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(54) **Monocomponent developer comprising surface treated toners**

(57) A monocomponent electrostatographic developer is disclosed. The developer contains a negative charging toner wherein the toner particle surface contains particles of titanium dioxide, cerium dioxide and hydrophobic silicon dioxide having a particle size of 0.005 to 0.03 μm .

EP 0 874 286 A1

Description**FIELD OF THE INVENTION**

5 This invention relates to electrostatography, particularly toners for electrostatographic image development methods.

BACKGROUND OF THE INVENTION

10 In electrostatography, an image comprising a pattern of electrostatic potential (also referred to as an electrostatic latent image), is formed on a surface of an electrophotographic element and is then developed into a toner image by contacting the latent image with an electrographic developer. If desired, the latent image can be transferred to another surface following development. The toner image may be transferred to a receiver, to which it is fused, typically by heat and pressure.

15 Electrostatographic developers can be monocomponent or two component developers. Two component developers comprise a mixture of carrier and toner particles. Monocomponent developers comprise nonmagnetic or magnetic toner particles but do not have separate carrier particles. Monocomponent developers can have additional components such as flow agents, and cleaning aids.

20 Cleaning aids in monocomponent developers are present to prevent an accumulation of toner or toner components on photoconductive elements. Silica, titania, alumina, zirconium oxide and cerium oxide among others are disclosed as cleaning aids.

Flow agents in monocomponent developer compositions are present to facilitate toner flow from the replenishment hopper to the developer station and the distribution of toner on the shell of the developer station. Silica, titania, alumina, finely divided polymers, zinc stearate are disclosed as flow agents.

25 U.S. Patent 5,504,559 discloses two types of silica in a two component developer. U.S. Patent 5,066,558 discloses the use of two types of silica added to a monocomponent developer such that a certain fraction is well embedded in the surface of the magnetic toner particles and a lesser fraction is attached but not embedded.

30 One problem, however, particularly with a combination of two oxides is that the triboelectric interaction between the toner and the two oxides makes it difficult to achieve good dispersion and uniform charging. The different oxides often charge in opposite directions. This results in manufacturing difficulties relating to proper dispersion of surface treatment additives. The charging and dispersion difficulties also translate into developer performance problems. These include high image background levels, toner bulk flow problems, and unacceptable film cleaning variability.

SUMMARY OF THE INVENTION

35 The present invention provides a monocomponent electrostatographic developer comprising negatively charging toner particles comprising a polymeric binder, magnetic material and, optionally, a charge-control agent wherein the toner particle surface contains particles of titanium dioxide, cerium dioxide and hydrophobic silicon dioxide having a particle size of 0.005 to 0.03 μm .

40 This developer makes possible excellent image density stability, excellent image background behavior and improved bulk flow. All of these improvements are observable over time.

The present invention also provides a method of preparing a monocomponent electrostatographic developer comprising the steps of:

45 providing negatively charging toner particles comprising a polymeric binder, magnetic material and, optionally, a charge-control agent;
treating the toner surface with a mixture of hydrophobic silicon dioxide having a particle size of 0.005 to 0.03 μm and titanium dioxide; and thereafter
treating the toner surface with cerium dioxide.

50 This method of making the developer results in improved dispersion of surface treatment additives during developer manufacture.

DETAILED DESCRIPTION OF THE INVENTION

55 The toners of the monocomponent developer contain a polymeric binder, charge control agent and a magnetic material. Optionally the toner may include a release agent, colorants and other additives. Electrostatographic toners are commonly made by polymerization of selected monomers followed by mixing with various additives and then grinding

to a desired size range.

The desired polymeric binder for toner application is first produced. During toner manufacturing, the polymeric binder is subjected to melt processing in which the polymer is exposed to moderate to high shearing forces and temperatures in excess of the glass transition temperature of the polymer. The temperature of the polymer melt results, in part, from the frictional forces of the melt processing. The melt processing includes melt blending of toner addenda, including the magnetic material, into the bulk of the polymer.

The polymer may be made using a limited coalescence reaction such as the suspension polymerization procedure disclosed in U.S. Patent No. 4,912,009 to Amering et al.

