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(54) Title: LIQUID ELECTROPHOTOGRAPHIC INK INCLUDING A TAGGANT

(57) Abstract: According to an example, a liquid electrophotographic ink includes a taggant and a carrier liquid.



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LIQUID ELECTROPHOTOGRAPHIC INK INCLUDING A TAGGANT

Background

[0001] Ink compositions containing charged particles are used in a wide variety of applications such as toners in electrophotography printing, pigmented ink, electrophoretic displays as well as many other applications. Liquid electrophotographic printing is a specific type of electrophotographic printing where a liquid ink is employed in the process rather than a powder toner.

DETAILED DESCRIPTION

[0002] For simplicity and illustrative purposes, the present disclosure is described by referring mainly to examples thereof. In the following description, numerous specific details are set forth in order to provide a thorough understanding of the present disclosure. It will be readily apparent however, that the present disclosure may be practiced without limitation to these specific details. In other instances, some methods and structures have not been described in detail so as not to unnecessarily obscure the present disclosure. As used herein, the terms “a” and “an” are intended to denote at least one of a particular element, the term “includes” means includes but not limited to, the term “including” means including but not limited to, and the term “based on” means based at least in part on.

[0003] A liquid electrophotographic ink may be formed by mixing a resin and a carrier liquid to form a paste. The paste may be ground with a taggant to form a slurry. A charge director may be added to form the liquid electrophotographic ink. Through implementation of the disclosed method, the presence of the taggant may be detected within the resultant liquid electrophotographic ink. The presence of the taggant may provide confirmation that the article printed with an image formed from the liquid electrophotographic ink is a true, original.

[0004] Generally, a carrier liquid may act as a dispersing medium for the other components in the liquid electrophotographic ink. The carrier liquid may have

or be a hydrocarbon, silicone oil, vegetable oil, etc. The carrier liquid may include, but is not limited to, an insulating, non-polar, non-aqueous liquid that may be used as a medium for articles. In an aspect, the carrier liquid may be a low dielectric (< 2 dielectric constant) solvent.

[0005] The carrier liquid may include, but is not limited to, hydrocarbons. The hydrocarbon may include, but is not limited to, an aliphatic hydrocarbon, an isomerized aliphatic hydrocarbon, branched chain aliphatic hydrocarbons, aromatic hydrocarbons, and combinations thereof. Non-limiting examples of a carrier liquid may include aliphatic hydrocarbons, isoparaffinic compounds, paraffinic compounds, dearomatized hydrocarbon compounds, and the like. In particular, the carrier liquid can be chosen from Isopar-G™, Isopar-H™, Isopar-L™, Isopar-M™, Isopar-K™, Isopar-V™, Norpar 12™, Norpar 13™, Norpar 15™, Exxol D40™, Exxol D80™, Exxol D100™, Exxol D130™, and Exxol D140™ (each sold by EXXON CORPORATION); Teclen N-16™, Teclen N-20™, Teclen N-22™, Nisseki Naphthesol L™, Nisseki Naphthesol M™, Nisseki Naphthesol H™, #0 Solvent L™, #0 Solvent M™, #0 Solvent H™, Nisseki Isosol 300™, Nisseki Isosol 400™, AF-4™, AF-5™, AF-6™ and AF-7™ (each sold by NIPPON OIL CORPORATION); IP Solvent 1620™ and IP Solvent 2028™ (each sold by IDEMITSU PETROCHEMICAL CO., LTD.); Amsco OMS™ and Amsco 460™ (each sold by AMERICAN MINERAL SPIRITS CORP.); and Electron, Positron, New II, Purogen HF (100% synthetic terpenes) (sold by ECOLINK™).

[0006] The carrier liquid may be present in the liquid electrophotographic ink composition in an amount ranging from about 20% to about 99.5% by weight of the electrophotographic ink composition, and in some examples about 50% to about 90% by weight of the electrophotographic ink composition. In another example, the carrier liquid may be present in an amount ranging from about 60% to about 80% by weight of the electrophotographic ink composition.

