

[54] **PROCESS AND COMPOSITIONS FOR PROTECTING IMAGES WITH RESIN FILMS**

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[57] **ABSTRACT**

Novel process and compositions are provided for forming protective coatings. The compositions feature a graft copolymer suspension in an electrically insulating carrier vehicle. The copolymer is comprised of a linear backbone chain formed of an unsaturated polymer soluble in the carrier vehicle, having grafted thereto pendant side chains substantially all of which are insoluble in the carrier vehicle. A developed image-bearing member which is contiguous with an electrically conductive backing is placed in close proximity to an electrode and a bias potential is applied between the electrode and the conductive backing. While this potential is being applied, the copolymer suspension is applied to the image-bearing surface of the member to deposit a uniform resin layer.

8 Claims, No Drawings

PROCESS AND COMPOSITIONS FOR PROTECTING IMAGES WITH RESIN FILMS

This invention relates to electrography, and in particular, it relates to a process and compositions for forming protective coatings and for fixing developed electrostatic charge patterns.

Electrographic and particularly electrophotographic imaging processes and techniques have been extensively described in both the patent and other literature, for example, U. S. Pat. Nos. 2,221,776; 2,227,013; 2,297,691; 2,357,809; 2,551,582; 2,825,814; 2,833,648; 3,220,324; 3,220,831; 3,220,833 and many others. Generally, these processes have in common the steps of employing a normally insulating photoconductive element which is prepared to respond to imagewise exposure with electromagnetic radiation by forming a latent electrostatic charge image. The electrostatic latent image is then rendered visible by a development step in which the charged surface of the photoconductive element is brought into contact with a suitable developer mix.

A particularly useful procedure for developing electrostatic latent images is through the use of a liquid developer as described, for example, by Metcalfe in LIQUID DEVELOPERS FOR XEROGRAPHY, Journal of Scientific Instruments, Vol. 32 (1955), pages 74 and 75. Developers of this type are comprised basically of a simple dispersion of pigment. Because no binder is present in the developer, the resultant images must be separately fixed. It was later proposed, for example, by Metcalfe and Wright, Journal of Oil and Color Chemists' Associations, 39, (1956), pages 851-853, that liquid developers containing a resin be used. The resultant images are then self-fixing because of the presence of the resin. These self-fixing developers are very useful for preparation of a limited number of images from a single batch of developer. However, problems often arise where it is desired to make a large number of copies from a single batch of developer. It appears as if the developer and the resin are not always depleted at the same rate. In view of the differing depletion rates, replenishment is often difficult or impossible. Thus, for certain applications, it is more desirable to use the simple developers disclosed earlier by Metcalfe or a variation containing only a small amount of resin and to fix the resultant image by a subsequent procedure.

One useful procedure for subsequent fixing of developed images is the application of a lacquer overcoat. Various procedures have been suggested for applying such a lacquer overcoat. The most popular method has been to spray a resin solution onto the developed image. Another method has been to apply a resin solution from an applicator roller. Both of these systems have the disadvantage that the resin solution is exposed to the air which causes drying of the solution. This in turn results in the applicating apparatus becoming "gummed up." A further disadvantage with such systems utilizing a resin solution is that the solvent for the resin is different from and generally incompatible with the carrier liquid of the developer used. Because of this incompatibility, it is necessary to dry a liquid developed image prior to applying the resin solution. In addition, there is often a problem of venting any toxic or dangerous solvent vapors.

The major difficulties with such prior overcoating procedures have been overcome by the procedures described in Mosehauer and Rodenberg copending application Ser. No. 854,324 filed Aug. 29, 1969, and entitled METHOD OF PROTECTING IMAGES now abandoned. The method described therein includes the electrophoretic deposition of a protective coating from a suspension of resin particles in an insulating liquid. Such resin suspensions are prepared by first dissolving a suitable polymer, along with a dispersing agent, in a solvent such as methylene chloride. This combination is then added to a hydrocarbon liquid in which the polymer is insoluble to form a dispersion. This procedure thus requires that the polymer be prepared in one operation and dispersed in another operation. A further difficulty with this procedure is that stable suspensions of many desirable polymers cannot be prepared in this manner.

