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(71) Applicants:

 Ricoh Company Ltd. Tokyo 143-8555 (JP)

 Ricoh UK Products Limited Telford, Shropshire TF2 9NS (GB) (72) Inventors:

 RAHMAN, Sazidur Telford, Shropshire TF2 9NS (GB)

 SUZUKI, Kazuya Telford, Shropshire TF2 9NS (GB)

 RYMER, Christian Lewis Telford, Shropshire TF2 9NS (GB)

 BALL, Martin Jonathan Telford, Shropshire TF2 9NS (GB)

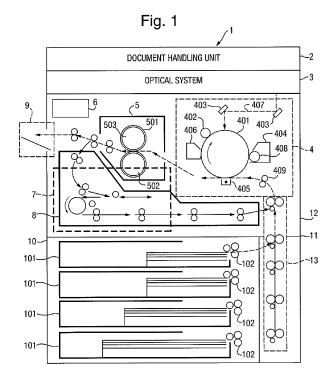
(74) Representative: Leeming, John Gerard
J A Kemp & Co
14 South Square
Gray's Inn

London Greater London WC1R 5JJ (GB)

(54) Method of removing coating material

(57) A method is described for removing coating material from a coated particle for use in an electrophotographic device. Such coated particles may be used as carrier particles in an electrophotographic developer and

typically consist of a core surrounded by a polymeric coating. The method comprises adding the coated particle to a cleaning solution to form a mixture and applying ultrasound to the mixture.



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Description

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[0001] The present invention relates to a method of removing coating material from a coated particle for use in an electrophotographic device, as well as to a cleaning solution suitable for use in such a method, and to particles obtainable by the method. The method may be used, for example, to remove coating material from carrier particles from an electrophotographic developer.

[0002] In electrophotography, developers are used to render visible a latent electrostatic image on a photoreceptor. The developer consists of toner and carrier particles which are mixed in the development unit prior to use. The carrier particles carry the toner onto the electrostatic image on the photoreceptor to form a toner image. The toner image is then transferred to a sheet of paper and fixed to create a resulting copy.

[0003] The carrier particles for use in these two-component (i.e. toner and carrier) dry developers typically consists of a magnetic core coated with a layer of resin. Typically, these resins which may be acrylic resins, polyester resins, or silicone resins, are formulated to provide good mechanical strength and strong adhesion to the magnetic core.

[0004] However, even though these resins are formulated to provide the optimum characteristics of strength and adhesion, the mechanical stresses to which they are subjected, by means of collisions with other particles or with the container walls, can result in the fracture and delamination of the coating. This results in a deterioration of the carrier particle properties over time, and eventually requires replacement of the carrier particles. The used carrier particles are then normally discarded as waste.

[0005] In order to reduce the environmental impact of the manufacture and maintenance of electrophotographic devices, and to reduce the ongoing maintenance costs of such devices, it is desirable to reclaim and reuse, as far as possible, the materials of the carrier particles.

[0006] Processes for removing the coating from the core of a used carrier particle are known. US Patent 6,464,797 describes a method in which carrier material is treated with water under supercritical or subcritical conditions. Preferred conditions are a temperature of at least 300 °C and a pressure of at least 20 MPa. This method therefore requires very high energy inputs to maintain the water in the supercritical or subcritical condition, thereby reducing the environmental benefits of recycling the core material, and increasing the financial cost of operating the process. Furthermore, the need for specialist equipment to handle the supercritical or subcritical conditions means that a high initial investment cost would be required to set up such a system.

[0007] Japanese published patent application number H07-072665 describes a method for regenerating a Mn-Zn ferrite carrier core, with a first heat treatment under air at a temperature up to 500 °C, to remove any adhered toner and coating material. This is then followed by a second heat treatment at 400-600 °C under a reducing atmosphere with saturation magnetization to restore the magnetic properties of the core. The ability of the first heat treatment to remove coating material is highly dependent on the nature of the coating material used, and is particularly suitable only for coatings of styrene-acrylic resin or similar. Additionally, the requirement for high temperatures means that the financial and environmental costs of such a method are higher than desired.

