

- [54] **BACKWETTING COATING FOR DIAZO MICROFILM**
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- [58] **Field of Search**..... **96/75, 87 R, 87 A, 78, 96/91 R**

[56]

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[57]

**ABSTRACT**

A transfer resistant diazo photographic element is provided by employing an improved backwet composition, which when coated on the back of light sensitive diazo elements will inhibit offset or transfer of the sensitized coating from the front to a juxtaposed or contracting back of the element. Such transfer inhibition is vital when the element is stored under conditions causing contact of the front and back sides of the film, e.g., in tightly rolled condition. This transfer inhibition is accomplished by employing either selected solid, particulate, components in a standard back-wetting solution to form a back-wet slurry or selected cured thermosetting resins soluble in standard back-wetting solutions. The particulate components are small particle size inorganic compounds, for example, silica. An example of the thermo-setting resins that may be employed is ureaformaldehyde resin. The composition thus provided, has at least the dual functionality of preventing offset, and of controlling curl in the film or element.

**3 Claims, No Drawings**

## BACKWETTING COATING FOR DIAZO MICROFILM

This invention relates to improved diazo photographic elements. An additive is employed with a back-wetting solution to inhibit any offset between the front, sensitized portion of the element and the back of the element. This inhibition enables the element to be stored in tightly wound rolls, or in other ways where contact is permitted between the sensitized and unsensitized sides of the element or film, without any offset, thus eliminating a primary cause for the loss of film and information contained thereon.

In some cases film may be interleaved with paper layers or the like which separate the backing from the sensitizing layer. In many instances, however, certain film and particularly microfilm is tightly wound into rolls for insertion into magazines or the like, so that the sensitive layer is of necessity, in direct and intimate contact with the film backing.

Backing layers or back-wetting layers are known to the photographic art. Generally, these layers have been used to provide a means of insuring that the coated photographic element or film will not curl. Many additives have been incorporated into back-wetting gelatin emulsions, for application on silver halide sensitized films. These additives have been used for specific purposes, for example, antihalation, antistatic, lubrication and transfer inhibition. Some of these coatings have been discussed in the Patent Literature, see for example, U.S. Pat. Nos. 3,617,286; 3,262,782; 3,206,311; 3,163,535; 3,053,662; 2,993,793; 2,322,037; 2,095,018; 1,631,421 and 1,597,727. While these coatings may be useful with gelatin emulsions in silver halide sensitized systems, they are not completely satisfactory when applied to the diazo photographic elements of this invention.

Silver halide emulsions, of necessity, have a grain which can be large or small, depending on emulsion formation parameters. Diazo coatings, on the other hand, do not have a grain. This absence of any grain structure is of paramount importance in resolution especially with microfilm, because of the requisite large image magnification to facilitate reading. Accordingly, the indiscriminate use of comminuted solids, disposed on diazo films would tend to introduce an intolerable graininess which would completely negate the inherent resolution advantage of a diazo microfilm system. Further, due to the complexity of a diazo system in regard to balancing reactivity and stability, care must be exercised in selecting components that come in contact with the sensitized system. For practical purposes, the problems encountered with diazo sensitized elements are completely different from those encountered with silver halide sensitized elements. Diazo microfilm, for example, containing a number of extremely sensitive and reactive components, is sensitive to any changes in chemical composition. Transfer of the entire coating or of any of the components from the sensitized portion to the contacting back of the film will result in either loss of that portion of the film or destruction of information recorded on the film. Since microfilm of that type is often used to record vital documents for storage, a means to prevent this transfer from the sensitized portion to the back of the film is necessary.

It is therefore an object of this invention to provide a transfer inhibiting back-wetting composition and a

diazo sensitized photographic element coated with same which will not be subject to one or more of the above disadvantages.

Another object of this invention is to provide a back-wetting coat incorporating selected additives to inhibit the transfer or offset of the sensitive portion of diazo film to the contacting or juxtaposed back portion.

A further object is to provide a method of insuring against the obscuring of information and loss of film due to the transfer of sensitive components to the back of the film in tightly wound rolls or multi-layered stacks.

Yet another object is to provide a more efficient diazo microfilm by eliminating waste due to the offset of sensitive materials from the sensitive portion to the back portion of the film.