Useful binder polymers include vinyl polymers, such as homopolymers and copolymers of styrene. Styrene polymers include those containing 40 to 100 percent by weight of styrene, or styrene homologs, and from 0 to 40 percent by weight of one or more lower alkyl acrylates or methacrylates. Also included are fusible styrene-acrylic copolymers that are covalently lightly crosslinked with a divinyl compound such as divinylbenzene. See United States Reissue Patent 31,072.

Copolymers rich in styrene such as styrene butylacrylate and styrene butadiene are also useful as binders as are blends of polymers. In such blends the ratio of styrene butylacrylate to styrene butadiene is 10:1 to 1:10. Ratios of 5:1 to 1:5 and 7:3 are particularly useful. Polymers of styrene butylacrylate and/or butylmethacrylate (30 to 80% styrene) and styrene butadiene (30 to 80% styrene) are also useful polymers.

Styrene polymers include styrene, alpha-methylstyrene, parachlorostyrene, and vinyl toluene; and alkyl acrylates or methacrylates or monocarboxylic acids having a double bond selected from the group consisting of acrylic acid, methyl acrylate, 2-ethylhexyl acrylate, 2-ethylhexyl methacrylate, ethyl acrylate, butyl acrylate, dodecyl acrylate, octyl acrylate, phenyl acrylate, methylacrylic acid, methyl methacrylate, ethyl methacrylate, butyl methacrylate and octyl methacrylate.

Also useful are condensation polymers such as polyesters and copolyesters of aromatic dicarboxylic acids with one or more aliphatic diols, such as polyesters of isophthalic or terephthalic acid with diols such as ethylene glycol, cyclohexane dimethanol and bisphenols.

A useful binder can also be formed from a copolymer of a vinyl aromatic monomer; a second monomer selected from either conjugated diene monomers or acrylate monomers such as alkyl acrylate and alkyl methacrylate.

The magnetic materials included in the toner are generally of the soli type magnetic materials disclosed in the prior art. Examples of useful magnetic materials include mixed oxides of iron, iron silicon alloys, iron aluminum, iron aluminum silicon, nickel iron molybdenum, chromium iron, iron nickel copper, iron cobalt, oxides of iron and magnetite.

The term "charge-control" refers to a propensity of a toner addendum to modify the triboelectric charging properties of the resulting toner. A very wide variety of charge control agents for positive and negative charging toners are available. Suitable charge control agents are disclosed, for example, in U.S. Patent Nos. 3,893,935; 4,079,014; 4,323,634; 4,394,430 and British Patent Nos. 1,501,065; and 1,420,839. Additional charge control agents which are useful are described in United States Patents 4,624,907; 4,814,250; 4,840,864; 4,834,920; 4,683,188 and 4,780,553. Mixtures of charge control agents can also be used. Particular examples of charge control agents include chromium salicylate organo-complex salts, and azo-iron complex-salts, an azo-iron complex-salt, particularly ferrate (1-), bis[4-[(5-chloro-2-hydroxyphenyl)azo]-3-hydroxy-N-phenyl-2-naphthalenecarboxamidato(2-)], ammonium, sodium and hydrogen (Organoiron available from Hodogaya Chemical Company Ltd.)

Release agents are useful additives in some copier configurations. Useful release agents are well known in this art. These include low molecular weight polypropylene, natural waxes, low molecular weight synthetic polymer waxes, commonly accepted release agents, such as stearic acid and salts thereof and others. More specific examples are copolymers of ethylene and propylene having a molecular weight 1000-5000 g/mole, particularly a copolymer of ethylene and propylene having a molecular weight about 1200 g/mole.

An optional additive for the toner is a colorant. In some cases the magnetic component acts as a colorant negating the need for a separate colorant. Suitable dyes and pigments are disclosed, for example, in U.S. Reissue Patent No. 31,072 and in U.S. Patent Nos. 4,160,644; 4,416,965; 4,414,152; and 2,229,513. One particularly useful colorant for toners to be used in black and white electrostatographic copying machines and printers is carbon black. Colorants are generally employed in the range of from about 1 to about 30 weight percent on a total toner powder weight basis, and preferably in the range of about 2 to about 15 weight percent.

