

- [54] **DEVELOPING LIQUID FOR ELECTROSTATIC IMAGES**
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- [51] Int. Cl. G03g 9/04
- [58] Field of Search 252/62.1

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[57] **ABSTRACT**

A liquid developer for use in an electrostatic copying process in which the developer is made up of an organosol part comprising a tacky component and a minor amount of a release agent, both of which have electrophoretic properties and both of which are substantially insoluble in a light hydrocarbon liquid having a kauributanol number less than 30, the organosol part being suspended in such light hydrocarbon liquid together with a pigment part.

15 Claims, No Drawings

DEVELOPING LIQUID FOR ELECTROSTATIC IMAGES

BACKGROUND OF THE INVENTION

In the electrostatic photographic processes of the prior art two forms of developers have been used. One process, known as the Xerox method, uses a dry powder which is formed of particles of synthetic resins which can be fused by heat. The resins are attracted to the latent electrostatic image and then fixed by melting the synthetic resins and then permitting the melted resins to cool. The other method of developing latent electrostatic images is by use of a developing liquid. This comprises the carrier liquid through which particles having electrophoretic properties have been dispersed. These particles, which are pigmented, are attracted to the electrostatic charge and thus make the latent electrostatic image visible. The Xerox method is one in which there is a transfer of the particles to a charged paper surface on which the thermoplastic resin particles in the form of the image are fused. In the processes using a liquid developer the electrostatic charge is deposited on a paper coated with a photoconductor such as zinc oxide, and there is no transfer of the image from the zinc oxide photoconductive surface to another surface. In another system disclosed in Metcalfe et al. U.S. Pat. No. 3,078,231 pigment particles are enrobed in a control agent for the purposes of giving the particles the required charge for deposition on the photoconductor surface carrying the latent image. The control agent, which is insoluble in the developer carrier liquid, also is of a non-tacky nature.

In our copending application Ser. No. 155,108 filed June 21, 1971, we disclose and teach a Method of Contact Transfer of Developed Electrostatic Images using a liquid toner for developing liquid. The developing liquid comprises essentially a carrier fluid in which a tacky toner material is dispersed. The tacky or sticky toner material can be transferred from the photoconductive surface to plain paper provided the tacky toner has a greater affinity for the paper surface than for the photoconductive surface. In our copending application we have taught that the affinity for the photoconductive surface can be decreased by coating it with a silicone or a urethane film so that the image developed with the tacky toner will have greater affinity for the paper than for the photoconductive surface. The coating of a photoconductive surface tends to reduce its efficiency. That is, the presence of the coating or film between the photoconductive surface and the latent electrostatic image itself results in a slight loss of definition. We have succeeded in improving the developing liquids disclosed in our copending application so that the developed electrostatic images will transfer substantially completely and rapidly to plain paper leaving little or none of the tacky toner behind on the photoconductive surface.

SUMMARY OF THE INVENTION

One object of our invention is to provide an improved developing fluid for electrostatic images which facilitates the contact transfer of such developed images from the surface of the photoconductor to sheet material such as plain paper.

Another object of our invention is to provide for an improved developing liquid which will develop latent electrostatic images formed on organic photoconduc-

tor material such as trinitro-fluorenone and polyvinyl carbazole coated on a conductive base such as Mylar coated with vacuum evaporated aluminum.

A further object of our invention is to provide a developing fluid for latent electrostatic images containing an organosol provided with a release agent resulting in a reduced affinity for the photoconductive surface.

Other and further objects of our invention will appear from the following description.

In general our invention contemplates the provision of an improved liquid developer for use in an electrostatic copying process in which we mill an organosol comprising a tacky component and a minor amount of a release agent together with a pigment component in a light hydrocarbon liquid having a kauri-butanol number of less than 30, and in which the tacky component and the release agent both have electrophoretic properties and both are substantially insoluble in the hydrocarbon liquid.