Other objects and many of the attendant advantages of this invention will be readily appreciated as the same become better understood by reference to the following description:

The subject invention, involving the use of a backwet coating comprising a mixture of a transfer preventing additive selected from the group consisting of particulate solid inorganic pigments having a particle size of about 0.005 to 0.020 microns and cured thermosetting resins, with a solution of a polymeric binder resin in a liquid organic solvent medium, on diazo photographic elements overcomes one or more of the advantages of the prior art heretofore described. This is accomplished with the advantage that such element is thus rendered capable of being stored in tightly wound or other juxtaposed condition for an extended period of time, and that the efficiency of such film is greatly increased as by reduction of the amount of unusable film due to such transfer.

In the practice of this invention, by the incorporation of particulate solids such as inorganic pigments or selected soluble thermo-setting resins into a back-wetting solution, applied to diazo photographic elements, transfer or offset is effectively inhibited. The incorporation of the solids into a standard back-wetting solution forms a back-wetting slurry. The slurry, when coated on the film and dried, forms a substantially continuous coating phase having dispersed in it a substantially discontinuous phase of solid particles. The thermosetting resins on the other hand form a homogeneous solution with no substantial discontinuous phase, yet are found to be equally as effective in transfer inhibition.

The back-wetting layer, to prevent offset, must conform to certain practical parameters. It must be strongly bonded to the base film, it must have an index of refraction close to or identical with the base film, it must be compatible with the base film, it must minimize curl of the base film, and it must effectively prevent transfer. With different types of base film, different types of binder for the back-wetting coating may be used.

The base films that may be used with my invention are any common transparent film compositions known to the art including cellulose acetate butyrate and cellulose acetate esters such as cellulose triacetate; mixed cellulose esters such as cellulose acetate butyrate and cellulose acetate propionate and synthetic resins such as polyvinyl chloride acetate, polystyrene, polyethylene terephthalate and bisphenol-A polycarbonate. The binder portion of the back-wetting solution is advanta-

geously the same as the base film to minimize curling tendency and to increase compatability, but any suitable resin binder may be used so long as it falls within the above described parameters. Organic colloids, such as gelatin, due to the excessive thickness of the film coating required, and due to the highly hydrophilic properties of a colloid such as gelatin, are not suitable.

The particulate solids of this invention are subject to a limitation on their size. They must be large enough to effectively prevent transfer of the sensitive agent, and small enough so that they will not produce graininess in the film, and destroy the excellent resolution obtainable with diazo films, especially microfilm. These particles may vary from about 0.005 to 0.020 microns in size to impart the advantages of the invention, preferably from about 0.007 to 0.012 microns in size.

The inorganic pigments that are suitable are silica and other common inorganic pigments and fillers such as finely divided alumina. Silica exists in a variety of polymorphic crystalline forms as well as in amorphous modifications, which latter are the preferred additives of this invention, as they are generally of smaller particle sizes. Amorphous silica is substantially dehydrated, polymerized silica which may be considered as a condensation polymer of silicic acid. The amorphous silicas may be further typified as silica gel, colloidal silica, precipitated silica, or fumed silica. Silica gel may be prepared by either the bulk set process, or the slurry process; colloidal silica may be prepared by a peptization of silica hydrogel or by a gradual growth of silica micelles; precipitated silica is prepared by precipitation; and fumed silica may be prepared by a vaporization and recondensation process or a process wherein crystalline silica is fed directly to a very high temperature jet or by a flame hydrolysis process. For example, Cab O Sil M-5 (a registered trademark for a silica product marketed by The Cabot Corporation) may be used advantageously. The only limitation on the silica to be used is the size limitation specified above.

The resins that are suitable are cured, thermo-setting resins, for example, amino resins, such as urea-formaldehyde or melamine; polyesters, such as allylic resins, for example, diallyl phthalate and diallyl isophthalate; cured epoxy resins; and cured phenolic resins such as C stage or resite, phenol-formaldehyde resins. Selected thermoplastic resins having a heat seal temperature of at least 175°F and a softening point of at least 130°C such as hydroxyl modified vinyl chloride acetate copolymers are also suitable. The molecular weight of these polymers must be high enough to insure that they are effective against transfer. Further, they must be soluble in the back-wet solution, and non-reactive with both the sensitizing composition, and with any solvents used in the entire coating process.