[0007] The resin for use in the liquid electrophotographic ink composition may include a polymer. The resin may include, but is not limited to, a thermoplastic polymer. The polymer of the resin may be selected from ethylene acrylic acid

copolymers; methacrylic acid copolymers; ethylene vinyl acetate copolymers; copolymers of ethylene (e.g. from about 80 wt. % to about 99.9 wt. %), and alkyl (e.g. C₁ to C₅) ester of methacrylic or acrylic acid (e.g. from about 0.1 wt. % to about 20 wt. %); copolymers of ethylene (e.g. from about 80 wt. % to about 99.9 wt. %), acrylic or methacrylic acid (e.g. from about 0.1 wt. % to about 20.0 wt. %) and alkyl (e.g. C₁ to C₅) ester of methacrylic or acrylic acid (e.g. from about 0.1 wt. % to about 20 wt. %); polyethylene; polystyrene; isotactic polypropylene (crystalline); ethylene ethyl acrylate; polyesters; polyvinyl toluene; polyamides; styrene/butadiene copolymers; epoxy resins; acrylic resins (e.g. copolymer of acrylic or methacrylic acid and at least one alkyl ester of acrylic or methacrylic acid wherein alkyl is in some examples from about 1 to about 20 carbon atoms, such as methyl methacrylate (e.g. from about 50 wt. % to about 90 wt. %)/methacrylic acid (e.g. from about 0 wt. % to about 20 wt. %)/ethylhexylacrylate (e.g. from about 10 wt. % to about 50 wt. %)); ethylene-acrylate terpolymers: ethylene-acrylic esters-maleic anhydride (MAH) or glycidyl methacrylate (GMA) terpolymers; ethylene-acrylic acid ionomers and combinations thereof.

[0008] The resin may constitute from about 5% to about 90%, in some examples from about 5% to about 80%, by weight of the non-volatile solids of the liquid electrophotographic ink composition, such as from about 5% to about 70%. Additionally, the resin may constitute about 10% to about 60% by weight of the non-volatile solids of the liquid electrophotographic ink composition. Moreover, the resin may constitute about 15% to about 40%, such as from about 20 to about 30% by weight of the non-volatile solids of the liquid electrophotographic ink composition.

[0009] Non-limiting examples of the resin include the Nucrel family of toners (e.g. Nucrel 403™, Nucrel 407™, Nucrel 609HS™, Nucrel 908HS™, Nucrel 1202HC™, Nucrel 30707™, Nucrel 1214™, Nucrel 903™, Nucrel 3990™, Nucrel 910™, Nucrel 925™, Nucrel 699™, Nucrel 599™, Nucrel 960™, Nucrel RX 76™, Nucrel 2806™, Bynell 2002, Bynell 2014, and Bynell 2020 (sold by E. I. du PONT)); the Aclyn family of toners (e.g. Aclyn 201, Aclyn 246, Aclyn 285, and

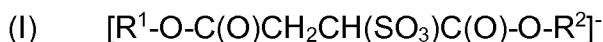
Aclyn 295); the Lotader family of toners (e.g. Lotader 2210, Lotader, 3430, and Lotader 8200 (sold by Arkema)); and AC5120 (an ethylene acrylic acid copolymer from Allied Signal).

[0010] The taggant can be made of any material or composition capable of showing real-time change. The taggant can be a chemical or physical marker added to materials. Some materials or compositions that can be used as taggants include, but are not limited to, nanoparticles, birefringent materials, luminescent dyes, inks doped with magnetic or trace elements. In an example, the taggant may be any material that has at least one optical property such as fluorescence, absorption, polarization, Raman scattering, and nonlinear optical properties. The taggant may emit or absorb a signal in at least one of the following spectral ranges: infrared, visible, and ultraviolet. The taggant may be a fluorescent material which upon irradiation with infrared light may emit or absorb at a wavelength ranging from about 700 nm to about 14 μ m, and for example range from about 760 nm to about 860 nm, for example about 780 nm to about 800 nm, for example from about 800 nm to about 820 nm, for example from about 820 nm to about 840 nm, and as a further example from about 840 nm to about 860 nm. The taggant may emit or absorb visible light at wavelengths in the range of about 400 nm to about 700 nm. The taggant may be a fluorescent material, which upon irradiation with ultraviolet light may emit or absorb a signal at a spectral range of from about 200 nm to about 400 nm, for example from about 260 nm to about 380 nm, and as a further example from about 320 nm to about 340 nm.