DESCRIPTION OF THE PREFERRED EMBODIMENT

As is pointed out hereinabove, we suspend a tacky component and a release agent, both of which have electrophoretic properties and both of which are insoluble in a light hydrocarbon liquid having a kauri-butanol number of less than 30 in such liquid. Since the release agent has electrophoretic properties, instead of having to coat the entire photoconductive surface with a release agent the release agent is deposited along with the tacky toner. Presence of the release agent makes it easy for the developed electrostatic image to transfer from the photoconductive surface to the film to which it is being transferred such as a sheet of paper or the like. We have discovered that the amount of the release agent in the liquid toner should not exceed a certain amount by weight of the tacky component, such as high molecular weight resin on the organosol employed in the liquid toner composition. For example if the organosol comprises 37.5 grams of "Pliolite VTAC" not more than about 15 grams of a silicone resin should be dispersed through the developing fluid. "Pliolite" VTAC is the registered trademark of Goodyear Tire & Rubber Co., Akron, Ohio, for a vinyl-toluene, acrylate copolymer resin having a specific gravity of 1.026 and a softening point of $50 \pm 3^\circ\text{C}$. If too much release agent is used there is a loss of cohesion and the air knife shown in our copending application will tend to blow the developed electrostatic image from the surface of the uncoated organic photoconductor. In determining the suitability of the release agent, materials are chosen which are insoluble in the suspension fluids such as "Isopar G" or "Isopar H." "Isopar G" is an isomerized paraffinic hydrocarbon having a specific gravity of 0.75 at 60°F manufactured and sold by the Standard Oil Company of New Jersey. It has a kauri-butanol number of 27. "Isopar H" is a similar substance. The proposed release agent is then dispersed through the suspension fluid. A latent electrostatic image is formed and exposed to the suspension of the release agent in the carrier liquid. If the release agent deposits and forms a faintly visible image composed of particles of the release agent then that release agent may be used. We have tested the following materials:

Polyethylene wax dispersed in Isopar G;

A solution of polyvinyl stearate in Solvesso 100 dispersed through Isopar G;

Polyvinyl chloride resin dispersed through Isopar G;

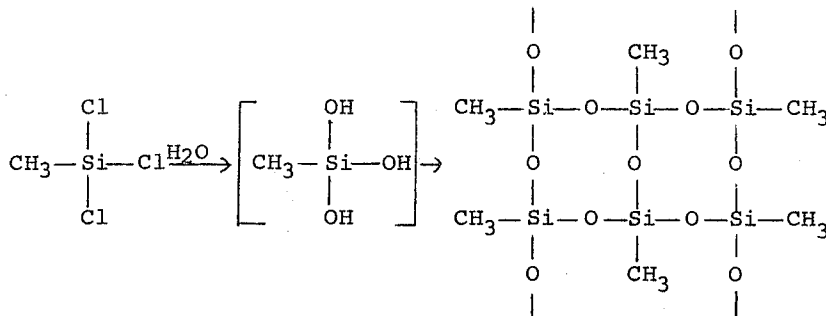
Silicon resin dispersed in Isopar G;

Reactive silicon fluid dispersed through Isopar G; and

Metallic soaps, such as aluminum oleate and aluminum palmitate.

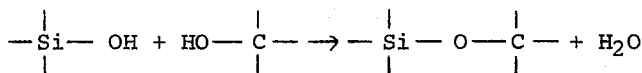
These release agents were deposited on organic photo-

by their three-dimensional branched chain structure. They are normally prepared from a blend of chlorosilanes, containing a mixture of di- and tri-functional monomers which are subsequently hydrolyzed. Mixtures of di-alkyl and/or di-aryl dichlorosilanes and alkyl or aryl trichlorosilanes are normally used. The following reaction takes place in an organic solvent such as xylene or toluene:



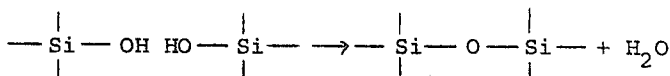
conductors which were negatively charged. We also charged an organic photoconductor positively and exposed the latent image areas to developer comprising 5 grams of polyvinyl chloride iso-butyl ether dissolved in 25 grams of "Solvesso 100". "Solvesso 100" is the registered trademark of Humble Oil and Refining Co. for one of a line of aromatic solvents prepared synthetically from petroleum. Specifically, "Solvesso 100" is a

The reaction may be continued, and final curing may be effected by heating with a catalyst such as the octoates or naphthenates. "Rhodorsil 6354" silicone resins are low molecular weight siloxanols with a hydroxyl content of between three and five percent. The hydroxyl groups act as weak acids, and for this reason the intermediate can be viewed as a highly functional polybasic acid. Copolymerization takes place as follows:



solvent comprising 98% aromatics, having a specific gravity of 0.873, a flashpoint of 108°F, an aniline cloud

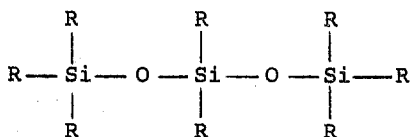
This reaction is to some extent retarded by a side reaction:



point of 14.9 M, a kauri-butanol number of 91 and a distillation range of 159–182°C. This solution was then dispersed in Isopar G. Although the polyvinyl chloride iso-butyl ether may be used as a negative developer conferring its release properties to a negative developing fluid, the positive charge adhesive toners tested using this negative charge polyvinyl chloride iso-butyl ether produced images inferior to release agents exhibiting positive charge properties. The major application for this material is as a release additive for an adhesive toner to be used with a positive charged photoconductor.