Some of the thermo-setting amino resins, which are applicable, are condensation products of formaldehyde and such compounds as urea, melamine, dicyandiamide and ethylene urea. With regard to the amino resins, the urea-formaldehyde and melamine-formaldehyde resins are preferred with my invention. Preparation of these resins is conventional and synthetic preparation techniques are well known. The ratio of formaldehyde to urea may vary from about 1.3 to 1.8 parts by weight formaldehyde to 1 part urea. Higher amounts of formaldehyde may be used, but then the resins are difficult to produce. The melamine-formaldehyde resins of my invention usually have a 1:2

to 1:3 weight ratio of melamine to formaldehyde. These compounds can be cured by well known methods such as elevated temperatures or direct or latent catalysis, for example, in the case of urea-formaldehyde, acid catalysis is used to advantage. Unalkylated, melamine-formaldehyde resins, on the other hand, can be cured by elevated temperature alone under neutral or slightly alkaline conditions.

The cured epoxy resins which are useful with this invention are highly crosslinked reaction products of bisphenol A and epichlorohydrin cured with a polyfunctional amine or anhydride curing agent. The epoxy resins generally have an average molecular weight above 350 and have an epoxide equivalent of greater than 175. The curing agents may be any of the polyfunctional amines or anhydride curing agents or crosslinking agents of the art such as diethylene, triamine, trimethylamine, ethylene diamine, benzyldimethylamine triethylene tetramine, piperidine, metaphenylene diamine, phthalic anhydride and dodeceny succinic anhydride, as well as commercially modified or blended amine and anhydride curing agents, and in some cases aluminum chloride.

The phenolic resins which may be used are alkaline catalyzed condensation products of phenolic compounds; such as phenol, ortho and meta cresols, resorcinol, xylenol, naphthol and hydroquinone with an aldehyde or an aldehyde producing compound; such as formaldehyde, furfural, crotonaldehyde, formaline, acrolein, paraformaldehyde and acetaldehyde. These partially cured condensation products are of high molecular weight, and have a high degree of crosslinking. They are prepared by well known synthetic techniques. Generally, the ratio of phenolic compound to aldehyde or aldehyde producing compound in the starting materials, is 1 part by weight phenolic to 1.1-3 parts by weight aldehyde or aldehyde producing compound.

The thermosetting polyester resins which are useful with the invention are unsaturated resins such as alkyl resins, for example, diallyl phthalate. Polymerization by well known methods converts the monomer into a thermo-setting, highly crosslinked polymer, which is useful with the invention. Known initiators and curing agents may be used to accomplish this polymerization.

In some cases, the cured resin is obtained as a finely divided powder, in others, a mass of the cured resin is obtained.

Solvents that may be used to form the back-wetting composition of the invention are any organic liquids having a boiling point below about 140°C that will dissolve the binder and the thermo-setting resin, when used, but remain unreactive toward the particulate additive. Additionally, the solvent must be capable of softening the base film so that good bonding between the back-wetting layer and the base film is obtained. Examples of such solvents include alcohols such as methanol, ethanol and isopropanol; ketones such as acetone, methyl ethyl ketone and methyl isobutyl ketone; esters such as ethyl acetate and amyl acetate and ethers such as ethylene glycol monomethyl ether and mixtures of these solvents.

The thickness of the back-wetting layer is generally governed by the thickness of the photosensitive layer. Both layers are usually of the same approximate thickness to minimize curl and the like, but the primary criterion is that the back-wet layer must be thick enough

when dried to prevent transfer of the photosensitive material.

The amount of resin to be incorporated as a binder is controlled by both the thickness of the film desired and rheological coating parameters. The amount generally may range from about 30 to 95% by weight of the total solid concentration of the back-wet slurry.

The amount of transfer inhibitor generally present is from about 5% to about 70% by weight of the total solid content of the back-wet slurry.

The back-wetting slurry is usually dilute and has from about 0.5% to 10% total solids, including both binder and transfer inhibiting additive, by weight. This, of course, again depends upon the rheological properties of the slurry, upon the thickness of the film desired, and the drying time required.