[0011] The taggant may be combined with a paste (which may be formed from introducing a carrier liquid to a resin) in a ratio of about 1:10 (taggant:paste), for example from about 1:8, for example from about 1:5, and as a further example from about 1:3. The taggant may be present in the ink composition in a de minimis amount, *i.e.*, any amount so long as the taggant may be detected.

[0012] A charge director imparts a charge to the liquid electrophotographic ink, which is identical to the charge of a photoconductive surface. The electrophotographic ink composition may include a charge director having a

sulfosuccinate salt of the general formula MAn, wherein M is a metal, n is the valence of M, and A is an ion of the general formula (I):



[0013] wherein each of R¹ and R² may be an alkyl group.

[0014] The charge director may be added in order to impart and/or maintain sufficient electrostatic charge on the ink particles.

[0015] The sulfosuccinate salt of the general formula MAn may be an example of a micelle forming salt. The charge director may be substantially free or free of an acid of the general formula HA, where A is as described above. The charge director may include micelles of the sulfosuccinate salt enclosing at least some of the nanoparticles. The charge director may include at least some nanoparticles having a size of 200 nm or less, and/or in some examples 2 nm or more.

[0016] The charge director may further have a simple salt. Simple salts are salts that do not form micelles by themselves, although they may form a core for micelles with a micelle forming salt. The ions constructing the simple salts are all hydrophilic. The simple salt may include a cation selected from the group consisting of Mg, Ca, Ba, NH₄, tert-butyl ammonium, Li⁺, and Al³⁺, or from any sub-group thereof. The simple salt may include an anion selected from the group consisting of SO₄²⁻, PO₄³⁻, NO₃⁻, HPO₄²⁻, CO₃²⁻, acetate, trifluoroacetate (TFA), Cl⁻, BF₄⁻, F⁻, ClO₄⁻, and TiO₃⁴⁻, or from any sub-group thereof. The simple salt may be selected from CaCO₃, Ba₂TiO₃, Al₂(SO₄), Al(NO₃)₃, Ca₃(PO₄)₂, BaSO₄, BaHPO₄, Ba₂(PO₄)₃, CaSO₄, (NH₄)₂CO₃, (NH₄)₂SO₄, NH₄OAc, Tert-butyl ammonium bromide, NH₄NO₃, LiTFA, Al₂(SO₄)₃, LiClO₄ and LiBF₄, or any sub-group thereof. The charge director may further include basic barium petronate (BBP).

[0017] In the formula [R¹-O-C(O)CH₂CH(SO₃⁻)C(O)-O-R²], for example each of R¹ and R² may be independently an aliphatic alkyl group, such as a C₆₋₂₅ alkyl. The aliphatic alkyl group may be linear or branched. The aliphatic alkyl group may

have a linear chain of more than 6 carbon atoms. R^1 and R^2 may be the same or different. In some examples, at least one of R^1 and R^2 is $C_{13}H_{27}$. In some examples, M is Na, K, Cs, Ca, or Ba.

[0018] The charge director may further include one of, some of or all of (i) soya lecithin, (ii) a barium sulfonate salt, such as basic barium petronate (BPP), and (iii) an isopropyl amine sulfonate salt. Basic barium petronate is a barium sulfonate salt of a 21-26 hydrocarbon alkyl, and can be obtained, for example, from Chemtura. An example isopropyl amine sulphonate salt is dodecyl benzene sulfonic acid isopropyl amine, which is available from Croda.

[0019] In some examples, the charge director may constitute from about 0.001% to about 20%, for example from about 0.01 % to about 20% by weight, as an additional example from about 0.01 to about 10% by weight, and as a further example from about 0.01 % to about 1% by weight of the solids of an electrophotographic ink composition. The charge director may constitute from about 0.001 % to about 0.15 % by weight of the solids of the electrophotographic ink composition, for example from about 0.001 % to about 0.15 %, as a further example from about 0.001 % to about 0.02 % by weight of the solids of an electrophotographic ink composition, for example from about 0.1 % to about 2 % by weight of the solids of the electrophotographic ink composition, for example from about 0.2 % to about 1.5 % by weight of the solids of the electrophotographic ink composition in an example from about 0.1 % to about 1 % by weight of the solids of the electrophotographic ink composition, for example from about 0.2 % to about 0.8 % by weight of the solids of the electrophotographic ink composition. The charge director can be present in an amount of at least 1 mg of charge director per gram of solids of the electrophotographic ink composition (which will be abbreviated to mg/g), for example at least 2 mg/g, in a further example at least 3 mg/g, in another example at least 4 mg/g, for example at least 5 mg/g. The moderate acid can be present in the amounts stated above, and the charge director can be present in an amount of from about 1 mg to about 50 mg of charge director per gram of solids of the electrostatic ink composition (which will be abbreviated to mg/g), for example

