United States Patent Office

3,296,140

Patented Jan. 3, 1967

3,296,140

LIQUID DEVELOPER FOR ELECTROGRAPHIC PRINTING

Daniel M. Zabiak, Chicago, Ill., assignor to A. B. Dick Company, Niles, Ill., a corporation of Illinois

No Drawing. Filed Feb. 13, 1964, Ser. No. 344,555
4 Claims. (Cl. 252—62.1)

This invention relates to the production of copy by an electrostatic copy process and it relates more particularly to a liquid toner for use in the development of a latent electrostatic image.

Liquid toners for use in the development of latent electrostatic images have been described in the issued Metcalfe et al. patents No. 2,907,674 and No. 3,058,914, and in the copending application of Metropoulos et al., Ser. No. 205,690, filed June 27, 1962, now abandoned, and entitled "Composition and Method for Developing Electrostatic Images." Such liquid toners have been found to be suitable for use in the development of latent electrostatic images formed by various means, such as by the "Xerox" process described by Dr. Carlson in Patent No. 2,297,691, wherein the surface of a dielectric coating on an electrically conductive base is charged by corona discharge from a suitable electrical source to produce a surface charge which can be imaged by exposure to light to destroy the surface charge in the exposed or non-imaged areas, leaving a latent electrostatic image on the unexposed areas; or such as by the "Videograph" process described in Patent No. 3,136,917 wherein a dielectric coating on an electrically conductive base is provided with a latent electrostatic image by discharge from conductive elements extending through the face of a cathode ray tube to write the latent electrostatic image on the grounded writing medium; or by the process described by Kalmon and Rennert, August 28, 1959, Electronics, entitled "Data Storage and Display With Polarized Phosphors," wherein a phosphor layer is exposed in a D.-C. field on the order of 50–2000 volts to cause polarization in depth of negative and positive charge carrying particles to produce a latent electrostatic image which remains rather permanent in the phosphor coating in the absence of light; or in the electroelectret process described in the copending application of Kalman et al., Ser. No. 333,542, filed December 26, 1963, entitled "Method of Producing Imaged Spirit Master Directly From Original," wherein the phosphor layer or electret is exposed first to a high D.-C. voltage between electrodes to cause movement of the negative and positive charge particles whereby one face of the phosphor acquires a highly positive charge while the opposite face acquires a highly negative charge after which the charged electret can be exposed to an original or a negative to dissipate the charge in the exposed areas, leaving a latent electrostatic image in the unexposed areas which remains in the absence of light.

It has been found that the character of the copy produced by liquid toners for the development of latent electrostatic images is dependent greatly on the speed of development, especially in substantially continuous copy processes, such as in the "Videograph" process wherein an image is being retained substantially continuously on an endless writing medium or tape for substantially continuous development of the latent electrostatic image that is written on the tape. A similar situation would apply to electrostatic copy processes which are in substantially constant usage, such as in the "Xerox" type copy machines.

When the latent electrostatic image is developed slowly or over a prolonged period of time by exposure to the liquid toner, the copy tends to become over-developed

and vice versa if the exposure to the liquid developer is short. When copy production is substantially continuous or frequent, toner particles are removed at a more rapid rate whereby the liquid toner becomes depleted of pigment particles such that subsequent copy tends to become under-developed whereas, when copy production is slow or less frequent, pigment concentration tends to build up in the liquid toner with the result that the copy tends to become over-developed.

It is an object of this invention to provide a liquid toner system for use in development of latent electrostatic images whereby copy of good quality can be secured substantially independently of the many variables in development such as speed of development frequency, and it is a related object to provide a liquid toner system formulated into elements for ready compensation of the variables without noticeable change in the characteristics or composition of the liquid toner thereby to provide a liquid developing system which is easy to control; which enjoys universal use in the development of electrostatic images substantially independently of the manner of formation and substantially independently of the mode or speed of development; which is easily formulated of readily available and low cost materials; which maintains substantially the same flow characteristics independent of concentrations; in which the pigment level can be varied in use without the need for modification in other of the operating characteristics of the copy process or machine; which is self-setting upon drying on the copy paper to form a substantially permanent image; and which can be stored as separate elements over extended periods of time and combined in the desired ratio for optimum copy quality.

