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(54) Title: PHOTOGRAPHIC ANTISTATIC COMPOSITIONS AND ELEMENTS COATED THEREWITH

(57) Abstract

Photographic antistatic coating compositions comprising a hydrophilic binder, an anionic fluorinated surfactant and an inorganic nitrate. Photographic base elements and radiation-sensitive elements are protected against the adverse effects resulting from accumulation of static electrical charges by incorporating therein an antistatic layer formed from the aforementioned coating compositions.

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PHOTOGRAPHIC ANTISTATIC COMPOSITIONS AND ELEMENTS COATED THEREWITH

This invention relates to photography and in particular to photographic antistatic coating compositions and radiation-sensitive elements containing antistatic layers, formed from such compositions. More specifically, this invention relates to the use of such photographic antistatic coating compositions to provide protection from the adverse effects of static for photographic bases and radiation sensitive photographic elements, such as photographic papers and films.

The accumulation of static electrical charges on photographic films and photographic papers has long been a serious problem in the photographic art. These charges arise from a variety of factors during the manufacture, handling and use of radiation-sensitive, image-recording materials. For example, they can occur on photographic sensitizing equipment and on slitting and spooling equipment, and can arise when the paper or film is unwound from a roll or as a result of contact with transport rollers. The generation of static is affected by the conductivity and moisture content of the photographic material and by the atmospheric conditions under which the material is handled. The degree to which protection against the adverse effects of static is needed is dependent on the nature of the particular radiation-sensitive element. Thus, elements utilizing high speed emulsions have a particularly acute need for antistatic protection. Accumulation of static charges can cause irregular fog patterns in a photographic emulsion layer and this is an

especially severe problem with high speed emulsions. Static charges are also undesirable because they attract dirt to the photographic recording material and this can cause repellency spots, desensitization, fog and physical defects.

To overcome the adverse effects resulting from accumulation of static electrical charges, it is conventional practice to include an antistatic layer in radiation-sensitive elements. Such antistatic layers are often formed from photographic antistatic coating compositions composed of materials which dissipate the electrical charge by providing a conducting pathway. A large number of different materials have been proposed heretofore for use in antistatic layers of photographic elements. For example, U. S. Patent 584,862 issued June 22, 1897, describes the addition of an alkali nitrate such as potassium nitrate to a gelatin layer on the film support to prevent static discharges. Further, U. S. Patent 3,754,924 issued August 28, 1973, relates to photographic elements having antistatic layers comprising a fluorinated surfactant and solid water-insoluble discrete particles of a matting agent, such as silica. Unfortunately, the use of alkali nitrates or fluorinated surfactants to provide antistatic protection in radiation-sensitive elements, as described in the art, has suffered from one or more significant disadvantages. For example, in certain instances, antistatic layers containing only one of these types of materials have provided inadequate protection against static for high speed emulsions, such as those used in phototypesetting papers. Hence, there has been an unacceptable level

of defects in such elements caused by static discharge. Clearly, there is a need in the art for antistatic compositions which provide improved protection from static for radiation-sensitive elements.

While anionic fluorinated surfactants and inorganic nitrates have individually been used heretofore to provide static protection for radiation-sensitive elements, in the present invention, an anionic fluorinated surfactant and an inorganic nitrate are employed in combination. This combination of materials has been found to provide an antistatic layer which has highly improved static protection properties. Contrary to what may have been expected, and as illustrated in the following Example 1, such improved protection is significantly greater than either the protection provided by each component individually or the additive effect of the sum of their contribution to antistatic individual protection.

Moreover, the antistatic layers described herein provide important advantages, including the advantage that they can be coated from aqueous solution and the fact that they are durable, strongly adherent to the support, abrasion-resistant and non-tacky. Consequently, they do not contaminate equipment employed in manufacturing the radiation-sensitive elements nor processing baths or equipment used in processing the radiation-sensitive elements.