[0008] US Patent 5,965,317 describes a method for removing a coating material comprising a silicone-based resin ,or a resin having a silane coupling agent, from a carrier particle. The method involves separating the carrier particle from any residual toner, and then immersing the carrier particle in aqueous alkali, such as a solution of potassium or sodium hydroxide at 7.5 to 12.5% by weight, at a temperature of 70 to 100 °C. The method is necessarily restricted to the silicon-based coating materials.

[0009] There is therefore a desire for a method of removing coating material from a coated carrier particle which is not limited to particular types of coating material, and which has low financial and environmental costs. The present inventors have surprisingly found that the application of ultrasound to a mixture of carrier particles and a cleaning solution is effective to remove coating material from the carrier particles.

[0010] According to a first aspect of the invention, there is provided a method of removing coating material from a coated particle for use in an electrophotographic device, the method comprising adding the coated particle to a cleaning solution to form a mixture and applying ultrasound to the mixture.

[0011] Methods according to the first aspect of the invention may be suitable for removing a wide variety of coating material types, such as coating materials comprising one or more of polyolefins; polyvinyl or polyvinylidene resins; silicone resins which include straight silicone resin or one modified with acrylic, polyester, epoxy, alkyl, fluorine, urethane, etc.; fluororesins; amino resins; and epoxy resins.

[0012] The use of ultrasound provides, for a relatively low power requirement and low initial investment cost, effective removal of a range of different coating material types.

[0013] It will be understood that 'ultrasound' refers to a cyclic sound pressure wave greater than the upper limit of the human hearing range, which may be defined as 20 kHz. Typically, ultrasound is used at one or more frequencies between 20 and 60 kHz.

[0014] In some embodiments, the coated particle is a carrier particle from an electrophotographic developer.

[0015] In some embodiments, the cleaning solution comprises a polar solvent. For example, the cleaning solution may

comprise a solvent having a dielectric constant of at least 8, at least 10, at least 12, at least 20, at least 30, at least 40 or at least 60. In some further embodiments, the cleaning solution comprises at least one polar solvent selected from water, dimethyl sulfoxide, dimethylformamide, acetonitrile, tetrahydrofuran, C_1 - C_8 alcohols, C_1 - C_6 esters of C_1 - C_8 mono-, di-, and tri-carboxylic acids, particularly C_1 - C_4 diesters of C_4 - C_6 di-carboxylic acids (also known as dibasic esters, or DBE). In some still further embodiments, the cleaning solution comprises at least one polar solvent selected from water, benzyl alcohol, dimethyl succinate, dimethyl adipate and dimethyl glutarate. For example, the polar solvent may comprise a mixture of dimethyl succinate, dimethyl adipate and dimethyl glutarate, optionally together with water. Mixtures of dimethyl succinate, dimethyl adipate and dimethyl glutarate are sold under the trade name FlexiSolvTM DBE® by INVISTA of Wichita, Kansas, USA.

[0016] The polar solvent may form up to 100% by weight of the cleaning solution. In some embodiments, the polar solvent forms at least 50%, at least 60%, at least 70%, at least 75%, at least 80%, at least 85%, at least 90% or at least 95% by weight of the cleaning solution.

[0017] The preferred solvents of the present invention have the advantages of having relatively low environmental impact, and being easily recoverable by distillation.

[0018] In some embodiments, the cleaning solution comprises an acid, such as a weak acid. For example, the cleaning solution may comprise an organic weak acid, such as an organic acid having at least one carboxylic acid group. In some further embodiments, the cleaning solution comprises an organic hydroxycarboxylic acid or di- or tri-carboxylic acid. Examples of hydroxycarboxylic acids and di- and tri-carboxylic acids include oxalic, malonic, succinic, glutaric, adipic, pimelic, glycolic, lactic, malic, citric and tartaric acids. In some still further embodiments, the cleaning solution comprises an organic weak acid selected from malic and citric acids.