The sensitizing components that may be used with the film of this invention are standard diazo sensitizing components. These sensitizing formulations may for example make use of such light sensitive diazonium compounds as are disclosed in U.S. Pat. No. 2,501,874 and in the article by Van Der Grinten in the *Photographic Journal*, vol. 92B, 1952, page 46. Stabilized diazos derived from N-substituted-p-phenylenediamines are satisfactory and, in this connection, reference may be made to those diazos obtained from N,N-diethyl-p-phenylenediamine; N-benzyl-N-ethyl-p-phenylenediamine; N-ethyl-p-phenylenediamine; N-beta-hydroxy-ethyl-N-methyl-p-phenylenediamine and the like. Additionally, stabilized 4-N-pyrrolidinyl benzene diazonium compounds with additional substituents in the 3-position on the benzene nucleus are advantageous, such as 3-methyl-4-pyrrolidinylbenzenediazonium hexafluoroarsenate. According to the usual practice, these diazos are used in the form of salts stabilized with zinc chloride, cadmium chloride and the like.

Any of the usual coupling components are also satisfactory for my purpose. Examples of such couplers are 2,3-dihydroxynaphthalene; 1,8-dihydroxynaphthalene; resorcinol; octyl resorcinol; p-methyl-N-phenyl-pyrazolone; 2,3-dihydroxy-naphthalene-6-sulfonic acid; 5,5-dimethyl-1,3-cyclohexanedione; metahydroxyphenyl urea and the like.

The sensitizing solution may also contain the various adjuncts usual in the manufacture of light-sensitive diazotype materials.

These include metal salts for intensification of the dyestuff image, such as ammonium sulfate, nickel sulfate, zinc chloride and the like; stabilizing agents such as thiourea, thiosinamine, naphthalene trisulfonic acid and the like; acids acting to retard precoupling such as acetic acid, boric acid, tartaric acid and the like; hygroscopic agents such as glycol, glycerin and the like; and wetting agents such as saponin, lauryl sulfonate, keryl benzene sulfonate, the oleic acid amide of N-methyl taurine and the like.

The following examples are only illustrative. All parts and proportions referred to herein and in the appended claims are by weight, unless otherwise indicated.

#### EXAMPLE I

(Comparative)

A black line sensitizing composition of the following components:

sulfosalicylic acid	2.0	gm.
stannic chloride	0.25	gm.
m,m'-ethylenedioxydiphenol	2.0	gm.
dirosorcy sulfide	0.2	gm.
acetoacetanilide	0.3	gm.
beta-oxynaphtoic toluidide	1.5	gm.
3-methyl-4-pyrrolidinylbenzene diazonium chlorozincate	2.0	gm.

10 is dissolved in a solvent composed of 50 cc of acetone and 50 cc of methanol to form a sensitizing coating solution containing about 9.5% by weight of sensitizing composition. The sensitizing coating solution is then applied to one side of a self-supporting cellulose acetate film by a standard reverse roll, bead coating method. The sensitizing coating has a thickness of 0.001 mm. The base film has a thickness of 0.09 mm and an acetyl content of 62%. The backside of this film is then coated in a similar manner with a backwetting composition to control curl. The backwetting composition contains 0.3 g of cellulose acetate resin dissolved in a solvent composed of 50 cc acetone and 50 cc methanol to form a solution of about 0.5% resin content by weight. The sensitized and backwetted film is then air dried. The thickness of the backwetting or backing layer is about 0.001 mm.

Fifteen 4" x 4" sheets of the dry, sensitized and backwetted material are stacked in an oven at a pressure of 1 lb/in<sup>2</sup> above atmospheric and a temperature of 120°F for 3 hours. This test simulates, in a few hours, the conditions that a tightly wound roll of microfilm would experience over a greatly extended period of time at ambient conditions. After the film is thus stored, it is removed and the backside passed through an ammonia chamber to develop any sensitized component transferred thereto, the front or sensitized side is exposed to U.V. light to destroy the diazo component and permit better detection of transferred material. The amount of offset or transfer of sensitized material from the front side of the film to the contacting backside is then visually observed. It is found that approximately 10% of the film is unusable due to transfer of the sensitized component.