Still another difficulty is encountered in replenishing a coating bath of this type. Preferably, such a bath would be replenished by the addition of a resin concentrate. Typically, a coating bath would be used to coat a number of elements. During the coating procedure the resin and dispersing aid will be depleted from the suspension although only a small amount of the suspension medium will be used. Consequently, replenishment requires the addition of a concentrated suspension which contains, by necessity, a higher concentration of polymer and dispersing agent than does the operating suspension. However, it is difficult to accurately maintain the balance of materials in that all of the components are not depleted at the same rate. Consequently, this can result in a buildup of one component over a period of time which in turn results in non-uniform coating conditions.

Accordingly, there is a need for resin suspensions which are easily prepared, which are stable and which can be readily replenished during use.

It is, therefore, an object of this invention to provide novel resin suspensions which are readily prepared and which are stable.

It is another object of this invention to provide novel resin suspensions for use in forming protective coatings, which suspensions can easily be replenished during use.

It is a further object of this invention to provide a novel process for forming protective coatings particularly over developed images.

Still another object of this invention is to provide simple means for preparing resin suspensions for use in overcoating developed images.

It is an additional object of this invention to provide novel resin suspensions for use in fixing developed electrostatic charge patterns.

Still a further object of this invention is to provide resin suspensions containing resin particles which are self-fixing when deposited from the suspension.

These and other objects and advantages are accomplished in accordance with this invention by electrophoretically applying to an image-bearing member a uniform layer of resin from a dispersion of graft copolymer particles in a liquid suspension medium. It is readily apparent that the present invention can be utilized to form protective coatings over a variety of different types of images such as silver images, diazo images, etc., as well as electrographic images. However, for ease of presentation, reference will be made only to images prepared by electrographic means with the understanding that the image-bearing member could bear a silver image, for example.

The image-bearing members used in the present invention can take a variety of forms. A particularly useful member is in the form of an electrophotographic element. Such elements are typically comprised of a conducting support having coated thereon a photoconductive composition. Such elements are well known in the art and particularly useful ones employ a photoconductive layer comprised of an organic or an inorganic photoconductor in an electrically insulating resin binder. An element of this type is given a uniform surface charge and then exposed to a pattern of actinic radiation. This exposure causes reduction in the surface potential in accordance with the relative energy contained in the radiation pattern. This charging and exposing sequence results in the formation of an electrostatic charge pattern on the photoconductive element. Another useful image-bearing member is comprised simply of a sheet of an electrically insulating material such as poly(ethylene terephthalate). An electrostatic charge pattern can be placed on such a member by charge transfer of a pattern carried on a photoconductive element as described above. In addition, an electrostatic charge pattern can be applied by placing a stencil over the insulating sheet and subjecting the sheet to a corona discharge. The insulating sheet in this instance can be provided with a permanent conductive backing or it can be simply placed in contact with a separable conductive backing.

The resultant electrostatic charge pattern formed on the image-bearing member is then ready for development. The pattern can be readily developed by the now well-known liquid development techniques as disclosed, for example, in U. S. Pat. No. 2,907,674. In general, liquid developers useful for developing the electrostatic charge pattern are comprised of a colorant dispersed in an electrically insulating carrier vehicle. These colorants can be pigment particles or colored resin particles which are electrostatically deposited in accordance with the charge pattern carried on the image-bearing member. In addition, the charge patterns can be developed by dry development techniques which utilize dry particles of coloring material such as pigment particles and/or pigmented resin particles.

After development of the electrostatic charge pattern to form a visible image, the resultant image is fixed in accordance with this invention by the electrophoretic deposition of a uniform layer of a graft copolymer over the entire image surface of the image-bearing member. The electrophoretic deposition is accomplished by providing an electrode (or counter electrode as it is sometimes called) closely spaced from the image-bearing surface of the image-bearing member and providing an electrical bias potential between this counter electrode and the conductive backing of the image-bearing member. The counter electrode is usually spaced about $\frac{1}{2}$ to about 6 mm from the image-bearing member, with a preferred spacing of about $\frac{1}{2}$ to about 2 mm. However, this distance can be varied to suit particular needs. The bias voltage applied between the counter electrode and the conductive backing can range from about 500 to about 5,000 volts depending upon the spacing of the counter electrode. A preferred bias voltage ranges between about 800 and 2,000 volts, but of course, the voltage can be varied depending on the particular graft copolymer suspension used. The conductive backing is usually negative with respect to the counter electrode; however, this will vary depending upon the polarity of the copolymer particles in the resin suspension.