[0019] The weak acid may form up to 50% by weight of the cleaning solution. In some embodiments, the weak acid forms no more than 40%, no more than 30%, no more than 25%, no more than 20%, no more than 15%, no more than 10%, or no more than 5% by weight of the cleaning solution. In some embodiments, the weak acid forms at least 0.5%, at least 1%, at least 2%, or at least 5% by weight of the cleaning solution.

[0020] The presence of a weak acid in the cleaning solution has been found to increase the effectiveness of removal of coating material, without negatively affecting the core material.

[0021] In some embodiments, the cleaning solution comprises an emulsifier. Examples of suitable emulsifiers include fluorinated alkyl esters; polyethoxylated sorbitan esters such as polyethoxylated sorbitan monopalmitate, polyethoxylated sorbitan monostearate, polyethoxylated sorbitan tristearate, polyethoxylated sorbitan monooleate; trioleate polysorbates; and mixtures thereof.

[0022] The emulsifier may form up to 20% by weight of the cleaning solution. In some embodiments, the emulsifier forms no more than 15%, no more than 12%, no more than 10%, no more than 7%, or no more than 5% by weight of the cleaning solution. In some embodiments, the emulsifier forms at least 0.5%, at least 1%, at least 2%, or at least 5% by weight of the cleaning solution.

[0023] The presence of emulsifier in the cleaning solution has been found to increase the effectiveness of removal of coating material, without negatively affecting the core material.

[0024] In some embodiments, the cleaning solution comprises:

- a) from 1% to 100% by weight of one or more of benzyl alcohol, dimethyl succinate, dimethyl adipate and dimethyl glutarate;
- b) from 0% to 50% by weight of an organic hydroxycarboxylic acid or di- or tri-carboxylic acid;
- c) from 0% to 20% by weight of emulsifier; and
- d) from 0% to 90% by weight of water.

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- 45 **[0025]** In some further embodiments, the cleaning solution comprises:
 - a) from 80% to 100% by weight of benzyl alcohol, dimethyl succinate, dimethyl adipate, dimethyl glutarate, and mixtures thereof;
 - b) from 0% to 5% by weight of an organic hydroxycarboxylic acid or di- or tri-carboxylic acid;
 - c) from 0% to 5% by weight of emulsifier; and
 - d) from 0% to 10% by weight of water.

[0026] In some embodiments, applying ultrasound to the mixture is carried out a temperature of from 0 to 100 °C. In some further embodiments, applying ultrasound to the mixture is carried out at a temperature of from 20 to 70 °C, or a temperature of from 40 to 60 °C, or a temperature of from 45 to 55 °C. Typically, a temperature of about 50 °C may be used. In general, higher temperatures will increase the rate at which carrier material is removed from the coated particle, but will also increase the risk of damage to the core of the coated particle, and will increase the operating costs of the process.

[0027] In some embodiments, the ultrasound has a frequency of from 20 to 60 kHz. In some further embodiments, the ultrasound has a frequency of from 30 to 50 kHz, or from 35 to 45 kHz, or from 40 to 45 kHz.

[0028] In some embodiments, the ultrasound has a power of at least 50 W. In some further embodiments, the ultrasound has a power of at least 100 W, at least 200 W, at least 500 W, at least 1 kW or at least 2 kW.

[0029] In some embodiments, the method removes at least 50% by weight of the coating. In some further embodiments, the method removes at least 60%, at least 70%, at least 75%, at least 80%, at least 85% or at least 90% of the coating. Typically, such removal is achieved within 5 hours. In some embodiments, the coating removal is achieved within 1 hour, within 40 mins, within 30 mins, within 20 mins, within 15 mins or within 10 mins.

[0030] According to second aspect of the invention, there is provided a carrier particle core obtainable according to a method of the first aspect. Thus, for example, the use of ultrasound may produce a characteristic appearance in the surface of the cleaned particle core.