EXAMPLE A

In formulating the organosol part, we first mix 250 grams of a silicon resin with 250 grams of Isopar G. The silicon resin is an organo polysiloxane having the general formula of:



in which R is either a methyl or phenyl group and a number of reactive groups which allow the linear polymer and catalyst to form a cross-linked space polymer. These silicon resins may be obtained from Rhone-Poulenc, of Paris, France, and are sold under the trademarks "Rhodorsil 10310 C" and "Rhodorsil 6354". "Rhodorsil 10310 C" are silicone resins characterized

An appropriate catalyst, such as zinc methylate, cobalt methylate, manganese methylate, zinc octoate, cobalt octoate or manganese octoate, in the amount of 25 grams, is mixed with 50 grams of Isopar G. The silicon mix and the catalyst mix are then mixed and stirred together. The silicon resin is mixed with Isopar G in order to lower its viscosity so the catalyst can be stirred into the mix and distributed evenly therethrough. The catalyst is also diluted with Isopar G to improve the mixing of the catalyst with the silicon resin. To form the organosol component of the toner concentrate we add 25 grams of the silicon resin catalyst mixture comprising 10 grams of the actual silicon resin to 37.5 grams of "Pliolite VTAC" and 40 grams of "Solprene 1205" in 100 grams of Solvesso 100. "Pliolite VTAC" is the registered trademark of Goodyear Tire and Rubber Co., Akron, Ohio, for a vinyl-toluene/acrylate copolymer having a specific gravity of 1.026 and a softening point of $50 \pm 3^\circ\text{C}$. "Solprene 1205" is the registered trademark of Phillips Petroleum Company for a block copolymer of butadiene and styrene in the ratio of 75 to 25 made by a solution polymerization process.

Having formed the organosol component, we prepare a suitable pigment component. One pigment part may, for example, be made up of 200 gm microlith black, 25 gm reflex blue, 50 gm Pliolite VTAC dissolved one to one in Solvesso 100, 70 gm Solvesso 100, and 40 gm Solprene 1205 dissolved one to two in mineral turps. This material is milled on a triple roll mill and then dispersed in a colloid mill with 900 mls of Isopar G to form the pigment part.

Having formed both the pigment part and the organosol part, 120 mls of the pigment part are added to the organosol and milled in a colloid mill with 1100 mls of Isopar G to form the toner concentrate.

The silicon resin with the catalyst forms a dispersion which will produce a visible image acting as electrostatic toner per se. In this manner, instead of putting a silicon coating on the photoconductive surface where it would occupy the entire surface, we deposit the silicon along with the tacky toner in a manner so that it acts as a release coating between the image and the photoconductor.

EXAMPLE B

The organosol comprises 37.5 grams of Pliolite VTAC, 30 grams of Solprene 1205, 10 grams of "Pliolite AC3", an amount of the silicon resin-catalyst mix which is described above to provide 10 grams of the silicon resin, and 200 grams of Solvesso E 100. "Pliolite AC3" is the registered trademark of Goodyear Tire and Rubber Company for a styrene/acrylate thickening agent which forms thixotropic gels in both aliphatic and aromatic solvents. It has a specific gravity of 1.026 and a softening point of $50 \pm 3^\circ\text{C}$. The organosol mix containing the silicon is then added to 1100 mls of Isopar G circulating in a cavitation mill together with 120 mls of toner component. The toner component may comprise 100 grams of microlith black, 12.5 grams of reflex blue, 25 grams of Pliolite VTAC, and 35 grams of Solvesso 100. This material is milled on a triple roll mill and the mill mix is stirred into 25 gms of Solprene 1205 previously dissolved in 50 gms of Solvesso 100. This mix is milled in a cavitation mill for about three minutes with 600 mls of Isopar G to form the pigment part. The organosol and 120 mls of toner component then are milled approximately three minutes to form the developing fluid concentrate. This concentrate is further diluted to form a 15% solution by mixing 300 mls of the toner concentrate with 1700 mls of Isopar G. This forms the working toner which is used to form images on uncoated organic photoconductive surfaces. These images will readily release from the organic photoconductor and transfer readily to plain paper.