Next, a graft copolymer suspension of this invention is introduced in the space between the counter electrode and the surface bearing the developed image. This suspension can be flowed through the space or the image-bearing member, etc., can be immersed in the copolymer suspension. The image-bearing member is then removed, the bias potential is turned off and the member is subsequently heated or otherwise treated to eliminate excess carrier liquid and fix the copolymer which is uniformly deposited over the entire surface of the image-bearing member.

The resin suspensions of the present invention are generally comprised of an electrically insulating carrier vehicle having formed therein insoluble particles of a graft copolymer having a linear backbone chain formed of a polymer which is soluble in the carrier vehicle. Grafted to the backbone chain are pendant side chains substantially all of which are insoluble in the carrier vehicle. In general, about 75 percent by weight of the pendant polymeric side chains are insoluble in the carrier. The side chains comprise repeating units derived from one or more monomers of the type described below. The suspensions of this invention are relatively simple to prepare. A dilute solution of the soluble polymer which forms the backbone chain is prepared using a liquid hydrocarbon solvent which becomes the carrier vehicle of the final resin suspension. Next, a substantially insoluble monomer is added to the solution along with a free radical initiator. The combination is then heated and polymerization occurs thereby forming insoluble graft copolymer particles as a concentrated stable suspension in the hydrocarbon solvent. This concentrated suspension can then be diluted with a hydrocarbon liquid to prepare a working resin suspension of the desired concentration. The concentrated suspensions as formed can also be used to replenish a working suspension which has become depleted during use.

The method of this invention has the advantage that the copolymer particles and the resin suspensions are formed in one operation. The resultant suspensions also have the ad-

vantage of being comprised of only two components, namely the graft copolymer particles and the suspension medium or carrier vehicle. Unlike the prior resin suspensions discussed above, there is no need in the present invention for a dispersing agent. The soluble starting polymer forming the linear backbone chain of the graft copolymer serves as a suspending agent without the need of any further additives.

The starting polymers useful in accordance with this invention can be selected from a wide variety of materials. The starting polymers must be soluble in the carrier vehicle desired to be used and the polymer must be capable of forming a graft copolymer with the monomer used. In general, the starting polymers are unsaturated polymers such as various polyenes. Typical examples of polymers of this type would include those made from isoprene, butadiene, 2,3-dimethylbutadiene, chloroprene, etc., as well as mixtures thereof. If desirable, saturated polymers, such as atactic polypropylene, could be used if oxidized to form grafting sites. The starting dilute polymer solutions can have from about $\frac{1}{4}$ to about 20 percent by weight of the polymer depending upon the molecular weight of the starting polymer. With high molecular weight polymers such as natural rubber, useful polymer concentrations should not exceed about 1 percent. However, with lower molecular weight polymers, the concentration can be increased to about 20 percent by weight.

The liquids useful both as solvents for the starting polymer and as the suspension medium or carrier vehicle for the final graft copolymer particles include a variety of materials. Suitable liquids are generally electrically insulating and include aliphatic hydrocarbons in which the starting polymer is soluble and in which the graft copolymer is insoluble. Electrically conductive liquids can be used if it is desired to overcoat conductive image-bearing members, provided, of course, that the solvent properties of the liquids are suitable for forming a graft copolymer suspension. A further property of the carrier liquid which is desirable is that the liquid be relatively volatile in order to facilitate drying after application of the protective resin overcoat. Volatility may be considered in a relative manner as the time required for 0.5 cc. of a liquid to evaporate from filter paper at room temperature and atmospheric pressure. Kerosene is found to have an evaporation rate in excess of 300 minutes in accordance with this test and thus represents the lower limit of volatility for useful liquids. In addition, preferred liquids should have a high flash point, preferably above about 30° C. (Tag Open Cup Test), in that an explosion danger is present with highly volatile materials having a lower flash point. Of course, liquids having a low flash point can safely be used when mixed with a liquid having a higher flash point. Additionally, in the case when the image to be protected has been liquid developed but not dried, the carrier vehicle in the resin dispersion should be compatible with the electrically insulating liquid of the developer. Thus, the carrier should be soluble in or miscible with this developer liquid and the two liquids should not be reactive. Furthermore, the suspension medium should have no substantial solvent effect on the previously deposited toner particles. Useful carrier liquids include a wide variety of relatively volatile organic liquids. Suitable liquids useful alone or in mixtures include many aliphatic hydrocarbons such as cyclohexane, n-hexane, n-heptane, n-decane; halogenated hydrocarbons such as carbon tetrachloride, chlorinated fluorinated lower alkanes; isoparaffinic hydrocarbons such as Isopar G (Humble Oil & Refining Co.); and the like.