[0031] According to a third aspect of the invention, there is provided a cleaning solution for use to remove coating material from a carrier particle, the cleaning solution comprising:

- a) from 80% to 90% by weight of one or more of benzyl alcohol, dimethyl succinate, dimethyl adipate and dimethyl glutarate;
- b) from 0% to 5% by weight of an organic hydroxycarboxylic acid or di- or tri-carboxylic acid;
- c) from 0% to 5% by weight of emulsifier; and
- d) from 0% to 10% by weight of water.

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[0032] Specific embodiments of the method according to the first aspect of the invention, using specific embodiments of a cleaning solution according to the third aspect of the invention, are described below by way of Example, with reference to the accompanying Figures, in which:

Figure 1 shows a schematic representation of an electrophotographic image forming apparatus.

[0033] Referring to Figure 1, the image forming apparatus 1 is tall so that a user can dispose it on the floor and use it comfortably. The image forming apparatus 1 is provided with the following: a document handling unit 2 for transporting documents; an optical system 3 for applying light to the document transported by the document handling unit 2 and reading images; an image forming unit 4 for transferring the image read by the optical system 3 to a recording member such as paper; a fixing unit 5 for fixing the transferred image to the recording member; a main power supply 6 for supplying electric power to the entire apparatus; a secondary power supply 7 charged by the main power supply 6 for supplying electric power to the fixing unit 5; a reversing unit 8 for reversing the front and back of the recording member when an image is transferred and fixed on the front and the back of the recording member; a discharging unit 9 for discharging the recording member on which the image has been fixed to an exterior; a paper feed unit 10 for storing recording members and feeding the recording members to the image forming unit 4; a partition plate 11 for partitioning internal space, a chassis 12 that covers the above components; and a transportation path 13 for transporting the recording member output by the paper feed unit 10 or the reversing unit 8 to the image forming unit 4. The document handling unit 2, the optical system 3, the image forming unit 4, the fixing unit 5, the main power supply 6, the secondary power supply 7, the reversing unit 8, and the discharging unit 9 are disposed over the partitioning plate 11, and the paper feed unit 10 is provided under the partitioning plate 11. The partitioning plate 11 is parallel to the bottom face of the chassis 12. The partitioning plate 11 is made of resin, for example, as a part of the chassis 12.

[0034] The image forming unit 4 is provided with a photosensitive body 401 which is a drum-shaped rotative body. A charging roller 402, a mirror 403, a developing unit 404, a transferring unit 405, and a cleaning unit 406 are disposed around the outer periphery of the photosensitive body 401. When no light is applied to the photosensitive body 401, the charging roller 402 charges the surface of the photosensitive body 401 uniformly. The mirror 403 scans the charged photosensitive body 401 with exposure light 407, and forms an electrostatic latent image on the surface of the photosensitive body 401, the electrostatic latent image corresponding to an image to be formed on the recording member. The developing unit 404 forms a toner image by making the electrostatic latent image formed on the photosensitive body 401 visible using a developing roller 408. The transferring unit 405 transfers the toner image to a recording member such as paper by electric field. The cleaning unit 406 removes remaining toner that the transferring unit 405 left on the surface of the photosensitive body 401. The image forming unit 4 is provided with a resist roller 409. The resist roller 409 adjusts timing in which the recording member is transported so that the position of the toner image formed on the surface of the photosensitive body 401 and the position of the recording member match at the transferring unit 405. The recording member on which the toner image has been transferred by the transferring unit 405 is transported to the fixing unit 5.

[0035] Within the developing unit 404 is contained a developer, which comprises a finely-powdered toner and carrier particles, each consisting of a ferromagnetic core coated with a polymer resin. The carrier particles assist in the transfer

of toner to the developing roller 408, forming a uniform coating of toner on the developing roller. The toner then adheres to the electrostatic latent image on the photosensitive body 401 to form the toner image. The carrier particles remain within the developer unit 404.

[0036] The fixing unit 5 receives the recording member on which the toner image has been transferred, from the image forming unit 4. The fixing unit 5 fixes the toner image on the recording member, and then, discharges the recording member to the discharging unit 9. The fixing unit 5 is provided with a fuser roller 501 for heating the recording member and a pressure roller 502 for applying pressure to the recording member.