from about 1 mg/g to about 25 mg/g, as a further example from about 1 mg/g to about 20 mg/g, for example from about 1 mg/g to about 15 mg/g, as an additional example from about 1 mg/g to about 10 mg/g, for example from about 3 mg/g to about 20 mg/g, as a further example from about 3 mg/g to about 15 mg/g, and for example from about 5 mg/g to about 10 mg/g.

[0020] The electrophotographic ink composition may include a charge adjuvant. A charge adjuvant may promote charging of the particles when a charge director is present. The method as described here may involve adding a charge adjuvant at any stage. The charge adjuvant may include, but is not limited to, barium petronate, calcium petronate, Co salts of naphthenic acid, Ca salts of naphthenic acid, Cu salts of naphthenic acid, Mn salts of naphthenic acid, Ni salts of naphthenic acid, Zn salts of naphthenic acid, Fe salts of naphthenic acid, Ba salts of stearic acid, Co salts of stearic acid, Pb salts of stearic acid, Zn salts of stearic acid, Al salts of stearic acid, Zn salts of stearic acid, Cu salts of stearic acid, Pb salts of stearic acid, Fe salts of stearic acid, metal carboxylates (e.g., Al tristearate, Al octanoate, Li heptanoate, Fe stearate, Fe distearate, Ba stearate, Cr stearate, Mg octanoate, Ca stearate, Fe naphthenate, Zn naphthenate, Mn heptanoate, Zn heptanoate, Ba octanoate, Al octanoate, Co octanoate, Mn octanoate, and Zn octanoate), Co lineolates, Mn lineolates, Pb lineolates, Zn lineolates, Ca oleates, Co oleates, Zn palmitate, Ca resinate, Co resinate, Mn resinate, Pb resinate, Zn resinate, AB diblock copolymers of 2-ethylhexyl methacrylate-co- methacrylic acid calcium and ammonium salts, copolymers of an alkyl acrylamidoglycolate alkyl ether (e.g., methyl acrylamidoglycolate methyl ether-co-vinyl acetate), and hydroxy bis(3,5-di-tert-butyl salicylic) aluminate monohydrate. In an example, the charge adjuvant may be or may include aluminum di- or tristearate. The charge adjuvant may be present in an amount of from about 0.1 to about 5 % by weight, for example from about 0.1 to about 1 % by weight, in some examples from about 0.3 to about 0.8 % by weight of the solids of the electrophotographic ink composition, in some examples from about 1 wt. % to about 3 wt.% of the solids of the electrophotographic ink composition, in some

examples from about 1.5 wt. % to about 2.5 wt.% of the solids of the electrophotographic ink composition.

[0021] In some examples, the electrophotographic ink composition may include, *e.g.* as a charge adjuvant, a salt of multivalent cation and a fatty acid anion. The salt of multivalent cation and a fatty acid anion can act as a charge adjuvant. The multivalent cation may, in some examples, be a divalent or a trivalent cation. In some examples, the multivalent cation may be selected from Group 2, transition metals and Group 3 and Group 4 in the Periodic Table. In some examples, the multivalent cation may include a metal selected from Ca, Sc, Ti, V, Cr, Mn, Fe, Co, Ni, Cu, Zn, Al and Pb. In some examples, the multivalent cation is Al^{3+} . The fatty acid anion may be selected from a saturated or unsaturated fatty acid anion. The fatty acid anion may be selected from a C_8 to C_{26} fatty acid anion, in some examples a C_{14} to C_{22} fatty acid anion, in some examples a C_{16} to C_{20} fatty acid anion, in some examples a C_{17} , C_{18} or C_{19} fatty acid anion. In some examples, the fatty acid anion may be selected from a caprylic acid anion, capric acid anion, lauric acid anion, myristic acid anion, palmitic acid anion, stearic acid anion, arachidic acid anion, behenic acid anion and cerotic acid anion.