In preparing the monocomponent developer the toner is first treated with a mixture of silicon dioxide and titanium dioxide. Thereafter the toner is treated with cerium dioxide. In the first step, based on the weight of the toner, the toner is treated with 0.1 to 2.0 weight percent of a mixture of silicon dioxide and titanium dioxide and then (b) treated with 1.0 to 6.0 weight percent of cerium dioxide, based on the combined weight of the toner and the mixture of silicon dioxide and titanium dioxide. The ratio of silicon dioxide to titanium dioxide in the mixture is 90:10 to 10:90. Within this range ratios of 70:30 to 30:70, and particularly 50:50, are useful.

The hydrophobic fumed silica dioxide has particle sizes of 0.005 to 0.03 μm . The silica dioxide is dichlorodimethylsilane treated, hexamethyldisilazane treated or dimethylsiloxane treated. All three materials are commercially available

from Degussa as Aerosil R 812, R 972 and R 202.

Titanium dioxide having an average particle size in the range of 0.015 to 0.030 μ m is useful.

The cerium oxide added to the developer can be either pure cerium oxide or cerium oxide-rich polishing aids. The cerium oxide particles have a mean volume average particle size of 0.5 to 5 microns. Cerium oxide and cerium oxide-rich polishing aids are commercially available from Transelco Division of the Ferro Corporation and Microabrasives Corporation.

The developer is generally made in several steps. In the first step the polymer, magnetic material, release agent and charge control agent are melt blended in a two roll mill or an extruder. The blend is ground, and classified to achieve a particular toner size distribution. The desired toner has a number average mean diameter between 3 to 15 μ m, or has a volume average mean diameter between 5 and 20 μ m. The toner has a number average mean diameter between 6.5 to 10 μ m and a volume average mean diameter between 8.5 to 12 μ m. To the toner is added the mixture of silica and titanium dioxide particles and cerium oxide particles and mixed according to the procedural steps described above and exemplified in the following examples. Mixing is carried out in a high-speed mixer, such as a Henschel mixer. As stated above the silica dioxide and titanium dioxide are added in a first mixing step and the cerium oxide particles in a second mixing step.

The toner comprises, based on the weight of the toner, 40 to 65 % polymer; 30 to 55 % magnetic material; optionally 1 to 5 % release agent; 0 to 4 % charge control agent and the concentrations of silica dioxide, titanium dioxide and cerium dioxide described above.

The toner can also contain other additives of the type used in previous toners, including magnetic pigments, leveling agents, surfactants, stabilizers, and the like.

The term "particle size" used herein, or the term "size", or "sized" as employed herein in reference to the term "toner particles", means the mean volume average diameter as measured by conventional measuring devices, such as a Coulter Multisizer, sold by Coulter, Inc. of Hialeah, Florida.

Examples:

The following examples will further clarify the monocomponent developer of the invention and the method by which the developer is made. Surface treatment materials used in the examples are listed in Table 1:

Table 1

	Name	BET Surface Area (m ² /g)	Avg. Primary Particle Size (nm)	% SiO ₂	Silane Treatment
Hydrophobic Silicon Dioxide #1	Aerosil R 812 ^a	260 $\pm$ 30	7	> 99.8	Hexamethyldisilazane
Hydrophobic Silicon Dioxide #2	Aerosil R 972 ^a	110 $\pm$ 20	16	> 99.8	Dichlorodimethyl Silane
Hydrophobic Silicon Dioxide #3	Aerosil R 202 ^a	90 $\pm$ 20	14	> 99.8	Silicon Oil
Titanium Dioxide	Aerosil T 805 ^a	55 $\pm$ 10	21	< 2.5	Trimethoxyoctyl Silane
Cerium Dioxide	Ce-Rite 4191 ^b		2.5 x 10 ³		

^aAvailable from Degussa

^bAvailable from Ferro Corporation

Example 1

A toner is prepared according to the formulation recipe below:

Monocomponent Toner Core Production

5	Styrene butylacrylate/butylmethacrylate polymer	36.1 %
	Styrene butadiene copolymer	15.4 %
	Magnetite	45.0 %
10	Organoiron complex	1.5 %
	Ethylene-propylene copolymer wax	2.0 %

15 The above materials were melt blended on a twin screw extruder at about 120 °C average zone temperature to yield a uniform dispersion. The blended material was then jet milled and classified to give a toner product with an average volume particle size distribution of about 10.0 μ .