This invention provides a photographic antistatic coating composition, characterized in that it comprises an aqueous solution of a hydrophilic binder, an anionic fluorinated surfactant and an inorganic nitrate.

This invention also provides a photographic base element comprising a support coated with an antistatic layer of the photographic antistatic coating composition just described.

Further, this invention provides a radiation-sensitive element comprising a support, a radiation-sensitive, image-forming layer and an antistatic layer of the photographic antistatic coating composition described hereinbefore. Generally, the radiation-sensitive, image-forming layer is on one side of the support and the antistatic layer is on the other side of the support.

In addition, this invention provides a method of providing antistatic protection for a radiation-sensitive element. Such method comprises coating a surface of the element with the photographic antistatic coating composition described herein, and drying the resulting coating.

Any anionic fluorinated surfactant can be used in the coating composition and elements of this invention so long as it is compatible with the other components in such compositions and does not adversely affect the sensitometric properties of any radiation-sensitive layers in such elements. As used in this specification and in the claims, the term "surfactant" refers to a surface-active substance which alters (usually reduces) the surface tension of water. Such compounds are sometimes known as surface active agents. Useful anionic fluorinated surfactants include those described in U. S. Patent 3,754,924 issued August 28, 1973. Generally, such surfactants are either water soluble or water dispersible.

Particularly useful anionic fluorinated surfactants are those of the formula R-X wherein R is a fluorinated hydrocarbon, wherein some or all of the hydrogen atoms are replaced by fluorine atoms. Such hydrocarbons include alkyl, often of from 1 to 30 carbon atoms; cycloalkyl, often of from 5 to 30 carbon atoms; and aryl, often of from 6 to 30 carbon atoms. In the preceding formula, X is a hydrophilic anionic group, such as $-SO_3M$, $-OSO_3M$ or -COOM. M is a monovalent cation, such as hydrogen; an alkali metal ion, such as Na^+ or K^+; ammonium ion or an organic ammonium group such as diethanolammonium, morpholinium, pyridinium, tetramethylammonium and tetraethylammonium.

In a preferred embodiment, in the formula R-X, R is partly or wholly fluorinated alkylene of from 6 to 30 carbon atoms and X is $-SO_3M$. Particularly useful surfactants are those of the formula $CF(CF)_7SO_3M$. Mixtures of anionic fluorinated surfactants can be used if desired.

The inorganic nitrates useful in the compositions and elements of this invention are any of the water soluble or water dispersible salts of nitric acid. Examples of useful salts include ammonium nitrate and metal nitrates, such as aluminum nitrate, alkali metal nitrates and alkaline earth metal nitrates. Preferred nitrates are the alkali metal nitrates, such as lithium nitrate, sodium nitrate and potassium nitrate and alkaline earth metal nitrates, such as calcium nitrate and magnesium nitrate. Mixtures of nitrates can be used if desired.

The photographic antistatic coating compositions used in practicing this invention also contain one or

more water-soluble, film-forming hydrophilic binders. Suitable hydrophilic binders include both naturally occurring substances such as proteins, protein derivatives, cellulose derivatives, e.g. cellulose esters, gelatin, gelatin derivatives, polysaccharides, collagen derivatives; and synthetic hydrophilic polymeric materials. Examples of useful hydrophilic binders are described, for example, in Research Disclosure, publication 17643, December, 1978, p. 26, paragraph IX (published by Industrial Opportunities, Ltd., Homewell, Havant Hampshire P09 1EF United Kingdom) and U. S. Patent 4,196,001 (issued April 1, 1980 to Joseph et al). A particularly useful binder is gelatin.

The proportions of the components making up the antistatic coating compositions of this invention can be varied widely to meet the requirements of the particular element which is to be provided with antistatic protection. In general, the anionic fluorinated surfactant is present in such compositions in an amount in the range of from 0.05 to 5 percent, by weight, based on total dry solids content of the composition. The inorganic nitrate is typically employed in an amount in the range of from 5 to 20 percent, by weight, based on the total dry solids content of the composition. The hydrophilic binder is present in an amount in the range of from 30 to 95 percent, by weight, based on the total dry solids content of the composition.