#### EXAMPLE II

A sensitizing coating is applied to a film base in exactly the same manner as in Example I. The sensitizing composition, film base, film thickness and coating thickness are also the same as in Example I. A backwetting composition is prepared by dissolving 0.3 grams of cellulose acetate resin in a solvent of 50 cc acetone and 50 cc methanol, then 0.5 parts by weight of Cab O Sil M-5 (a registered trademark for a silica pigment marketed by The Cabot Corporation), having a particle size averaging from 0.007 to 0.012 microns is added with agitation to the solution. The back-wetting composition is then applied in the same manner as in Example I. The sensitized back-wetted film is then dried, conditioned and tested in exactly the same manner as that in Example I. It is found that transfer of the sensitized component to the back of the film is substantially reduced.

#### EXAMPLE III

A sensitized coated film is prepared in exactly the same manner as in Example I. A back-wetting composition is prepared by dissolving 0.3 grams of cellulose ac-

etate resin in a solvent made up of 50 cc acetone and 50 cc methanol. To this solution is added 0.5 parts by volume of a urea-formaldehyde resin solution in the same solvent. The resulting back-wetting composition is then applied to the film in exactly the same manner as in Example I.

In addition to the sensitizing composition used in Ex-

The film base, sensitizing composition and backwetting composition are varied according to the description in Table I below. In Examples IV and VII, no transfer inhibitor is used, and the results of tests are similar to that of Example I. The remainder of the Examples use the transfer inhibitor of this invention and results are similar to those of Examples II and III.

TABLE I

EXAM- PLES	BASE FILM	SENSI- TIZER	BACKWETTING BINDER	COMPOSITION INHIBITOR	AVERAGE PARTICLE SIZE IN MICRONS	GMS.	SOLVENT
IV	Cellulose acetate Butyrate	A	Cellulose acetate Butyrate	3.0 gm. —	—	—	50 cc methyl ethyl ketone 50 cc isopropanol
V	Cellulose acetate Butyrate	C	Cellulose acetate Butyrate	2.0 gm. Silica Cab O Sil M-5	0.007 to 0.012	1.0	50 cc acetone 50 cc methanol
VI	Cellulose acetate Butyrate	B	Cellulose acetate Butyrate	2.0 gm. Phenol-form- aldehyde	—	3.0	50 cc methanol 50 cc methyl isobu- tyl ketone
VII	Cellulose acetate Propionate	A	Cellulose acetate Propionate	0.3 gm. —	—	—	50 cc ethyl acetate 50 cc methyl ethyl ketone
VIII	Cellulose acetate Propionate	B	Cellulose acetate Propionate	0.2 gm. Melamine-form- aldehyde	—	1.0	50 cc acetone 50 cc methanol
IX	Cellulose acetate Propionate	C	Cellulose acetate Propionate	0.2 gm. Silica Cab O Sil M-5	0.007 to 0.012	—	50 cc acetone 50 cc methanol
X	Polyethylene Terephthalate	A	Cellulose Triacetate	1.0 gm. Diallyl phth- alate	—	1.0	50 cc ethylene gly- col monomethyl ether 50 cc acetone
XI	Bisphenol A Polycarbonate	A	Bisphenol A Polycarbonate	0.5 gm. Silica Cab O Sil M-5	0.007 to 0.012	—	50 cc ethyl acetate 50 cc acetone
XII	Bisphenol A Polycarbonate	B	Bisphenol A Polycarbonate	1.5 gm. Urea Formal- dehyde	—	3.0	50 cc ethyl acetate 50 cc acetone

ample I, which for convenience will be designated as sensitizing composition A, two other sensitizing com-  
positions are set forth as follows:

## Sensitizing Composition B

Ethylene glycol	10.0	gm	40
Citric Acid	2.0	gm	
Thiourea	1.0	gm	
Zinc Chloride	0.30	gm	
2,3-dihydroxynaphthalene-6-sulfonic acid	2.0	gm	
Para-diethylaminobenzenediazonium chloride zinc chloride double salt	2.0	gm	45
Saponin	0.1	gm	
Dissolved in 100 cc of a 50% by volume acetone 50% by volume methanol solvent.			