[0037] The paper feed unit 10 is provided with paper feed trays 101 and paper feed rollers 102. The paper feed trays 101 store recording members of multiple sizes by the size. The paper feed rollers 102 take out the recording member stored in the paper feed tray 101 and send it to the transportation path 13. The recording member is transported through the transportation path 13 to the resist roller 409.

Example 1

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[0038] After 300,000 copies, the developer from a digital copy apparatus was collected and cleaned of toner, treated and examined as follows:

The toner was separated from the carrier particles by rinsing with a dibasic ester (DBE) mixture, containing dimethyl succinate, dimethyl adipate and dimethyl glutarate and commercially available as FlexiSolv™ DBE® from INVISTA of Wichita, Kansas, USA. The amount of residual toner particles on the surface after rinsing was minimal, as observed by visual inspection under a microscope. The DBE used in the rinsing process was recovered via vacuum distillation. The carrier particles were then added to a stirred container with a cleaning solution having the following composition by weight:

Dibasic ester	80%
Malic acid	5%
Polyoxyethylene 20 sorbitan monooleate emulsifier (available commercially as TEGO® SMO 80 V from Evonik Industries AG of Essen, Germany)	5%
Water	10%

[0039] The stirred container was placed in an ultrasonic water bath set to a temperature of 50 °C, and subjected to ultrasonic treatment at a frequency of 42 kHz with a power of 100 W. After 5 hours, the cores were separated from the detached resin by decanting off the resin solvent mixture, and washed with water. The wash process was repeated a further two times. The washed cores were subsequently dried in a drying oven at 100 °C for 1 hour. An evaluation of the magnetic core was then completed using a scanning electron microscope and an XRF spectrometer (Philips Venus 200 Minilab). Results indicated that almost all of the silicone resin had been removed from the surface of the cores. The measured Si content (deriving from the silicone-based coating material) was 7.50% (by mass), by comparison with samples of virgin core particles and unused coated particles.

[0040] The magnetic and electrical characteristics of the cores were also measured using a compact full-automatic vibration sample magnetometer VSM-C7-10A (available from Toei Industry Co., Ltd of Tokyo, Japan). In particular, the saturation magnetization, remnant magnetization, and coercive force of the cores were found to be comparable to those of the virgin core material.

Example 2

[0041] The carrier particles in Example 2 were collected and cleaned of toner in the same manner as for Example 1, the developer having completed 300,000 copies.

[0042] The carrier particles were then added to a stirred container with a cleaning solution having the following composition by weight:

Dibasic ester	100%
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[0043] The stirred container was placed in an ultrasonic water bath set to a temperature of 50 °C, and subjected to ultrasonic treatment at a frequency of 42 kHz with a power of 100 W. Process time, wash procedure and evaluation were the same as for Example 1. The measured Si content was 13.92%.

Example 3

[0044] The carrier particles in Example 3 were collected and cleaned of toner in the same manner as for Example 1, the developer having completed 300,000 copies.

[0045] The carrier particles were then added to a stirred container with a cleaning solution having the following composition by weight:

Dibasic ester	80%
Citric acid	5%
Polyoxyethylene 20 sorbitan monooleate emulsifier	5%
Water	10%

[0046] The stirred container was placed in an ultrasonic water bath set to a temperature of 50 °C, and subjected to ultrasonic treatment at a frequency of 42 kHz with a power of 100 W. Process time, wash procedure and evaluation were the same as for Example 1. The measured Si content was 7.80%.

Comparative example 1

[0047] The carrier particles in Comparative example 1 were collected and cleaned of toner in the same manner as for Example 1, the developer having completed 300,000 copies.