EXAMPLE C

In this example the organosol part was formed from the same components as outlined in Example B but the amount of silicon-catalyst mix was such as to provide 12.0 gms of the silicon resin. The resultant organosol together with 120 mls of the pigment part used in Example B is milled with 1100 mls of Isopar G.

EXAMPLE D

The organosol was formed in the same manner as Examples B and C but the amount of silicon-catalyst mixture was increased to provide a resultant of 14.0 gms of the silicon resin. The pigment part is the same as that described in our copending application. It is made up of 100.0 gms of microlith black; 12.5 gms reflex blue; 25.0 gms Pliolite VTAC, and 25.0 gms of Solprene 1205 which were milled on a triple roll mill with sufficient toluol to afford the correct milling viscosity. After milling the mixture was diluted with 600 mls of Isopar G to form the toner part. The organosol part was mixed with 240 mls of this pigment part and milled in a cavitation mill with 1100 mls of Isopar G for about three minutes to form the toner concentrate. This concentrate is

further diluted in the ration 1 part by volume of concentrate to 4 parts of Isopar G to form the toner.

EXAMPLE E

We have further discovered that formulation of the toner concentrate is dictated to some degree by the apparatus which is used to remove excess developer from the photoconductor surface following development of a latent image. Our copending application discloses an air nozzle for removing excess developer from the photoconductor surface. The toner described in Example D has provided good results in an air nozzle machine.

It has been proposed that a squeegee roll will provide an effective means for removing excess developer. We have found that use of a toner such as that of Example D in apparatus incorporating a squeegee roller results in somewhat "squashed" images. We have developed the following toner for use in apparatus employing the squeegee roller.

In this example we formulated the organosol part using 37.5 gms of Pliolite VTAC, 10.0 gms of Solprene 1205, 200 gms of Solvesso 100 and an amount of the silicon catalyst mix providing 14.0 gms of the silicon resin in the organosol. We then milled the organosol together with 120 mls of the pigment part described in Example D in 1100 mls of Isopar G to form the toner concentrate.

EXAMPLE F

An organosol was formed in the manner described above from 37.5 gms Pliolite VTAC, 30 gms Solprene 1205, 100 gms Solvesso 100, and an amount of the silicon-catalyst mix to provide 12.0 gms of the silicon resin. This organosol was milled with 240 mls of the toner part of Example A in 1100 gms of Isopar G in a colloid mill for about three minutes to form the toner concentrate.

EXAMPLE G (S91 Adhesive Toner)

We milled the following formulation on a triple roll mill:

20.0 gms Hoechst Special Balck 15
25.0 gms Pliolite VTAC
5.0 gms Solprene 1205
75.0 gms Dibutyl Maleate

This mix was milled on a triple roll mill and then let down on the P.U.C. colloid mill at high speed minimum clearance with 700 mls Isopar G to form the toner component.

We then formed an organosol solution by dissolving the following at room temperature:

37.5 gms Pliolite VTAC
5.0 gms Solprene 1205
100.0 gms Solvesso 100
10.0 gms Rhodorsil Silicone 6354

120 mls of the toner were stirred into the organosol solution to form a toner paste, which paste was milled as described in connection with Example H. This adhesive toner produced a 100% transfer.

EXAMPLE H (T18 Adhesive Toner)

1. Toner Component B923

-Continued

100.0 gms Microlith Black CT.
12.5 gms Reflex Blue AG conc.
25.0 gms Pliolite VTAC.
48.5 gms DiButyl Furmarate.

The VTAC and DiButyl Furmarate are pre-dissolved. The above mix is milled on a triple roll mill.

20 gms Solprene 1205, pre-dissolved, is stirred into the mill mix. This mix is then milled on the P.U.C. colloid mill at high speed and minimum clearance for three minutes together with 600 mls of Isopar G to form the B923 toner.

2. Organosol Solution

37.5 g. Pliolite VTAC
5.0 g. Solprene 1205
100.0 g. Solvesso 100
10.0 g. Rhodorsil silicone 6354

This mix is allowed to dissolve at room temperature.

3. Adhesive Toner T18

120 mls of B923 is stirred into the organosol solution to form the toner paste.

This paste is added to one litre of Isopar G circulating in the Mark 9B toner mill during a five-minute period. Total milling time is ten minutes.