[0022] The charge adjuvant, which may, for example, be or include a salt of multivalent cation and a fatty acid anion, may be present in an amount of from about 0.1 wt.% to about 5 wt.% of the solids of the electrophotographic ink composition, in some examples in an amount of from about 0.1 wt.% to about 2 wt.% of the solids of the electrophotographic ink composition, in some examples in an amount of from about 0.1 wt.% to about 2 wt.% of the solids of the electrostatic ink composition, in some examples in an amount of from about 0.3 wt.% to about 1.5 wt.% of the solids of the electrophotographic ink composition, in some examples from about 0.5 wt.% to about 1.2 wt.% of the solids of the electrophotographic ink composition, in some examples from about 0.8 wt.% to about 1 wt.% of the solids of the electrophotographic ink composition, in some examples from about 1 wt. % to about 3 wt.% of the solids of the

electrophotographic ink composition, in some examples from about 1.5 wt. % to about 2.5 wt.% of the solids of the electrophotographic ink composition.

[0023] The electrophotographic ink compositions disclosed herein may be prepared by mixing at least one resin with a carrier liquid to form a paste. The rotation of the mixing may range from about 30 rpm to about 80 rpm. A temperature for the mixing may be carried out a temperature ranging from about 10° C to about 140° C, for example from about 30° C to about 120° C, and as a further example from about 60° C to about 85° C. The mixing may be for a period of time ranging from about 10 minutes to about 300 minutes, for example from about 20 minutes to about 280 minutes, and for example from about 30 minutes to about 240 minutes.

[0024] A taggant may be ground with the paste and a charge adjuvant in an attritor, such as a ceramic attritor, and ground for a period of time, such as from about 2.5 hours to about 25 hours, for example from about 3 hours to about 20 hours, and as a further example from about 5 hours to about 15 hours.

[0025] The grinding process may be carried out at a temperature ranging from about 10° C to about 60° C, for example from about 20° C to about 50° C, and as a further example from about 26° C to about 45° C. The grinder may rotate at a rate of from about 100 rpm to about 300 rpm, for example at a rate of from about 100 rpm to about 300 rpm, such as about 200 rpm.

[0026] The following examples illustrate examples of the disclosure that are presently best known. However, it is to be understood that the following are only examples or illustrative of the application of the principles of the present disclosure. Numerous modifications and alternative compositions, methods, and systems may be devised by those skilled in the art without departing from the spirit and scope of the present disclosure. The claims are intended to cover such modifications and arrangements. Thus, while the present disclosure has been described above with particularity, the following examples provide further details in connection with what are presently deemed to be the most practical and preferred examples of the disclosure.

[0027] Examples

[0028] Example 1

[0029] A resin (NUCREL® 699, an ethylene methacrylic acid copolymer available from DuPont®, Wilmington, DE) and AC5120 (an ethylene acrylic acid copolymer by Allied Signal) were added at 40% non-volatile solids with a carrier liquid (ISOPAR L, available from Exxon Corporation) to a Ross double planetary mixer having a temperature of from about 120° C to about 140° C. The mixing process was carried out at about 50 rpm for about 90 minutes. The rpms were raised to about 70 rpm for about 120 minutes, the temperature was then lowered to room temperature and the rpm lowered to 50 rpm for 30 minutes. The resultant product was a paste.

[0030] A taggant (3051F, available from Bsecure, Ltd. in Israel) was ground with the paste at a ratio of 1:5 (taggant:paste) by weight for 8 hours in the presence of a carrier liquid (ISOPAR L) and 2% VCA (aluminum stearate as a charge adjuvant, available from Sigma Aldrich) in a ceramic attritor, without any prior treatment. The grinding conditions were: about 26° C to about 45° C, 250 rpm and the overall material in the attritor was 1700 g. A slurry was formed comprising 20% non-volatile solids and.

[0031] From the formed slurry a 2% non-volatile solids working dispersion was used to form a liquid electrophotographic ink. The taggant in the liquid electrophotographic ink may emit or absorb visible light at a wavelength in the range of from about 400 nm to about 700 nm.