[0048] The carrier particles were then added to a stirred container with a cleaning solution having the following composition by weight:

Dibasic ester	80%
Malic acid	5%
Polyoxyethylene 20 sorbitan monooleate emulsifier	5%
Water	10%

[0049] The stirred container was placed in an ultrasonic water bath set to a temperature of 50 °C, but no ultrasonic treatment was applied. Process time, wash procedure and evaluation were the same as for Example 1. The measured Si content was 85.07%.

Example 4

[0050] The carrier particles in Example 4 were collected and cleaned of toner in the same manner as for Example 1, the developer having completed 300,000 copies.

[0051] The carrier particles were then added to a stirred container with a cleaning solution having the following composition by weight:

Benzyl alcohol	80%
Malic acid	5%
Polyoxyethylene 20 sorbitan monooleate emulsifier	5%
Water	10%

[0052] The stirred container was placed in an ultrasonic water bath set to a temperature of 50 °C, and subjected to ultrasonic treatment at a frequency of 42 kHz with a power of 100 W. Process time, wash procedure and evaluation were the same as for Example 1. The measured Si content was 9.86%.

55 Example 5

[0053] The carrier particles in Example 5 were collected and cleaned of toner in the same manner as for Example 1,

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the developer having completed 300,000 copies.

[0054] The carrier particles were then added to a stirred container with a cleaning solution having the following composition by weight:

Dibasic ester	95%
Polyoxyethylene 20 sorbitan monooleate emulsifier	5%

[0055] The stirred container was placed in an ultrasonic water bath set to a temperature of 50 °C, and subjected to ultrasonic treatment at a frequency of 42 kHz with a power of 100 W. Process time, wash procedure and evaluation were the same as for Example 1. The measured Si content was 8.46%.

Example 6

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[0056] The carrier particles in Example 6 were collected and cleaned of toner in the same manner as for Example 1, the developer having completed 300,000 copies.

[0057] The carrier particles were then added to a stirred container with a cleaning solution having the following composition by weight:

Dibasic ester	80%
Malic acid	5%
Polyoxyethylene 20 sorbitan monooleate emulsifier	5%
Water	10%

[0058] The stirred container was placed in an ultrasonic water bath set to a temperature of 40 °C, and subjected to ultrasonic treatment at a frequency of 42 kHz with a power of 100 W.

[0059] Process time, wash procedure and evaluation were the same as for Example 1. The measured Si content was 10.49%.

Example 7

[0060] Cleaning was carried out on carrier particles having an iron oxide core coated with a polymer comprising silicone resin/acrylic resin, with aluminium oxide and titanium dioxide as resin additives.

[0061] The carrier particles in Example 7 were collected and cleaned of toner in the same manner as for Example 1, the developer having completed 300,000 copies.

[0062] The carrier particles were then mixed with a cleaning solution having the following composition by weight:

Dibasic ester	100%

[0063] The mixture was circulated past a sonotrode in a flow through cell system, and subjected to ultrasonic treatment at a frequency of 20 kHz with a power of 1 kW, using a UIP1000hd homogenizer available from Hielscher Ultrasonics GmbH of Teltow Germany. Samples were taken at 5-minute intervals up to 20 minutes, and after 5 hours, and measurements made using X-ray microanalysis as before. The results are shown below:

Time (mins)	%Si	%Al
5	35.53	
10	19.61	
15	12.93	
20	12	10.2

Example 8

[0064] Cleaning was carried out on carrier particles having an iron oxide core coated with a polymer comprising silicone resin/acrylic resin, with aluminium oxide and titanium dioxide as resin additives.

[0065] The carrier particles of Example 8 were collected from a digital copy apparatus and cleaned of toner in the same manner as for Example 1, the developer having completed 300,000 copies.