## Sensitizing Composition C

Ethylene glycol	5.0	gm	50
Citric acid	2.0	gm	
Zinc chloride	0.15	gm	
2,3-dihydroxynaphthalene-6-Sulfonamide	2.0	gm	
Resorcinol	0.5	gm	
Acetoacetanilide	0.2	gm	
Para-dimethylaminobenzene diazonium chloride	1.0	gm	
Para-diethylaminobenzenediazonium chloride	1.0	gm	55
Saponin	0.1	gm	
Dissolved in 100 cc of a 50% by volume acetone, 50% by volume methanol solvent.			

A second set of examples are formulated, applied on 5 mil cellulose diacetate film and tested in exactly the same manner as Examples I through III. The sensitizing solution is the same in all cases, its components are as follows:

Parts by weight	Component
34	acetone
53	2-methoxyethanol
0.2	cellulose acetate
2	sulfosalicylic acid
0.1	stannic chloride
2	m,m' ethylene dioxy- diphenol
0.2	dirosorcyldisulfide
0.1	acetoacetanilide
1.5	orthoanisidide of but- oxynaphtholic acid
1.5	3 methyl-4-pyrroli- danyl benzene dia- zonium hexafluoro- arsenate

The samples set forth in Table 2, which follows, vary the type of thermo-setting resin used for a backwet solution. All of the resins are dissolved in a mixture containing 80 ml. acetone, 20 ml. 2 methoxyethanol as solvent and 0.3 g cellulose acetate as binder.

TABLE 2

Example	Resin	Amount	Additive	Amount
XIII	Control	—	—	—
XIV	Urea-formaldehyde 1.5 parts formal- dehyde to 1 part urea.	.5g. .5g	p. chlorobenzene sulfonic acid	0.2g.

A number of coatings are formulated, applied and tested in exactly the same manner as in Examples I-III.

TABLE 2—Continued

Example	Resin	Amount	Additive	Amount
XV	bisphenol A-epichlorohydrin epoxy resin having a molecular weight of 800 and an acid number of 200.	1.0g.	aluminum chloride	0.1g.
XVI	linear saturated polyester formed from ethylene glycol and 70:30 terephthalic and isophthalic acid, the copolymer having an acid number of 1.68 and a glass transition temperature of 67°C.	2.0g.	aluminum chloride	0.1g.
XVII		1.0g.		
XVIII	Acrylic resin copolymer formed from 3 parts ethylacrylate to 1 part methyl acrylate, containing 5% polyacrylonitrile crosslinking agent.	2.0g.		
XIX	Acrylic copolymer as in XIX hydroxyl modified vinyl acetate vinyl chloride copolymer	2.0		
XX		2.0g.		
		1.0g		

Results of tests performed on these Examples in exactly the same manner as in Example 1 show that transfer is substantially inhibited.

This invention has been developed with respect to certain preferred embodiments, and various modifications and variations thereof will become obvious to persons skilled in the art, and are to be included within the spirit and purview of this application and the scope of the appended claims.

What is claimed is:

1. A photographic element having a transparent polymeric film base support; a diazo-sensitizing composition coated on one side of said support; and, coated on the other side of said support, a backwet composition comprising a mixture of (a) a transfer preventing additive selected from the group consisting of: (1) particulate solids having a particle size of about 0.007 to about 0.012 microns selected from the group consisting of silica and alumina; (2) a cured thermosetting resin selected from the group consisting of amino resins, polyester resins, epoxy resins and phenolic resins; and (3)

a hydroxyl-modified vinyl chloride-vinyl acetate copolymer; and (b) a polymeric binder resin selected from the group consisting of cellulose triacetate, cellulose acetate butyrate, cellulose acetate propionate, polyvinyl chloride acetate, polystyrene, polyethylene terephthalate, and bisphenol-A-polycarbonate, said backwet composition being coated from an organic solvent containing coating composition, said solvent having a boiling point below about 140°C, wherein said binder, said resins and said copolymer being dissolved in said coating composition, and said polymeric film base support being the same polymer as said polymeric binder resin.

2. An element as defined in claim 1 wherein said element is disposed in layered assemblies in such a manner that said sensitized side is in at least close proximity to said backwet coated side.

3. A photographic element as defined in claim 1 wherein said element is microfilm.

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