[0032] Although described specifically throughout the entirety of the instant disclosure, representative examples of the present disclosure have utility over a wide range of applications, and the above discussion is not intended and should not be construed to be limiting, but is offered as an illustrative discussion of aspects of the disclosure.

[0033] What has been described and illustrated herein is an example of the disclosure along with some of its variations. The terms, descriptions and figures used herein are set forth by way of illustration only and are not meant as

limitations. Many variations are possible within the spirit and scope of the disclosure, which is intended to be defined by the following claims -- and their equivalents -- in which all terms are meant in their broadest reasonable sense unless otherwise indicated.

What is Claimed Is:

1. A liquid electrophotographic ink comprising:
a taggant; and
a carrier liquid.
2. The ink of claim 1, wherein the taggant has at least one optical property selected from the group consisting of fluorescence, absorption, polarization, Raman scattering and nonlinear optical properties.
3. The ink of claim 1, wherein the taggant is present in the ink in a de minimus amount so that the taggant is detected.
4. The ink of claim 1, wherein the taggant is a fluorescent material which upon irradiation with ultraviolet light emits or absorbs at a wavelength within a range of from about 320 to about 340 nm.
5. The ink of claim 1, wherein the taggant is a fluorescent material which upon irradiation with infrared light emits or absorbs at a wavelength within a range of from about 760 to about 860 nm.
6. The ink of claim 1, wherein the taggant is a fluorescent material that emits or absorbs visible light at a wavelength within a range of from about 400 nm to about 700 nm.
7. The ink of claim 1, comprising a resin selected from the group consisting of ethylene acrylic acid copolymers; methacrylic acid copolymers; ethylene vinyl acetate copolymers; copolymers of ethylene and alkyl ester of methacrylic or acrylic acid; copolymers of ethylene acrylic or methacrylic acid and alkyl ester of methacrylic or acrylic acid; copolymers of acrylic or methacrylic acid and at least

one alkyl ester of acrylic or methacrylic acid; ethylene-acrylate terpolymers; ethylene-acrylic esters-maleic anhydride or glycidyl methacrylate terpolymers; ethylene-acrylic acid ionomers; and combinations thereof.

8. The ink of claim 1, wherein the ink includes from about 1% to about 3% non-volatile solids.

9. The ink of claim 1, wherein the carrier liquid is a low dielectric solvent.

10. A method comprising:
grinding a taggant and a paste having a resin and a carrier liquid to form a slurry;
and
diluting the slurry to form a liquid electrophotographic ink.

11. The method of claim 10, wherein the grinding is performed in a ceramic attritor.

12. The method of claim 10, wherein the electrophotographic ink includes from about 1% to about 3% non-volatile solids.

13. The method of claim 10, wherein paste comprises from about 10% to about 50% non-volatile solids.

14. The method of claim 10, wherein a temperature for the method progressively decreases over time from about 170°C to about room temperature.

15. The method of claim 10, wherein a weight ratio of taggant to paste ranges from about 1:3 to about 1:10.

INTERNATIONAL SEARCH REPORT

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A. CLASSIFICATION OF SUBJECT MATTER INV. G03G9/12 G03G9/125 G03G9/13 ADD.		
According to International Patent Classification (IPC) or to both national classification and IPC		
B. FIELDS SEARCHED Minimum documentation searched (classification system followed by classification symbols) G03G		
Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched		
Electronic data base consulted during the international search (name of data base and, where practicable, search terms used) EPO-Internal, WPI Data		
C. DOCUMENTS CONSIDERED TO BE RELEVANT		
Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US 2008/233313 A1 (CHATOW UDI [US] ET AL) 25 September 2008 (2008-09-25) examples 4-7 claims 1-9 paragraphs [0028] - [0031] -----	1-15
X	US 2009/239174 A1 (INABA YOSHIHIRO [JP] ET AL) 24 September 2009 (2009-09-24) claims 1-20 -----	1-3,7
X	WO 01/88619 A1 (HEWLETT-PACKARD INDIGO BV) 22 November 2001 (2001-11-22) claims 1-57 -----	1-3,7,9
X	EP 0 528 737 A1 (EASTMAN KODAK CO [US]) 24 February 1993 (1993-02-24) claims 1-9 -----	1-3,9
☐ Further documents are listed in the continuation of Box C. ☒ See patent family annex.		
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