[0066] The carrier particles were then mixed with a cleaning solution having the following composition by weight:

Dibasic ester	100%
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[0067] The mixture was circulated past a sonotrode in a flow through cell system, and subjected to ultrasonic treatment at a frequency of 20 kHz with a power of 2 kW, using a DG-2000 generator and high power processing system with a titanium sonotrode, available from Telsonic UK Ltd of Dorset, UK. Samples were taken at 2-minute intervals up to 8 minutes, and measurements made using X-ray microanalysis as before. The results are shown below:

Time (mins)	%Si	%Al
2	22.81	16.68
4	13.66	8.90
6	11.52	7.09
8	9.07	5.44

Example 9

[0068] Cleaning was carried out on carrier particles having core comprising iron oxide (50-90%) and manganese oxide (14-45%) coated with a polymer comprising silicone resin/acrylic resin/coupling agent, with aluminium oxide as a resin additive.

[0069] The carrier particles of Example 9 were collected from a digital copy apparatus and cleaned of toner in the same manner as for Example 1, the developer having completed 300,000 copies.

[0070] The carrier particles were then mixed with a cleaning solution having the following composition by weight:

Dibasic ester	100%
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[0071] The mixture was circulated past a sonotrode in a flow through cell system, and subjected to ultrasonic treatment at a frequency of 20 kHz with a power of 2 kW, using a DG-2000 generator and high power processing system with a titanium sonotrode, available from Telsonic UK Ltd of Dorset, UK. Samples were taken at 2-minute intervals up to 8 minutes, and measurements made using X-ray microanalysis as before. The results are shown below:

Time (mins)	%Si	%Al
2	57.38	51.42
4	38.61	29.90
6	30.95	19.03
8	21.53	13.92

Claims

- 1. A method of removing coating material from a coated particle for use in an electrophotographic device, the method comprising adding the coated particle to a cleaning solution to form a mixture and applying ultrasound to the mixture.
- The method according to claim 1, wherein the coated particle is a carrier particle from an electrophotographic developer.

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- 3. The method according to claim 1 or claim 2, wherein the cleaning solution comprises a polar solvent selected from water, dimethyl sulfoxide, dimethylformamide, acetonitrile, tetrahydrofuran, C₁-C₈ alcohols, C₁-C₆ esters of C₁-C₈ mono-, di-, and tri-carboxylic acids, and mixtures thereof.
- 5 4. The method according to claim 3, wherein the polar solvent forms at least 80% of the cleaning solution.
 - **5.** The method according to any one of claims 1 to 4, wherein the cleaning solution comprises an acid selected from oxalic, malonic, succinic, glutaric, adipic, pimelic, glycolic, lactic, malic, citric and tartaric acids, and mixtures thereof.
- 10 **6.** The method according to claim 5, wherein the acid forms no more than 5% of the cleaning solution.
 - 7. The method according to any one of claims 1 to 6, wherein the cleaning solution comprises an emulsifier selected from fluorinated alkyl esters; polyethoxylated sorbitan monolaureate, polyethoxylated sorbitan monopalmitate, polyethoxylated sorbitan monostearate, polyethoxylated sorbitan tristearate, polyethoxylated sorbitan monooleate; trioleate polysorbates; and mixtures thereof.
 - 8. The method according to claim 7, wherein the emulsifier forms no more than 5% of the cleaning solution.
 - 9. The method according to any one of claims 1 to 8, wherein the ultrasound has a frequency of from 20 to 60 kHz.
 - 10. The method according to any one of claims 1 to 9, wherein the ultrasound has a power of at least 50 W.
 - 11. A carrier particle core obtainable by a method according to any one of claims 1 to 10.
- 25 **12.** A cleaning solution for use in a method according to any one of claims 1 to 10, comprising:
 - a) from 80% to 90% by weight of one or more of benzyl alcohol, dimethyl succinate, dimethyl adipate and dimethyl glutarate;
 - b) from 0% to 5% by weight of an organic hydroxycarboxylic acid or di- or tri-carboxylic acid;
 - c) from 0% to 5% by weight of emulsifier; and
 - d) from 0% to 10% by weight of water.