Monocomponent Developer Production

20 The toner prepared in the above example was blended in a two step operation with a mixture of three inorganic oxides: silicon dioxide (Aerosil R 812) ,titanium dioxide (Aerosil T 805) and cerium dioxide (Ce-Rite 4191). The mixture was effected using a Henschel high intensity mixer. In step 1 of the surface treatment operation a 50:50 mixture of SiO₂ and TiO₂ at a total concentration of 0.42%, based on the weight of the toner, was dry blended for two minutes with toner from above under high shear conditions. In a second step also under high shear conditions, 3.0% by weight of CeO₂ was dry blended with the toner, SiO₂ and TiO₂ from step 1 above, for one additional minute to yield the final developer. The weight of the CeO₂ was based on the weight of the entire mixture.

25 The developer from Example 1 was charged into a commercially available mid volume copier (EK 95 Copier - Eastman Kodak Co.) and run for a minimum of 10,000 copies. Image density, image background and photoconductor film cleanliness were monitored during the testing. Image density was monitored by measuring both the reflection and transmission density of a one inch square solid area density patch using an X-rite Model 310 Densitometer. Background measurements were made by measuring the background particle count of an imaged document using a Bausch and Lomb Omnicon Image Analyzer. Film cleanliness was subjectively evaluated by observing the photoconductor drum surface cleanliness over the course of the testing. Toner flow rates were measured by monitoring the flow time of a 10 gram toner sample through a fixed orifice vibrating flow funnel. Image background, image density, film cleaning and toner flow rate were all judged to be excellent for the monocomponent developer from Example 1. The results from Example 1 and all subsequent examples are summarized in Table 2.

Example 2

40 The toner from Example 1 was surface treated and tested as in Example 1 except the concentration of SiO₂ and TiO₂ was reduced from 0.21% to 0.17 % for each of the two first step additives, giving a total first step treatment level of 0.34%. The CeO₂ was held at 3.0% as in Example 1. As summarized in Table 2 image background, image density, film cleaning and toner flow rate were all judged to be excellent for the monocomponent developer from Example 2.

45 Example 3

The toner from Example 1 was surface treated and tested as in Example 1 except the concentration of SiO₂ and TiO₂ was increased from 0.21% to 0.24 % for each of the two first step additives, giving a total first step treatment level of 0.48%. The CeO₂ was held at 3.0% as in Example 1. Image background, image density, film cleaning and toner flow rate were all judged to be excellent for the monocomponent developer from Example 3.

Example 4

55 The toner from Example 1 was surface treated and tested as in Example 1 except the ratio of SiO₂ and TiO₂ was changed from 50:50 to 70:30 with SiO₂ levels at 0.29% and TiO₂ levels at 0.13%, giving a total first step treatment level of 0.42%. The CeO₂ was held at 3.0% as in Example 1. Image background, image density, film cleaning and toner flow rate were all judged to be excellent for the monocomponent developer from Example 4.

Example 5

The toner from Example 1 was surface treated and tested as in Example 1 except the ratio of SiO₂ and TiO₂ was changed from 50:50 to 30:70 with SiO₂ levels at 0.13% and TiO₂ levels at 0.29%, giving a total first step treatment level of 0.42%. The CeO₂ was held at 3.0% as in Example 1. Image background, image density, film cleaning and toner flow rate were all judged to be excellent for the monocomponent developer from Example 5.

Example 6

The toner from Example 1 was surface treated and tested as in Example 1 with the ratio of SiO₂ and TiO₂ at 50:50 and having a total first step treatment level of 0.42% as in Example 1. The CeO₂ level, however, was changed to 2.0%. Image background, image density and toner flow rate were all judged to be excellent for the monocomponent developer from Example 6. The photoconductor drum, however, started showing slight signs of toner filming on the drum surface and the film cleaning was judged to be only fair.