The photographic antistatic coating compositions of this invention can contain other ingredients in addition to hydrophilic binders, anionic fluorinated surfactants and inorganic nitrates. For example,

they can contain matting agents, such as silica, starch, titanium dioxide, polymeric beads, zinc oxide and calcium carbonate. Preferably, they contain silica. They can also contain coating aids, such as alcohols and surfactants, as long as they are compatible with the anionic fluorinated surfactants; hardeners, such as formaldehyde; and other addenda commonly employed in such compositions.

Photographic base elements which can be protected from the adverse effects of static with the antistatic layers described herein include photographic films prepared from a variety of support materials. For example, the film support can be cellulose nitrate film, cellulose acetate film, polyvinyl acetal film, polycarbonate film, polystyrene film, or polyester film. Polyester films, such as biaxially stretched, heat-set and heat-relaxed polyethylene terephthalate film, are especially useful. Photographic papers, especially those coated on one or both sides with a coating of a hydrophobic polymeric material, are also advantageously protected against static with the antistatic layers of this invention. Such polymer-coated photographic papers are well known and include paper coated with styrene polymers, cellulose ester polymers, linear polyesters, and polyolefins such as polyethylene or polypropylene.

The antistatic layers of this invention are usefully employed in radiation-sensitive elements intended for use in either black-and-white or color photography. In addition to the antistatic layer and one or more radiation-sensitive, image-forming layers, the radiation-sensitive elements can include

subbing layers, pelloid protective layers, filter layers, antihalation layers, and so forth. The radiation-sensitive, image-forming layers present in the elements can contain photographic silver halides as the radiation-sensitive material, for example, silver chloride, silver bromide, silver bromoiodide, silver chlorobromide, silver chloroiodide, silver chlorobromoiodide, and mixtures thereof. Often, these layers are photographic emulsion layers that contain a hydrophilic colloid. Illustrative examples of such colloids are proteins such as gelatin, protein derivatives, cellulose derivatives, polysaccharides such as starch, sugars such as dextran, plant gums, and synthetic polymers such as polyvinyl alcohol, polyacrylamide and polyvinylpyrrolidone. Conventional addenda such as antifoggants, stabilizers, sensitizers, development modifiers, developing agents, hardeners, plasticizers, coating aids, and so forth, can also be included in the photographic emulsion layers. The photographic elements protected with the antistatic layer as described herein can be films or papers sensitized with a black-and-white photographic emulsion, elements designed for reversal color processing, negative color elements, color print materials, and the like. Photographic silver halide emulsions, preparations, addenda, and processing techniques useful for the elements of this invention are described, for example, in Research Disclosure, publication 17643, December, 1978, pp. 22-31, noted previously herein.

The photographic antistatic coating composition described herein can be applied by any suitable

technique for the application of aqueous coating compositions. For example, it can be coated by spray coating, dip coating, swirl coating, extrusion hopper coating, curtain coating, air knife coating, or other coating techniques. The thickness of the coated layer will depend upon the particular requirements of the element involved. Typically, the dry weight coverage is in the range from 0.2 to 4 grams per square meter and most usually in the range from 1 to 3 grams per square meter. Drying of the coated layer can be carried out over a wide range of temperatures. For example, temperatures of from 20°C to 130°C and preferably from 75°C to 115°C generally give satisfactory results.

When the photographic antistatic coating composition is applied to a polyolefin coated paper support, it is advantageous to treat the polyolefin surface, by a suitable method such as corona discharge, ozone or flame treatment, to render it receptive to the coating composition. The paper which is used to prepare the support can also be tub sized with a solution of a conducting salt which acts as an internal antistat. It is also advantageous to employ paper stock containing at least 3%, and generally from 4 to 8% (by weight), moisture.