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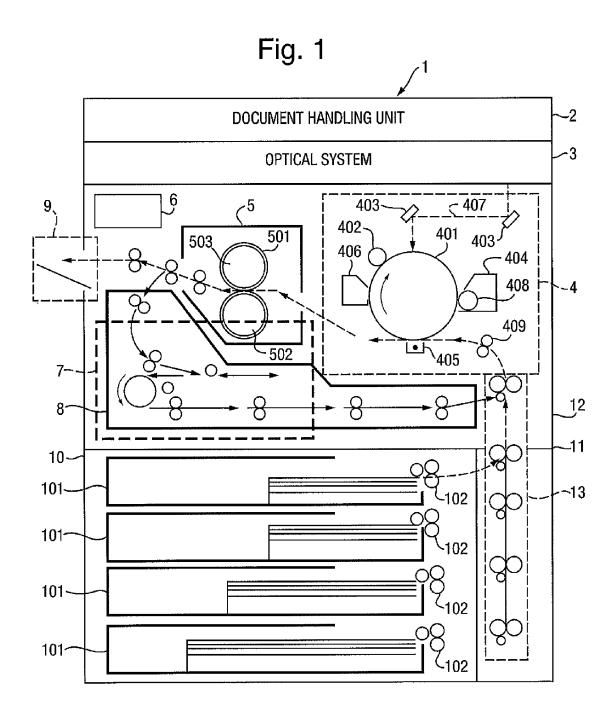
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EUROPEAN SEARCH REPORT

Application Number EP 12 19 8691

	DOCUMENTS CONSID	EKED TO BE KELE	VANI			
Category	Citation of document with in of relevant pass	ndication, where appropriate, ages		Relevant o claim	CLASSIFICATION OF THE APPLICATION (IPC)	
Х	US 2010/273103 A1 (AL) 28 October 2016 * paragraph [0147]	(2010-10-28)		2,9-11	INV. G03G9/113 G03G9/10	
Х	JP 2005 300676 A (F 27 October 2005 (20 * abstract * * claims 1-5 *	RICOH KK) 105-10-27)	1-	-11		
Х	JP 2007 224165 A (k 6 September 2007 (2 * abstract *		12	2		
Х	JP 2002 229275 A (F		11	L		
Α	14 August 2002 (200 * paragraph [0006]	2-08-14) - paragraph [0008] *		-10		
Χ	JP 2001 022130 A (F		11	L		
Α	26 January 2001 (20 * paragraph [0016]		1-	-10		
					TECHNICAL FIELDS SEARCHED (IPC)	
				•	G03G	
	The present search report has	heen drawn up for all claims				
	Place of search	Date of completion of t	he search	 T	Examiner	
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ANNEX TO THE EUROPEAN SEARCH REPORT ON EUROPEAN PATENT APPLICATION NO.

EP 12 19 8691

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This annex lists the patent family members relating to the patent documents cited in the above-mentioned European search report. The members are as contained in the European Patent Office EDP file on The European Patent Office is in no way liable for these particulars which are merely given for the purpose of information.

Patent family

Publication

16-05-2013

Publication

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Patent document

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FP 2252917 A1 24-1:	cited in search report		date		member(s)		date
JP 2007224165 A 06-09-2007 JP 5152816 B2 27-02 JP 2007224165 A 06-09 JP 2007224165 A 06-09 JP 2001022130 A 26-01-2001 NONE	US 2010273103	A1	28-10-2010	EP JP JP KR US	2252917 5106308 2009237525 20100122504 2010273103	A1 B2 A A A1	26-01-201 24-11-201 26-12-201 15-10-200 22-11-201 28-10-201 11-09-200
JP 2007224165 A 06-09 JP 2002229275 A 14-08-2002 NONE JP 2001022130 A 26-01-2001 NONE	JP 2005300676	Α	27-10-2005	NONE			
JP 2001022130 A 26-01-2001 NONE	JP 2007224165	Α	06-09-2007				27-02-201 06-09-200
	JP 2002229275	Α	14-08-2002	NONE			
	JP 2001022130	Α	26-01-2001	NONE			

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REFERENCES CITED IN THE DESCRIPTION

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Patent documents cited in the description

- US 6464797 B [0006]
- JP H07072665 B [0007]

US 5965317 A [0008]