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(54) Title: COATING COMPOSITION CURABLE WITH ULTRAVIOLET RADIATION

(57) Abstract: A UV radiation curable primer coating composition curable under a UV radiation emitting lamp or sunlight. The coating demonstrates good curing time and includes ethylenically unsaturated free radically polymerizable compounds and 0.1% photoinitiator or less

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COATING COMPOSITION CURABLE WITH ULTRAVIOLET RADIATION

CROSS REFERENCE TO RELATED APPLICATIONS

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[0001] This application claims priority to Provisional U.S. Patent Application Serial No. 60/477,413, filed June 10, 2003.

BACKGROUND OF THE INVENTION

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[0002] Coatings used for the repair of a vehicle surface comprise several layers of different coating compositions such as primer, basecoat and clearcoat. Primer coating compositions are usually the initial coating layer applied in a vehicle coating repair process. The primer may be applied directly to bare metal or to an electrocoated substrate. In refinish applications a primer may also be applied to a substrate having one or more coating layers thereon. It is desirable that the coating cures quickly and that it provide a layer of paint that is sandable with sandpaper or other abrasive to remove imperfections from the coating layer. Typically the thickness of the coating layer is from 25 μm to 100 μm .

20 [0003] One class of coating compositions, well known for use in primers, comprises a hydroxyl functional polymer, such as polyester or acrylic polymer, and a polyisocyanate. These two components react together after application to the substrate to form a polyurethane coating. These compositions are often thus referred to as 2K polyurethane coating compositions, although technically the polyurethane is formed through crosslinking. 2K Polyurethane primers of this general type have
25 been known for at least 20 years.

[0004] One problem with using such 2K polyurethane primers is that sufficient time must be allowed for curing to occur before it can be sanded and topcoated. Typically a primer must cure for 2-4 hours at ambient temperature. An additional drawback to polyurethane primers is that they contain relatively high
30 levels of organic solvents. Due to environmental considerations there is a general trend in the coatings industry towards coating compositions with reduced solvent content.

[0005] Primer/sealant compositions curable under ultraviolet and thermal
35 conditions are disclosed in PCT/EP00/03401. These coating compositions however

require resins with ultraviolet curable groups and thermally curable functionality such as isocyanate functionality. Thermal cure must be applied in addition to the ultraviolet cure to achieve adequate cure of the coating composition, requiring additional energy expenditure and longer curing times.

5 [0006] Primer compositions curable under ultraviolet conditions only are disclosed in WO-01/74499 A1. This application discloses a primer coating and process for applying a primer coating to a substrate. This coating cures only under ultraviolet light.

 [0007] The present invention provides a primer coating composition
10 curable under natural outdoor light or by ultraviolet lamp radiation. The coating dries quickly, is sandable and provides adhesion to bare metal, as well as corrosion and humidity resistance.

SUMMARY OF THE INVENTION

 [0008] This invention relates to a primer composition and a method of
15 applying a primer composition to a substrate. The present invention provides a primer composition that can be cured by exposure to UV radiation having a very low UV-B content and substantially no UV-C content or under natural outdoor light within 2 to 5 minutes. The primer provides a tack free surface after curing under these conditions and exhibits good sanding characteristics, good hiding, and
20 provides adhesion to cold rolled steel as well as e-coated substrates and substrates having aged coatings thereon.

 [0009] The primer coating composition comprises: A) one or more compounds containing one ethylenically unsaturated free-radically polymerizable group per molecule; B) one or more compounds containing two or more
25 ethylenically unsaturated free radically polymerizable groups per molecule; C) one or more pigments, fillers and/or dyes; D) less than 1.0% of one or more photoinitiators; and compounds selected from volatile organic solvents and customary additives and mixtures thereof.

 [0010] The process of priming a surface comprises: i) applying a UV
30 radiation curable primer to the area to be primed; ii) exposing the primer to natural outdoor light or UV radiation provided by one or more UV lamps. The primer cures within 2 to 5 minutes under natural outdoor light or by UV radiation provided

by one or more UV lamps having a UV-B:UV-A ratio of 1:1 or less and substantially no UV-C content. In one embodiment the process can be operated easily and safely in a typical automotive refinish bodyshop. The process is adaptable to an OEM coating application environment as well. Little or no organic solvent is emitted during the application and cure of the primer and the primer can be applied and cured to a non-tacky finish in a very short time.