When the photographic antistatic coating composition is applied to a polyester film support, a subbing layer is advantageously employed to improve the bonding of the antistatic layer to the support. Subbing compositions for this purpose are known in the art and include, for example, interpolymers of vinylidene chloride such as vinylidene chloride/acrylonitrile/acrylic acid terpolymers or vinylidene chloride/methyl acrylate/itaconic acid terpolymers.

The antistatic layers described herein can be in any position within the radiation-sensitive, image-forming element to provide effective protection against the adverse effects of static. However, they will ordinarily be the outermost layer of the element on the side opposite a radiation-sensitive photographic emulsion layer(s).

The antistatic properties exhibited by layers formed from the photographic antistatic compositions described herein can be evaluated by any one of a number of suitable techniques known in the prior art. One such technique is the so-called "impact electrification" method and apparatus for carrying out this method as described in U.S. Patent 3,501,653, issued March 17, 1970 and U.S. Patent 3,850,642, issued November 26, 1974. This impact electrification method was used to evaluate the antistatic layers prepared in the following examples.

A detailed description of an instrument suitable for measuring impact electrification is set forth in the aforementioned U.S. Patent 3,501,653. The instrument and the measured values obtained in the use thereof are defined and explained in detail therein. Stated simply, the theory of the instrument described in U.S. Patent 3,501,653 is that if accurate comparative values for impact electrification of a variety of surfaces is to be determined, a given reference surface must be impacted by a second (reference) surface and separated, all in a controlled and repeatable manner. The electrical charge generated by the impact and separation is accurately measured

and recorded. The values obtained are conveniently expressed in microcoulombs per square meter.

The invention is further illustrated by the following examples of its practice.

Example 1

A photographic antistatic coating composition according to this invention (designated Example 1 hereinafter) was prepared by adding the following components to sufficient water to give approximately 3.8% solids:

	parts*
gelatin (91% solids)	78.69
Alkanol®-XC surfactant*** (10% solids)	0.52
Fluortenside® FT-248** (2% solids)	0.08
$NaNO_3$	12.06
$Al(NO_3)_3$ (25% solids)	0.79
silica	7.34
formaldehyde (40% solids)	0.52

* parts per 100 parts solids

** anionic fluorinated surfactant available commercially from Bayer AG, Bayerwerk, Leverkusan NRW D-5090, Federal Republic of Germany

***salt of a naphthalene sulfonate, available commercially from E. I. Dupont de Nemours and Co., Wilmington 98, Delaware, U.S.A.

Control antistatic compositions A, B and C were also prepared having the following distinctions compared to the previously described antistatic composition (Example 1):

Control A	Fluortenside® FT-248 omitted
Control B	$NaNO_3$ and $Al(NO_3)_3$ omitted
Control C	Fluortenside® FT-248, $NaNO_3$ and $Al(NO_3)_3$ omitted

Photographic base elements were prepared by applying each of the antistatic compositions (Example 1 and Controls A-C) to polyethylene-coated paper photographic support in an amount sufficient to provide dry weight coverages ranging from 1 to 2 grams per square meter of support.

The antistatic properties of the antistatic layers formed from the compositions were evaluated by the "impact electrification" method as described previously herein. A test area of each photographic base element was impacted (impact pressure 20 psi (138 kPa)) with a polyurethane elastomer in the apparatus and according to the technique described in U.S. Patent 3,501,653. The charge generated (microcoulombs/square meter) was measured and recorded. The results are listed in Table I.

Table I

	Charge (microcoulombs/square meter)
Example 1	+27
Control A	+82
Control B	+63
Control C	+86

These results were evaluated to determine if the charging effect of the nitrates and the anionic fluorinated surfactant were additive or greater than additive. This evaluation was made by comparing the responses, as follows:

C-B = change in electrification level between Controls B and C;

C-A = change in electrification level between Controls A and C;

(C-B) + (C-A) = total theoretical effect assuming additive effects;

C-1 = actual change in electrification level between Control C and Example 1.