Mill speed is 2800 r.p.m. with smooth rotor and stator. The temperature rise is five degrees C and power consumption 50 to 75 Watts.

4. Test on OCD III Machine

- Squeegee roller drying
- Speed 45 ft/min.
- Paper heat 120-140°F.
- Hammermill electrocopy paper
- Endless belt transfer 8 inches image transfer length

The T18 adhesive toner produces 100% transfer immediately after toner milling.

The toner is then drained from the machine into a half-gallon tin and stored.

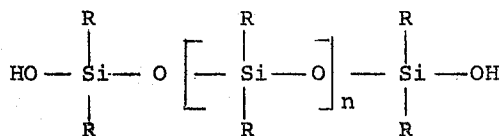
The test was repeated with this toner 7, 14 and 21 days later and showed no change in image quality.

The particle size is just visible to the naked eye and approximately 1/8 inch sediment forms on the bottom of the tin on standing for 7 days. This sediment is redispersed by agitation. This formulation has been repeated with 5 gms of silicone and produces similar results.

This formulation without silicone produces a comparatively inferior transferred image.

EXAMPLE I

The organosol in this instance comprises a reactive silicon fluid having the general formula:



where R = (CH₃) or (C₆H₅).

An amount of the fluid providing 10.0 gms of the silicon resin, 37.5 gms of VTAC Pliolite, and 30.0 gms of Solprene 1205 are mixed to form the organosol. We mill 240 mls of the pigment part of Example A with the organosol and 1100 mls of Isopar G to form the toner concentrate.

EXAMPLE J

Various metallic soaps, used as adhesive toner additives, change the properties of the image deposit. Referring to Example H, above, we replaced the 10 gms of silicone 6354 with 2 gms of aluminum oleate, the other components and method of mixing being the same. The toner with the metallic soap produced an acceptable transfer characterized by a low peel force at the point of separation between the photoconductor and the paper. A small amount of the image is left on the organic photoconductor, but this residue is easily removed and cleaned. If the metallic soap is increased to more than 5 gms, the cohesion of the toner decreases and produces an undesirable image squash. The aluminum oleate also acts as an anti-settling agent and reduces the time required for the toner paste let down in the toner mill.

EXAMPLE K (S71 Adhesive Toner)

We milled the following formulation on a triple roll mill:

37.5 gms Pliolite VTAC
20.0 gms Microlith Black CT
2.5 gms Silicone Rhodorsil 6354
37.5 gms Solvesso 100

A solution of 5 gms Solprene 1205 dissolved in 50 gms Solvesso 100 was stirred into this mill mix. The toner paste was let down in the P.U.C. colloid mill at low speed, minimum clearance with one litre of Isopar G and milled for ten minutes.

The toner particles are not visible to the naked eye and the toner produces 1/8 inch sediment on standing in a half-gallon can during a seven-day period.

This toner does not operate effectively until 24 hours after colloid milling. Let down in the toner mill Mark 9B, instead of the P.U.C. colloid mill, does not reduce this aging period.

While this toner operates, its image quality is not as good as that of toners produced by triple roll milling of the pigment toner component followed by mixing with the organosol solution to form a toner paste, which paste is then let down with Isopar G on the toner mill to form the toner concentrate.

EXAMPLE L

As has been pointed out hereinabove, we have discovered that materials other than silicon resins both are insoluble in Isopar G and are electrophoretic. We have formed toners from such materials. For example, we made up an organosol from 0.2 gm polyethylene wax, 37.5 gms Pliolite VTAC, 10.0 gms Solprene 1205, and 200.0 gms Solvesso 100. The polyethylene wax is heat-melted into Isopar G and then added to the organosol. This organosol was milled with 240 mls of the pigment part of Example D and 1100 mls of Isopar G to form the concentrate.

To summarize, our improved developer includes a release additive. This additive must possess two characteristics. First, it must be substantially insoluble in a light hydrocarbon liquid having a kauri-butanol number of less than 30 but must form a dispersion therein. Secondly, the release agent must be electrophoretic. In determining whether or not a material is suitable for use as a release additive, tests are performed to determine whether or not it possesses the required characteristics. First it is mixed with Isopar G to see if it forms a dispersion rather than a solution therein. If it forms

a dispersion a latent electrostatic image is subjected to the action of the dispersion. If the release agent possesses the required electrophoretic property observation of the surface bearing of the image reveals a more or less readily discernible development of the image.