DETAILED DESCRIPTION

[0011] This invention relates to a primer composition and a method of applying a primer composition to a substrate. The UV radiation curable primer comprises: component A comprising one or more compounds containing one ethylenically unsaturated free-radically polymerizable group per molecule, present in an amount between 5-50% by weight, more preferably 10 to 40% by weight and most preferably 25 to 35% by weight; component B) comprising one or more compounds containing two or more ethylenically unsaturated free radically polymerizable groups per molecule present in an amount between 5-50% by weight, more preferably from 10-40% by weight and most preferably from 25 to 35% by weight; component C) comprising one or more pigments, fillers and/or dyes is present in an amount from 1-60 % by weight, more preferably from 10 to 60 % by weight; component D) comprising one or more photoinitiators is present in an amount from 0.1 to 0.98 % by weight, more preferably form 0.1 to 0.95% by weight; component E) comprising volatile organic solvent is optionally present in an amount from 0-20% by weight; and component F) comprising customary additives is present in an amount between 0.1-20% by weight, more preferably from 0.1 to 15 % by weight and most preferably from 0.1 to 10 % by weight. All amounts are based on total coating composition weight. All ranges of amounts are intended to include each and every point within the range.

[0012] In general, the type and level of component A are chosen to give the composition a suitable viscosity and to dissolve components B and D. The choice and level of component A also affects the hardness of the coating. The type and level of component B is chosen to provide suitable adhesion, flexibility, hardness and solvent resistance. Preferably the ethylenically unsaturated groups in

component A and component B are principally acrylate groups as this results in rapid cure rate.

[0013] Examples of compounds suitable as component A are alpha-beta unsaturated aliphatic compounds such as 1-octene, 1-hexene and 1-decene, vinyl esters such as vinyl acetate, styrene, substituted styrenes such as alpha-methylstyrene or p-methylstyrene, esters of methacrylic acid and esters of acrylic acid. Preferably component A is selected from alkyl esters of acrylic acid such as butyl acrylate, t-butyl acrylate, isobornyl acrylate, isodecyl acrylate, 2-ethylhexyl acrylate, lauryl acrylate, cyclohexyl acrylate and octyl acrylate. More preferably component A comprises isobornyl acrylate and/or 2-ethylhexyl acrylate.

[0014] Examples of compounds suitable as component B include diacrylates such as hexanediol diacrylate or tripropyleneglycol diacrylate, triacrylates such as trimethylolpropane triacrylate, alkoxylated trimethylolpropane triacrylate or pentaerythritol triacrylate, polyacrylates such as pentaerythritol tetraacrylate or dipentaerythritol hexaacrylate, urethane acrylates such as Ebecryl 8402, or Ebercyl 8301 available from UCB Chemicals or Actilane 251 available from Akcros Chemicals or unsaturated polyesters such as polyesters prepared with maleic anhydride as one of the monomeric components. Preferably component B comprises one or more urethane acrylates, more preferably component B comprises a mixture of di- and poly-functional urethane acrylates.

[0015] In principle component C may be any known pigment or filler. Conventional pigments such as titanium dioxide iron oxide, lithopone and zinc oxide can be included. Suitable fillers include clays, barytes, mica, talcs, zinc sulfate, calcium sulfate and calcium carbonate. Component C can include those pigments known to inhibit corrosion such as strontium chromate, zinc phosphate and barium metaborate. Preferred pigments and fillers are those that do not interfere substantially with the radical cure. Preferably component C comprises zinc phosphate, barium sulphate, and talc.

[0016] The types and levels of pigments are chosen principally in order to achieve excellent sandability and corrosion resistance, while providing minimum cure time under outdoor natural light conditions or under ultraviolet lamp exposure conditions as specified herein. Preferably the levels and types of pigments and

fillers are chosen to achieve a pigment to binder ratio of between 0.8 and 2.0, most preferably between 1.2 and 1.8.

[0017] Primer compositions containing preferred pigments are often transparent or translucent. In many cases it is desirable if the thickness of the coating can be estimated during application. One way of achieving this is to include a dye in the composition. Suitable dyes include metal complex dyes such as the Zapon(R) range available from BASF Aktiengesellschaft. When a dye is present, it is preferably present at a level of 0.005-1% by weight based on the total composition, more preferably 0.02-0.05%. An alternative way of achieving this is to include a metal flake pigment in the composition. Suitable metal flake pigments include aluminium flake pigments such as the Stappa range available from Eckart-Werke. When metal flake pigment is present, it is preferably present at a level of 0.1-10% by weight based on the total composition, more preferably 0.25- 1%.