C-1 was then compared to [(C-B) + (C-A)] by subtracting the latter from the former. This difference between actual and theoretical additive effects was determined to be +59-[(23) + (+4)] = +59 - [27] = +32. This result indicates that the antistatic effect of combining the nitrates with the anionic fluorinated surfactant was significantly greater than the sum of the effects of using each individually.

Similar results are obtained with photographic base elements that are coated on the side of the resin-coated paper support opposite the antistatic layer with a green sensitive, high speed, high contrast, gelatino-silver bromoiodide black-and-white photographic emulsion of the type used in photo-type-setting.

Example 2

U.S. Patent 3,754,924, indicates that matting agents such as silica combine with fluorinated surfactants to provide superior and unexpected

antistatic properties in photographic elements. Although silica is a useful component in the photographic antistatic coating compositions of this invention, its use is not responsible for the results achieved in the practice of this invention. To illustrate, Example 1 was repeated except that silica was omitted from all antistatic compositions.

The results of the "impact electrification" evaluations are listed in Table II.

Table II

Example 2	-15
Control A	+85
Control B	+58
Control C	+81

A comparison of the actual and theoretical additive effects was then made as in Example 1. The difference between actual and theoretical additive effects was found to be +77. This indicates that the unexpected improvement in antistatic protection obtained with the photographic coating compositions of this invention is not due to the presence of silica in such compositions.

Claims:

1. A photographic antistatic coating composition, characterized in that said composition comprises an aqueous solution of:

(a) a hydrophilic binder,

(b) an anionic fluorinated surfactant, and

(c) an inorganic nitrate.

2. The composition of claim 1, characterized in that said anionic fluorinated surfactant has the formula R-X wherein R is fluorinated hydrocarbon, and X is $-SO_3M$, $-OSO_3M$ or -COOM wherein M is a monovalent cation.

3. The composition either of claims 1 or 2, characterized in that said nitrate is an alkali metal nitrate.

4. The composition of any of claims 1, 2 or 3, characterized in that said nitrate is silver nitrate and said composition additionally contains silica.

5. A photographic base element comprising a support coated with an antistatic layer of a photographic antistatic coating composition, characterized in that said coating composition is a coating composition of claim 1.

6. The base element of claim 5, characterized in that said support is a film support, a paper support or a polyolefin-coated paper support.

7. A radiation-sensitive element comprising:

(1) a support,

(2) a radiation-sensitive, image-forming layer, and

(3) an antistatic layer of a photographic antistatic coating composition, characterized in that said coating composition is a coating composition of any of claims 1, 2, 3 or 4.

8. The radiation-sensitive element of claim 7, characterized in that said image-forming layer is on one side of said support and said antistatic layer is on the other side of said support.

9. The radiation-sensitive element of any of claims 7 or 8, characterized in that said radiation-sensitive, image-forming layer is a photosensitive silver halide emulsion layer.

10. The radiation-sensitive element of any of claims 7, 8 or 9, characterized in that said anionic fluorinated surfactant has the formula $CF_3(CF_2)_7SO_3M$ wherein M is an alkali metal, and said inorganic nitrate is sodium nitrate.

11. A method of providing antistatic protection for a radiation sensitive element, which method comprises coating a surface of said element with a photographic antistatic coating composition and drying said coating, characterized in that said antistatic composition is a coating composition of any of claims 1, 2, 3 or 4.

INTERNATIONAL SEARCH REPORT

International Application No PCT/US81/01416

I. CLASSIFICATION OF SUBJECT MATTER (if several classification symbols apply, indicate all) [3]

According to International Patent Classification (IPC) or to both National Classification and IPC [3]

G03C 1/84, 1/38; C09K 3/16

II. FIELDS SEARCHED

Minimum Documentation Searched [4]

Classification System	Classification Symbols
U.S.	57/901; 106/125; 252/8.6; 260/Dig 15, 117; 430/527 528, 529, 631, 635

Documentation Searched other than Minimum Documentation to the Extent that such Documents are Included in the Fields Searched [5]