The monomers which are added to the dilute starting polymer solution can be varied widely. Particularly useful materials include acrylic and methacrylic acids and esters, styrene, acrylonitrile, vinyl esters, vinyl ethers, etc. Mixtures of monomers are also very well suited for use in this invention. Alkyl acrylates and alkyl methacrylates including mixtures thereof having from two to 18 carbon atoms in the alkyl moiety are very useful, with from two to five being preferred. The alkyl moiety of these monomers can be straight or branch

After the monomers are added to the dilute starting polymer solution, a free radical initiator is added to the mixture. Suitable catalysts or free radical initiators for use in the graft copolymerization reaction include soluble organic peroxides. The temperature at which the polymerization reaction is conducted will, of course, depend upon the rate of decomposition of the peroxide initiator. The reaction is generally conducted at temperatures above room temperature, with an upper temperature limit being about 150° C. Using benzoyl peroxide, for example, the reaction is run at a temperature of about 80° C.

After preparation of the copolymer as above, the concentrated suspension is diluted to a suitable working strength by addition of more of the carrier vehicle. In general, the copolymer concentration is kept at a relatively low value of from about 1/2 to about 10 g/l., with a preferred concentration being from about ** to about 5 g/l. The copolymer concentration is, of course, variable, with a particular concentration being dependent upon the results desired.

Desirable properties of the copolymers useful in this invention include the following: high gloss and hardness, water-white color and transparency, resistance to ultraviolet degradation, high abrasion resistance, resistance to discoloration at reasonably high temperatures, and resistance to water, alcohol and dilute acids and alkalis. A further useful property of the generally transparent copolymers is that after deposition on an image-bearing member, the copolymer must be capable of being permanently fixed thereto. Preferably, the copolymers are self-fixing and are thus permanently fixed simply upon removal of the carrier vehicle. Graft copolymers which are self-fixing have a glass transition temperature of below about 35° C. Glass transition temperature (T_g) as used herein refers to the temperature at which a polymeric material changes from a glassy polymer to a rubbery polymer. This temperature (T_g) can be measured by differential thermal analysis as disclosed in "Techniques and Methods of Polymer Evaluation," Vol. 1, Marcel Dekker, Inc., New York, 1966. Useful copolymers also include those which can be fixed by a slight heating to cause fusing or crosslinking thereby forming a protective coating. Copolymers of this latter type generally have a glass transition temperature below about 65° C.

The relative stability of the various graft copolymer suspensions of this invention is dependent upon the relative concentration of the monomers and polymers used in preparing the suspension. In general, useful results are obtained using a weight ratio of monomer to polymer of from about 15:1 to 1:1, with preferred compositions being prepared using a monomer to polymer ratio of from about 12:1 to 2:1. Of course, these limits can be varied depending upon the results desired and upon the particular monomers used.

The following examples are included for a further understanding of the invention.

Example 1

A solution is prepared using 5 percent by weight of polyisoprene (IR310, Shell Chemical Co.) in an isoparaffinic hydrocarbon solvent having a boiling range of about 160° to 180° C (Isopar G sold by Humble Oil & Refining Co.) with stirring for 15 hours at 140° C. The resultant solution is diluted with more solvent until the polyisoprene concentration is about 2 1/2 percent by weight. To 100 ml. of this 2 1/2 percent are added 10 g. of ethyl methacrylate and 0.10 g. of benzoyl peroxide. The mixture is flushed briefly with nitrogen and placed in a 80° C bath for 20 hours during which time graft copolymerization occurs. At the end of this time, a stable concentrated suspension of copolymer particles is removed from the bath and diluted with additional Isopar G until the solids content is about 2 percent by weight. The resultant diluted suspension remains stable and the copolymer particles do not settle out. The copolymer particles have a positive electrostatic charge.

