray nozzle at a lower pressure because the energy required for atomization is derived almost completely from the compressed air. The supply of air into the oxidation chamber may be partly or completely separated geographically from the place where atomization or the bath liquid takes place. Thus, for example a one-component nozzle for the bleaching or bleach fix bath and a nozzle for supplying air may be arranged opposite each other so that vigorous turbulence is produced by the opposite directions of the jets of spray and air, by which the reoxidation process of bleaching agent is greatly assisted. Alternatively, a one-component nozzle or two-component nozzle may be surrounded by a ring of air nozzles, by which a high rate of air flow in the same direction as the delivery of atomized bath liquid is provided which is particularly suitable for dealing with large outputs of bath liquid in large apparatus.

This technique is highly advantageous because the air can be passed through the oxidation chamber at a high velocity, because the energy necessary for warming up the air to the temperature of the spraying process can be kept low, and because the oxygen content can be controlled exactly and easily. During the process oxygen is used up and has to be replaced. If pure oxygen is supplied in the recirculating air process, it is even possible to have the circuit closed, so that no outgoing air is taken off. In this case in FIG. 4 the outlet connection is closed. The oxygen which may be fed in from an oxygen cylinder is used completely and on the other hand the mist formed by spraying has not to be separated thoroughly into gaseous and liquid phases. Since the technique of recirculating air process is quite generally known (e.g. for drying processes; reference is made for example to A. G. Kassatkin, "Chemische Verfahrenstechnik," volume II, VEB Deutscher Verlag für Grundstoffindustrie, 5th edition, Leipzig 1962) it requires no further explanation here. Since oxygen is used up in the reoxidation of bleaching agent, the air after it has passed through a separator may be fed into a reaction chamber for photographic processing stages which are sensitive to oxidation, e.g. development by a spray process, hobber application process or roller application process. It is particularly when an air recirculation process with admixture of fresh air is employed that the air partly depleted of oxygen may assume the function of a protective gas, e.g. if the recirculated air is introduced into the appropriate reaction chamber before it is mixed with fresh air.

The process is advantageously carried out at elevated temperatures (up to 50° C.) in order to accelerate the

reoxidation process. In that case, if the oxidation chamber is connected to a processing machine, the temperature may be regulated by the temperature control unit of the bleaching or bleach fix bath. It is especially advantageous if the oxidation chamber in which reoxidation of the bleaching agent takes place serves at the same time as the processing chamber for bleaching or bleach fixing because the integration of reconstitution of the bleaching agent with processing enables small machines to be used.

In one special embodiment, the wall of the oxidation chamber is partly formed by the silver halide emulsion layers of a photographic material which is moved at constant speed. The oxidation chamber may, for example, have a rectangular cross-section, two fixed side walls facing each other being formed by the apparatus while a band of photographic material passing through the apparatus is deflected through 180° by a deflecting roller so that it forms the other two side walls of the oxidation chamber.

Lastly, the air which has passed through the oxidation chamber may be conducted through a bubbling vessel which functions as a separator and which at the same time constitutes the processing tank or part of the processing tank for the bleaching or bleach fixing process. This arrangement not only ensures efficient separation of the atomized spray but also provides for thorough agitation of the processing bath so that the time required for bleaching may be expected to be reduced.

We claim:

1. A process for the continuous reoxidation of used photographic bleaching and bleach fix baths, characterized in that the used bleach fix bath is atomized and injected into contact with gaseous oxygen whereby the used bleach fix bath is reoxidized essentially to its original chemical state, and the atomized spray containing the oxidized bleach fix bath is caused to directly impinge upon a separation medium for separating out and recovering the liquid constituents.

2. A process according to claim 1, characterized in that the bleach fix baths are atomized in the presence of air.

3. A process according to claim 1 wherein the gaseous oxygen in contact with which the bleach fix baths are atomized is provided by recycled air and wherein the oxygen depleted in the reoxidation is replaced by the addition of fresh air or oxygen.

4. A process according to claim 1 wherein the bleach fix baths are obtained from photographic bleaching or bleach fixing processes which are performed at a range of processing temperatures, and wherein the reoxidation is performed at about the same processing temperature range.

5. A process according to claim 4 wherein the processing temperature range is up to about 50° C.

6. A process according to claim 1 wherein the bleach fix baths are obtained from photographic bleaching or bleach-fixing processes which are performed in a chamber, and the reoxidation is performed in the same chamber.

7. A process as set forth in claim 6 wherein the bleaching or bleach fixing is performed by atomizing the bleach fix baths and the reoxidation is performed by the atomization which occurs in the bleaching or bleach fixing.

8. A process according to claim 1, characterized in that the gaseous components which are depleted in oxygen and separated from liquid constituents are used as protective gas for other photographic steps which are sensitive to oxygen.

9. A process according to claim 1 wherein the reoxidation is performed in an oxidation chamber, the gaseous constituents being removed from the chamber, and the gaseous constituents being separated from the liquid constituents by bubbling them through a liquid.

10. A process according to claim 8 wherein the liquid through which the gaseous constituents are bubbled is utilized in the bleaching or bleach fixing process.

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5 M. F. KELLEY, Assistant Examiner

U.S. Cl. X.R.

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