III. DOCUMENTS CONSIDERED TO BE RELEVANT [14]

Category *	Citation of Document, [16] with indication, where appropriate, of the relevant passages [17]	Relevant to Claim No. [18]
A	US,A, 584,862, Published 22 June 1897 EASTMAN	1,3,5-6
A	US,A, 2,725,297, Published 29 November 1955, MOREY	1,3,5-9,11
A	US,A, 3,437,484, Published 8 April 1969, NADEAU	1
A	US,A, 3,514,289, Published 26 May 1970, GOFFE ET AL.	1,5-9,11
A	US,A, 3,525,621, Published 25 August 1970, MILLER	4
A	US,A, 3,630,740, Published 28 December 1971 JOSEPH ET AL.	4
X	US,A, 3,753,716, Published 21 August 1973, ISHIHARA ET AL.	1,2,5-9,11
A	US,A, 3,754,924, Published 28 August 1973, DE GEEST ET AL.	1,2,5-11

* Special categories of cited documents: [15]

"A" document defining the general state of the art

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"L" document cited for special reason other than those referred to in the other categories

"O" document referring to an oral disclosure, use, exhibition or other means

"P" document published prior to the international filing date but on or after the priority date claimed

"T" later document published on or after the international filing date or priority date and not in conflict with the application, but cited to understand the principle or theory underlying the invention

"X" document of particular relevance

IV. CERTIFICATION

Date of the Actual Completion of the International Search [2]	Date of Mailing of this International Search Report [2]
2 December 1981	10 DEC 1981
International Searching Authority [1]	Signature of Authorized Officer [20]
ISA/US	Mary F. Downey

Form PCT/ISA/210 (second sheet) (October 1977)

FURTHER INFORMATION CONTINUED FROM THE SECOND SHEET

A	US,A, 3,850,642, Published 26 November 1974, BAILEY ET AL.	1
A	US,A, 3,884,699, Published 20 May 1975, CAVALLO ET AL.	1;5-9,11
A	US,A, 3,888,678, Published 10 June 1975, BAILEY ET AL.	1
A	US,A, 4,175,969, Published 27 November 1979, MACKEY	1
A,P	US,A, 4,267,265, Published 12 May 1981, SUGIMOTO ET AL.	1,2,5-11

V. ☐ OBSERVATIONS WHERE CERTAIN CLAIMS WERE FOUND UNSEARCHABLE [10]

This international search report has not been established in respect of certain claims under Article 17(2) (a) for the following reasons:

1. ☐ Claim numbers _____, because they relate to subject matter [12] not required to be searched by this Authority, namely:

2. ☐ Claim numbers _____, because they relate to parts of the international application that do not comply with the prescribed requirements to such an extent that no meaningful international search can be carried out [13], specifically:

VI. ☐ OBSERVATIONS WHERE UNITY OF INVENTION IS LACKING [11]

This International Searching Authority found multiple inventions in this international application as follows:

1. ☐ As all required additional search fees were timely paid by the applicant, this international search report covers all searchable claims of the international application.

2. ☐ As only some of the required additional search fees were timely paid by the applicant, this international search report covers only those claims of the international application for which fees were paid, specifically claims:

3. ☐ No required additional search fees were timely paid by the applicant. Consequently, this international search report is restricted to the invention first mentioned in the claims; it is covered by claim numbers:

Remark on Protest

☐ The additional search fees were accompanied by applicant's protest.

☐ No protest accompanied the payment of additional search fees.

FURTHER INFORMATION CONTINUED FROM THE SECOND SHEET

A	JP,B,38-17896, Published 11 September 1963,	1
A	GB,A, 1,118,324, Published 3 July 1968	1,3
A	N, Research Disclosure, issued February 1978, publication 16630	1,2,5-11

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3. ☐ No required additional search fees were timely paid by the applicant. Consequently, this international search report is restricted to the invention first mentioned in the claims; it is covered by claim numbers:

Remark on Protest

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☐ No protest accompanied the payment of additional search fees.