Having determined the suitability of the release agent an organosol is made up of a high molecular weight tacky resin and of the release agent. The organosol is milled with a suitable pigment part and carrier liquid to form the toner concentrate. The toner concentrate is then diluted by mixing 1 part by volume of concentrate with 4 parts of carrier liquid.

The amount of release agent in the developer must not be too great. If too much release agent is used the air nozzle of the machine may blow the developed image off the photoconductor surface. Further where the machine incorporates a squeegee roll the developer must be so made up as to avoid image "squash". While the percentage of release agent is determined in some degree by the characteristics of the agent itself, it will be seen from the examples given above that the amount of release agent does not exceed 30% by weight of the high molecular weight tacky resin in the organosol.

It will be appreciated that, while we have given specific examples of the tacky resin component of the organosol, other resins such as those described in our pending application may be employed. Moreover, the release agent may be any material meeting the criteria set forth above.

Silicon resins of the type described above are manufactured and sold by Rhone Poulenc, of Paris, France, under the name "Rhodorsil" 10310, 6354 or 20019, 48V500 reactive fluid, and 10072 made up as a primer.

Developers of the type described above were used to develop latent images formed on an endless belt having an organic photoconductor on its surface. The surface was first charged to a surface potential of 700 volts, exposed to an image and subjected to the developer. The developed image was then readily transferred to a material such as paper at a temperature of 100° to 140°F.

It will be seen that we have accomplished the objects of our invention. We have provided an improved developing liquid for electrostatic images which facilitates the contact transfer of developed images from a photoconductive surface to sheet material such as paper. Our developer is especially adapted for use with organic photoconductors. We have provided a developing liquid containing an organosol having a release agent which reduces the affinity of the developer for the photoconductive surface.

It will be understood that certain features and sub-combinations are of utility and may be employed without reference to other features and sub-combinations. This is contemplated by and is within the scope of our claims. It is further obvious that various changes may be made in details within the scope of our claims without departing from the spirit of our invention. It is, therefore, to be understood that our invention is not to be limited to the specific details shown and described.

Having thus described our invention, what we claim is:

1. A developing liquid composition adapted to develop a latent electrostatic image to form a transferable visible image on a photoconductive surface including in combination a light isoparaffinic hydrocarbon carrier liquid means having a kauri-butanol number of less than 30, a tacky organosol means formed by dissolving a high molecular weight resin polymer in an aromatic hydrocarbon solvent, said high molecular weight resin being insoluble in the light isoparaffinic hydrocarbon carrier liquid and having electrophoretic properties, said organosol means being dispersed in said carrier liquid means, release agent means adapted to facilitate the transfer of said visible image from the photoconductor to a supporting surface, said release agent means being insoluble in said carrier liquid means and present in said composition between 6% and 30% by weight of said high molecular weight resin polymer, both said organosol means and said release agent means having such electrophoretic properties that both will codeposit on said latent electrostatic image from said developing liquid, the composition being such that the transfer of the developed image from the photoconductive surface to said supporting surface is facilitated.

2. A developing liquid as in claim 1 in which said release agent is a silicon resin.

3. A developing liquid as in claim 1 in which said release agent comprises a reactive silicon-resin fluid.

4. A developing liquid as in claim 3 in which the amount of silicon resin provided by said fluid is about 15% by weight of the amount of said high molecular weight resin component.

5. A developing liquid as in claim 1 in which said release agent comprises a silicon resin made up as a primer.

6. A developing liquid as in claim 5 in which said primer provides an amount of silicon resin which is at least 6% by weight of the amount of said high molecular weight component.

7. A developing liquid as in claim 1 in which said release agent comprises aluminum palmitate.

8. A developing liquid as in claim 7 in which said aluminum palmitate is present in about 2% by weight of the high molecular weight component.

9. A developing liquid as in claim 1 in which said release agent comprises polyethylene wax.

10. A developing liquid as in claim 9 in which said polyethylene wax comprises about 0.2% by weight of the amount of said high molecular weight resin component.

11. A developing liquid as in claim 1 in which said release agent comprises polyvinyl stearate.

12. A developing liquid as in claim 1 in which said release agent comprises polyvinyl chloride iso-butyl ether.

13. A developer liquid as in claim 1 in which said high molecular weight resin comprises a vinyl-toluene/acrylate copolymer.

14. A developing liquid as in claim 1 in which said release agent comprises a metallic soap.

15. A developing liquid as in claim 1 in which said release agent comprises aluminum oleate.

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