In accordance with the practice of this invention, the liquid developer is formulated of a group of compatible materials which combine to form a liquid developing mixture and which may be subdivided into (1) a compatible carrier system formulated of the materials making up the toner in the absence of pigment for dilution of the pigment concentration in the developing liquid without noticeable effect on the viscosity, flow, stability or other characteristics of the liquid and (2) a compatible concentrate formulated of a high concentration of pigment for addition to the developing liquid to increase the pigment concentration without undesirable effect on the stability, viscosity, flow, or other desirable characteristics of the liquid thereby to provide a compatible system of components which can readily be adapted to meet the many variables within the copy process.

The invention will be described first with reference to the formulation of the developing composition per se and thereafter with reference to the carrier composition and concentrate for preparation of the liquid developing composition and/or for the adjustment thereof to increase or decrease the pigment concentration and the like. The following example of the developing composition is given by way of illustration, but not by way of limitation:

EXAMPLE 1

	Grams
Polystyrene resin (Piccolastic C125)	2
Aromatic solvent (aromatic petroleum fraction having a boiling point of 284–325° F., flash point 90° F.) (Napoleon 100–A—Kerr McGee Oil Co.—Deep Rock Division)	27.5
Aliphatic petroleum solvent (flash point 65° F., boiling point range 285–310° F.)	18.5
Trichlorotrifluoroethane (Freon TF)	16.7
Trichloromonofluoromethane (Freon MF)	33.3
Nigrosine dye	0.4
Aliphatic petroleum solvent No. 429 (flash point 65° F., boiling point fraction 285–310° F.)	1.6

Procedure

Since the polystyrene dissolves rapidly in aromatic solvents, the polystyrene is dissolved first in the aromatic solvent and then the aliphatic solvent and Freons are added.

The nigrosine dye is ball milled in the second increment of the aliphatic solvent to reduce the pigment to a small particle size, such as to about 1–5 microns, and then the grind is dispersed in the solution containing polystyrene producing a relatively stable liquid developer.

The nigrosine pigment particles will assume a positive charge within the liquid and will thus be attracted to a latent electrostatic image having a negative electrostatic charge to provide a clean and precise development of copy.

Polystyrene, with or without plasticizers such as hydrogenated abietate, resinates, etc., is preferred as the resinous binder in the developing composition since polystyrene is characterized by extremely low solution viscosity such that wide variations in concentration can be effected without material change in the viscosity level and it imparts the desired charge to the particles. The polystyrene serves as a film former and binder to anchor the pigment particles to the copy sheet upon drying thereby to fix the image without the necessity for making use of heat, solvent or the like, as heretofore employed, for affixing the image as in dry powder development. The polystyrene is also a good film former to provide a protective coating over the face of the copy sheet and the image fixed thereon. It is sufficient if the polystyrene is present in an amount at least as great as 0.5 percent by weight in the developing composition but it is undesirable to exceed a concentration greater than 5 percent by weight and best results are secured when the polystyrene is present in an amount within the range of 1–3 percent by weight of the composition.

The nigrosine is employed in the final liquid developer in an amount within the range of 0.05 to 0.6 percent by weight of the composition and preferably in an amount within the range of 0.2 to 0.5 percent by weight. Nigrosine is only representative of the various dyestuffs which may be employed since other dispersible dyestuffs which are insoluble in aliphatic or aromatic solvents can be employed in equivalent amounts. When the coloring agent comprises a pigment formed of a dyestuff in a resinous base, the pigment may be as high as 5 percent by weight.

The following will represent the formulation of a carrier which may be used to dilute a liquid developer to reduce the pigment or nigrosine concentration:

EXAMPLE 2

Carrier:	Grams
Polystyrene resin (Piccolastic C125)	1.5
Aromatic solvent (Napoleon 100–A)	27
Aliphatic solvent (Solvent No. 429)	18
Trichlorotrifluoroethane	18
Trichloromonofluoromethane	36

Procedure

Again, the resin is first dissolved in the aromatic solvent and then the remainder of the ingredients are added to form a pigmentless fluid which still contains the fixing resin so that dilution of the liquid developer with the carrier liquid to reduce the concentration of pigment or dyestuff will not operate correspondingly to reduce or dilute the amount of fixing resin or film former. As in the liquid developer or toner, the amount of resin in the carrier can be varied to within the range of 0.5 to 5 percent by weight and preferably within the range of 1–3 percent by weight.