Comparative Example 1

The toner from Example 1 was surface treated and tested as in Example 1 with the exception that the TiO₂ was omitted from the recipe and the SiO₂ was added at the 0.3% level. The CeO₂ level was kept at 3.0%. While image density was judged to be excellent, the image background level increased and judged to be only fair while the photoconductor drum started showing slight signs of toner filming on the drum surface with the film cleaning being also judged to be only fair. Toner flow was also reduced to fair by the change.

Comparative Example 2

The toner from Example 1 was surface treated and tested as in Example 1 with the exception that the TiO₂ was omitted from the recipe and the SiO₂ was added at the 0.5% level. The CeO₂ level was kept at 3.0%. While image density was judged to be excellent and the photoconductive drum cleaning improved to good, deficiencies were still noted in flow and image background with both being judged only fair.

Comparative Example 3

In Comparative Example 3, the toner from Example 1 was surface treated and tested as in Example 1 and three inorganic oxides were used in the surface treatment but the step 1 treatment consisted of two silicon dioxide materials rather than the SiO₂ and TiO₂ combination in Examples 1 - 6. The step 1 treatment incorporated 0.1% of SiO₂ (Aerosil R 812) plus 0.3% of a different SiO₂ (Aerosil R 972). The CeO₂ level was kept at 3.0%. Excellent density was again seen but the ratings for background, film cleaning and toner flow were only fair.

Comparative Example 4

In Comparative Example 4, the toner from Example 1 was surface treated and tested as in Example 1 and three inorganic oxides were used in the surface treatment but the step 1 treatment again consisted of two silicon dioxide materials (Aerosil R 972 and R 202) rather than the SiO₂ and TiO₂ combination in Examples 1-6. The step 1 treatment in this case incorporated 0.28% of SiO₂ (R 972) and 0.07% of yet another SiO₂ (R 202). The CeO₂ level was kept at 3.0%. Excellent density was again seen but the ratings for background, although improved over Comparative Example 3, were rated good and the film cleaning and toner flow were rated only fair.

Comparative Examples 5 & 6

In Comparative Examples 5 & 6, the toner from Example 1 was surface treated and tested as in Example 1 but only two inorganic oxides were used in the surface treatment. The step 1 treatment in this case incorporated either 0.3% of SiO₂ (R 202) or 0.5% (R 202). The CeO₂ level was kept at 3.0%. No significant improvement was noted over Comparative Example 4.

Comparative Example 7

In Comparative Example 7, the toner from Example 1 was surface treated and tested as in Example 1 but again used a combination of only two inorganic oxides in the surface treatment. In this case the step 1 treatment consisted of

EP 0 874 286 A1

the use of only the titanium dioxide rather than the SiO_2 and TiO_2 combination in Examples 1 - 6. The step 1 treatment incorporated 0.42 % of the titanium dioxide (Aerosil T 805). The second inorganic oxide, CeO_2 , was added during a second mixing step, and was kept at the 3.0% level. Film cleaning was rated good and the ratings for background and toner flow improved to excellent, however image density decreased and was downgraded to fair because of the change in chemistry.

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TABLE 2

	Additives				Total Additive Conc.	Responses		
	SiO ₂ #1	TiO ₂	CeO ₂	SiO ₂ #2	SiO ₂ #3	Background	Density	Film Cleaning
Example 1	0.21	0.21	3.00		3.42	X	X	X
Example 2	0.17	0.17	3.00		3.34	X	X	X
Example 3	0.24	0.24	3.00		3.48	X	X	X
Example 4	0.29	0.13	3.00		3.42	X	X	X
Example 5	0.13	0.29	3.00		3.42	X	X	X
Example 6	0.21	0.21	2.00		2.42	X	X	X
Comparative Example 1	0.3		3.00		3.30	Δ	X	Δ
Comparative Example 2	0.5		3.00		3.50	Δ	X	Δ
Comparative Example 3	0.1		3.00	0.3	3.40	Δ	X	Δ
Comparative Example 4			3.00	0.28	3.35	Δ	X	Δ
Comparative Example 5			3.00		3.30	Δ	X	Δ
Comparative Example 6			3.00		3.50	Δ	X	Δ
Comparative Example 7		0.42	3.50		3.92	X	Δ	X
Composition Key SiO ₂ #1 = Aerosil R 812 SiO ₂ #2 = Aerosil R 972 SiO ₂ #3 = Aerosil R 202 TiO ₂ = Aerosil T 805 CeO ₂ = Ce-Rite 4191						Performance Key X = Excellent G = Good Δ = Fair P = Poor		

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.