Example 2

A solution is prepared of 2 grams of styrene-butadiene copolymer in 100 ml. of Isopar G using the general procedure

of Example 1. Next, 0.10 grams of benzoyl peroxide and 10 grams of ethyl methacrylate are added to the solution containing the styrene-butadiene copolymer. The mixture is flushed with nitrogen and placed in a 80° C bath for about 20 hours during which time graft copolymerization occurs. The resultant product is a stable concentrated suspension of graft copolymer particles dispersed in Isopar G. Additional Isopar G is added to the reaction product to form a working strength resin suspension.

Example 3

A liquid developer is prepared from the following ingredients:

15	Carbon black in a soya-modified phthalic alkyd resin (3046 Carbon Black Paste, Interchemical Corp.)	0.75 grams
	Alkali Blue RR Paste (Sherwin Williams Co.)	0.25 grams
	Cyclohexane	30 ml.
20	Cobalt naphthenate (6% solution) (Uversol Cobalt Liquid, Harshaw Chemical Co.)	1.2 ml.
	Isopar G (supra)	970 ml.

The developer is prepared by mixing the two pastes in a solution of cyclohexane and cobalt naphthenate liquid to form a developer concentrate. The Isopar G is then added to the concentrate to form the final working developer. This liquid electrophotographic developer is then used to develop an image carried on an electrophotographic element comprising a poly(ethylene terephthalate) support bearing an evaporated nickel conducting layer which in turn bears a photoconductive layer comprising 4,4'-diethylamino-2,2'-dimethyltriphenylmethane as the photoconductor in a polycarbonate binder. The electrophotographic element is charged, exposed and developed with the above developer to form a visible image. The resultant image is substantially unfixed as this developer does not fix well to the surface of this element. Even the application of a substantial amount of heat does not significantly increase the adhesion of the developed image. The developed image is then electrophoretically overcoated using the positive polarity graft copolymer suspension of Example 1. The developed electrophotographic element prepared above is placed parallel to and about 1 mm from an electrode. A bias potential (positive with respect to the electrode) of about 1,000 volts is applied between the electrode and the conducting layer of the electrophotographic element. While this potential is applied, about 20 to 30 ml. of the copolymer suspension of Example 1 are flowed through the space between the electrode and the surface of the element carrying the developed image. The electrical bias potential provides a sufficient electric field to cause complete uniform deposition of graft copolymer particles over the full surface of the element. After the copolymer is deposited, the carrier liquid is evaporated using a stream of warm air and the element is then heated on a hot plate at a temperature of about 120° C to soften and fix the resin to the surface of the element. The resulting image is physically stable, with a smooth surface capable of taking considerable physical abuse. The transparent copolymer overcoat does not affect the quality of the image or impair optical readout or printout capability.

Example 4

The procedure of Example 3 is repeated in its entirety only using the copolymer suspension prepared according to Example 2. Again, the resin coating produced with the graft copolymer of Example 2 produces a physically stable, smooth surfaced coating which can take much physical abuse. Similarly, the transparent copolymer overcoat does not affect the quality of the image or impair the optical readout or printout capability.

Example 5

The procedure of Example 1 is repeated again, replacing 3 grams of the ethyl methacrylate with 3 grams of ethyl acrylate. The resultant suspension is then used as in Example 3 to form a continuous coating over a developed electrophotographic

element. The procedure involved with the suspension of this Example differs from the procedure of Example 3 in that the resin is not fused after deposition. Upon simply air drying to remove the carrier vehicle, the resin forms a clear, continuous coating which protects the underlying image.

Example 6

The procedure of Example 1 is again repeated, replacing the ethyl methacrylate with 5 grams of ethyl acrylate and 5 grams of methyl methacrylate. The copolymer suspension which results is then used as in Example 5 to form a self-fixing protective coating over the developed electrophotographic element.