The following will represent the formulation of a concentrate which may be used, in accordance with the practice of this invention, to increase the concentration of pigment in the liquid developer or toner without loss in the stability or material change in the viscosity thereof:

EXAMPLE 3

Concentrate:	Grams
Nigrosine dye	7.5
Aliphatic solvent (DT–250—an aliphatic petroleum solvent having a flash point of 105° F. and a boiling point range of 312–349° F. and a KB number of 39)	22.5
Polystyrene resin	30
Aromatic solvent (Napoleon 100–A)	35

EXAMPLE 4

Concentrate:	Grams
Nigrosine dye	7.5
Aliphatic petroleum solvent	22.5
Polystyrene resin	30
Aromatic petroleum solvent	35
Freon BF	5

In the above formulations, the nigrosine dye is first ground with the aliphatic solvent to a particle size of 1–5 microns. The polystyrene resin is separately dissolved in the aromatic solvent and the Freon and the two compositions are mixed together and given a final dispersion to form the concentrate. The nigrosine concentration is the important component in the concentrate and, for such purpose, it is desirable to make use of nigrosine or other dispersible dyestuffs in an amount more than three times the pigment concentration in the liquid toner or more preferably an amount within the range of 3–15 percent by weight of the concentrate composition.

The fluorinated hydrocarbon solvents, such as the Freons, are employed primarily for the purpose and in an amount sufficient to elevate the flash point of the composition to well over 100° F. and to facilitate drying of the copy. For this purpose, fluorinated or fluorinated chlorinated hydrocarbons, other than those set forth in the specific examples, may be employed and the amount can be varied in the solvent system, depending upon the fluorinated or fluorinated chlorinated hydrocarbon, to give the desired increase in flash point and control in drying rate.

Similarly, other well known aliphatic and aromatic hydrocarbon solvents can be substituted for the aromatic and aliphatic solvents set forth in the Examples 1 to 4.

Thus the aliphatic and aromatic hydrocarbon solvents can be freely substituted by other conventionally well known aliphatic and aromatic hydrocarbon solvents with boiling point ranges selected to achieve desired evaporation rates.

Both the carrier and the concentrate are completely compatible one with the other in any desirable ratio with the liquid developer so that the dye concentration in the liquid developer can be easily adjusted or varied to raise the dye concentration by addition of concentrate or to lower the concentration by addition of carrier to provide optimum development conditions and to maintain the liquid developer at optimum dye concentration. Such additions of concentrate or carrier can be effected without noticeable change in the viscosity of the developer liquid to permit high loading at low viscosity.

As a result, where the dye content is being rapidly depleted by high speed development of latent electrostatic images, the optimum concentration of dyestuff in the liquid developer can be maintained by measured or substantially continuous additions of concentrate. By the same token, loss of diluent by reason of slow or a low rate of development can be compensated without undesirable increase in dye concentration by measured or substantially continuous additions of carrier.

It will be understood that changes may be made in the details of formulation and methods of application without departing from the spirit of the invention, especially as defined in the following claims.

I claim:

1. A liquid developer composition for latent electrostatic images consisting essentially of a liquid solvent, a polystyrene resin present in the dissolved state in the liquid solvent in an amount within the range of 0.5 to 5.0 percent by weight of the composition, and a dye or pigment suspended in finely divided form in the polystyrene solution in an amount within the range of 0.05 to 0.6 percent by weight of the composition, and in which the liquid solvent consists essentially of substantial proportions each of an aliphatic petroleum solvent, an aromatic hydrocarbon solvent present in an amount sufficient to maintain the polystyrene in solution and a chlorinated-fluorinated hydrocarbon solvent present in an amount sufficient to raise the flash point of the liquid solvent to above 100° F. and in which the aliphatic petroleum solvent and aromatic hydrocarbon solvent have boiling points which do not exceed about 349° F. and in which the liquid solvent has a flash point above 100° F.

2. A liquid developer composition as claimed in claim 1 in which the polystyrene resin is present in an amount within the range of 1 to 3 percent by weight.

3. A liquid developer as claimed in claim 1 in which the polystyrene resin is present in an amount within the range of 1 to 3 percent by weight and in which the dye is present in an amount within the range of 0.2 to 0.5 percent by weight.

4. A liquid developer as claimed in claim 1 in which the dye is insoluble in the solvent system and which is reduced to an average particle size of less than 5 microns.

References Cited by the Examiner

UNITED STATES PATENTS

2,899,335	8/1959	Straughan	252—62.1
3,058,914	10/1962	Metcalfe et al.	252—62.1
3,079,342	2/1963	Insalaco	252—62.1
3,135,695	6/1964	York	252—62.1

SAMUEL H. BLECH, *Primary Examiner.*

JULIUS GREENWALD, *Examiner.*

J. D. WELSH, *Assistant Examiner.*