Claims

1. A monocomponent electrostatographic developer comprising negatively charging toner particles comprising a polymeric binder, magnetic material and, optionally, a charge-control agent wherein the toner particle surface contains particles of titanium dioxide, cerium dioxide and hydrophobic silicon dioxide having a particle size of 0.005 to 0.03 μm .
2. The developer of claim 1 wherein the toner surface contains, based on the weight of the toner, from 0.2 to 0.6 weight percent of silicon dioxide and titanium dioxide in 90:10 to 10:90 ratio; and from 2.0 to 6.0 weight percent cerium dioxide based on total weight of the mixture of toner, silicon dioxide and titanium dioxide.
3. The developer of claim 2 wherein the ratio of silicon dioxide to titanium dioxide on the toner surface is 90:10 to 10:90 and from 3.0 to 4.0 weight percent cerium oxide.
4. The developer of claim 3 wherein the ratio of silicon dioxide to titanium dioxide on the toner surface is 70:30 to 30:70.
5. The developer of claim 1 or 2 wherein the polymeric binder comprises styrene and an alkyl acrylate and/or methacrylate and the styrene content of the binder at least 60 weight percent.
6. A method of preparing a monocomponent electrostatographic developer comprising the steps of:
 - providing negatively charging toner particles comprising a polymeric binder, magnetic material and, optionally, a charge-control agent;
 - treating the toner surface with a mixture of hydrophobic silicon dioxide having a particle size of 0.005 to 0.03 μm and titanium dioxide; and thereafter
 - treating the toner surface with cerium dioxide.
7. The method of claim 13 wherein the toner surface is first treated with, based on the weight of the toner, (a) 0.1 to 2.0 weight percent of a mixture of silicon dioxide and titanium dioxide and then (b) treated with 1.0 to 6.0 weight percent of cerium dioxide.
8. The method of claim 14 wherein the ratio of silicon dioxide to titanium dioxide in the mixture is 90:10 to 10:90.
9. The method of claim 15 wherein the ratio of silicon dioxide to titanium dioxide in the mixture is 70:30 to 30:70.
10. The method of claim 16 wherein the ratio silicon dioxide to titanium dioxide in the mixture is 50:50.



European Patent
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EUROPEAN SEARCH REPORT

Application Number
EP 98 20 1175

DOCUMENTS CONSIDERED TO BE RELEVANT			
Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int.Cl.6)
A	DATABASE WPI Week 9018 Derwent Publications Ltd., London, GB; AN 90134469 XP002073132 & JP 02 079 053 A (KONICA) , 19 March 1990 * abstract *	1-10	G03G9/097 G03G9/08
A	US 5 512 406 A (TAKEDA KENICHI ET AL) 30 April 1996 * column 11 - column 12; claim 1; example 2 *	1-10	
A	US 4 758 493 A (YOUNG EUGENE F ET AL) 19 July 1988 * column 7; claim 1; example 1 *	1-10	
A	EP 0 223 594 A (CANON KK) 27 May 1987 * page 22; example 1 *	1-10	
A	US 5 612 160 A (INOUE SATOSHI ET AL) 18 March 1997 * column 7 - column 8; example 1 *	1-10	TECHNICAL FIELDS SEARCHED (Int.Cl.6) G03G
The present search report has been drawn up for all claims			
Place of search THE HAGUE		Date of completion of the search 30 July 1998	Examiner Vogt, C
CATEGORY OF CITED DOCUMENTS X : particularly relevant if taken alone Y : particularly relevant if combined with another document of the same category A : technological background O : non-written disclosure P : intermediate document T : theory or principle underlying the invention E : earlier patent document, but published on, or after the filing date D : document cited in the application L : document cited for other reasons & : member of the same patent family, corresponding document			

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