The invention has been described in detail with particular reference to certain preferred embodiments thereof, but it will be understood that variations and modifications can be effected within the spirit and scope of the invention.

I claim:

1. A process for applying protective coatings to an image-bearing member having a developed image on a surface thereof which is contiguous to an electrically conducting backing, said process comprising the steps of applying an electric field to the image-bearing surface of said member; applying to the image-bearing surface of said member in the presence of said electric field a resin suspension comprising an electrically insulating liquid carrier vehicle having therein charge-bearing particles of a graft copolymer in order to deposit said copolymer particles uniformly over said member, said copolymer having a linear backbone chain formed of a polyene which is soluble in said carrier vehicle, said backbone chain having grafted thereto pendant side chains formed from monomers selected from the group consisting of acrylic and methacrylic acids, styrene, acrylonitrile, vinyl esters, and vinyl ethers, substantially all of said side chains being insoluble in said carrier vehicle; and affixing said uniformly deposited particles to said member.

2. A process for applying protective coatings to an image-bearing member, which member has a developed image on a surface thereof which is contiguous to an electrically conductive backing, using an electrode closely spaced from said image-bearing surface, said process comprising the steps of applying an electrical bias potential between such electrode and conductive backing; applying to the image-bearing surface of said member in the presence of said electrical bias potential a resin suspension comprising an electrically insulating liquid carrier vehicle having therein charge-bearing particles of a graft copolymer to deposit said copolymer particles uniformly over said member, said copolymer having a linear backbone chain formed of a polyene which is soluble in said carrier vehicle, said backbone chain having grafted thereto pendant side chains formed from monomers selected from the group consisting of acrylic and methacrylic acids, styrene, acrylonitrile, vinyl esters, and vinyl ethers, substantially all of said side chains being insoluble in said carrier vehicle; and affixing said uniformly deposited particles to said member.

3. A process as described in claim 1 wherein the carrier vehicle is an organic liquid having a volatility at least equal to that of kerosene.

4. A process as described in claim 1 wherein the polyene forming said backbone chain is formed by polymerizing a member selected from the group consisting of isoprene, butadiene, 2,3-dimethylbutadiene, chloroprene, styrene and mixtures thereof.

5. A process as described in claim 1 wherein the graft copolymer particles have a glass transition temperature below about 65° C. and wherein the carrier vehicle is an isoparaffinic hydrocarbon.

6. A process as described in claim 1 wherein the monomers forming said pendant side chains are vinyl esters selected from the group consisting of acrylic and methacrylic esters.

7. A process for fixing developed electrographic images on an electrographic element, which element has (a) an electrically conducting backing and (b) a visible, substantially unfixed, developed image on a surface thereof, using an electrode closely spaced from said image-bearing surface, said process comprising applying an electrical bias potential between such electrode and conductive backing; applying to the image-bearing surface of such element in the presence of said electrical bias potential a resin suspension comprising a hydrocarbon liquid carrier vehicle having therein charge-bearing particles of a graft copolymer to deposit said particles uniformly over the entire surface of said element, said copolymer having a linear backbone chain formed of a polyene which is soluble in said carrier vehicle, said backbone chain having grafted thereto pendant side chains formed from monomers selected from the group consisting of acrylic and methacrylic esters, said particles having a glass transition temperature below about 35° C.; and removing excess carrier vehicle to permanently fix the copolymer thereto.

8. A process for fixing developed electrographic images on a receiving member, which member (a) is contiguous to an electrically conducting backing and (b) has a visible, substantially unfixed, developed image on a surface thereof, using an electrode closely spaced from said image-bearing surface, said process comprising applying an electrical bias potential between such electrode and conductive backing; applying to the image-bearing surface of such member in the presence of said electrical bias potential a resin suspension comprising a hydrocarbon liquid carrier vehicle having therein charge-bearing particles of a graft copolymer to deposit said particles uniformly over the entire surface of said member, said copolymer having a linear backbone chain formed of a polyene which is soluble in said carrier vehicle, said backbone chain having grafted thereto pendant side chains formed from monomers selected from the group consisting of acrylic and methacrylic esters, said particles having a glass transition temperature below about 35° C.; and removing excess carrier vehicle to permanently fix the copolymer thereto.

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