[0018] Component D can be any photoinitiator(s) capable of generating free radicals when exposed to radiation having a UV-B: UV-A ratio of 1:1 or less. Preferably the photoinitiator(s) are capable of generating free radicals when exposed to radiation having a UV-B:UV-A ratio is 0.2:1 or less, more preferably 0.05:1 or less and even more preferably 0.025:1 or less. Preferred photoinitiators include acyl phosphine oxides, for example Irgacure 819 or benziketals such as Irgacure 651, available from Ciba Specialty Chemicals. Preferably the primer contains 0.1% to 0.98% by weight of component D.

[0019] The volatile organic solvent can be any solvent which will dissolve components A, B, D and F. It can be an aliphatic or an aromatic hydrocarbon such as Solvesso 100®, a mixture of aromatic solvents having an aromatic solvent content of 99.5% by weight and comprising primarily C9-10 dialkyl and trialkyl benzenes. Also suitable are toluene or xylene, alcohols such as n-butanol or isopropanol, esters such as iso-butyl acetate, n-butyl acetate, n-propyl acetate, methyl acetate or ethyl acetate, ketones such as acetone, methyl isobutyl ketone or methyl ethyl ketone, ethers, ether-alcohols or ether-esters such as ethyl 3-ethoxypropionate, propylene glycol methyl ether, propylene glycol ethyl ether, propylene glycol n-propyl ether or propylene glycol t-butyl ether or a mixture of any of these. Preferably one or a combination of acetone, methyl ethyl ketone, ethyl 3-ethoxypropionate and n-butyl acetate are utilized.

[0020] The level and type of solvent used will depend on the viscosity of the other components and the intended application method. In a preferred embodiment the primer contains no volatile organic solvent.

[0021] The primer composition may also contain, as component F, conventional additives such as photosynergists, adhesion promoters, pigment dispersants, flow aids, wetting aids and rheology modifiers.

[0022] The process of priming a surface (preferably a vehicle or vehicle component) comprises; i) applying a UV radiation curable primer to the area to be primed; ii) curing the primer with natural light or UV radiation provided by one or more UV lamps.

The primer composition is cured by exposure to UV radiation. When several applications of primer are used, the primer can be cured after each application or only after the final application. The ratio of UV-B content to UV-A content of the radiation is 1:1 or less. Preferably the UV-B to UV-A ratio is 0.2:1 or less, more preferably 0.05:1 or less and even more preferably 0.025:1 or less. The radiation has substantially no UV-C content. UV-A radiation is any radiation falling within the 320-400 nm wavelength interval, UV-B radiation is any radiation falling within the 280-320 nm wavelength and UV-C radiation is any radiation falling within the 200-280 nm wavelength interval.

The UV light source applied has intensity ranges as follows: UVA intensity is from 0.8 to 1.6 Joules/cm², the UVB intensity is from .001 to 0.5 Joules/cm² and the UVC intensity is from .001 to 0.3 Joules/cm².

[0023] The radiation may be provided by any suitable UV lamp and preferably the lamp(s) are positioned so that they are between 5 and 60 cm away from the surface of the primer. More preferably the lamp(s) are positioned so that they are from 10 to 30 cm away from the surface of the primer.

[0024] When the primer is cured by exposure to natural outdoor light to cure, preferably the primer is exposed to the natural outdoor light for 1 to 10 minutes. Outdoor natural light providing curing conditions have an intensity of from 5 to 100 mJoules/cm². In one embodiment the coating cured to a non-tacky surface after 5 minute exposure to outdoor light having an intensity of 45-65 mJoules/cm².

[0025] When the primer is cured by lamp radiation, exposure to the lamp radiation is for from 30 seconds to 10 minutes, more preferably from 1 to 3 minutes. Alternatively, the primer may be cured by exposure to a combination of both natural outdoor light and UV lamp radiation. The outdoor visible light has a wavelength of between 320 and 430 nm.

[0026] The spectral output of a given radiation source can be measured with an energy dispersive spectrograph comprising a monochrometer and light detector whose sensitivity is known at the relevant wavelengths. The ratio of UB-B:UV-A is arrived at by integrating the intensities of spectral output in the respective wavelength ranges. An example of a suitable instrument is the model 440 spectrometer available from Spectral Instruments, Tucson Ariz., USA.

[0027] Following cure, the primer can be sanded using sand- or glasspaper prior to topcoating. If any sticky uncured surface layer exists, it is removed by wiping the surface of the primer with a volatile organic cleaning solvent prior to optionally sanding and topcoating. The volatile organic cleaning solvent can be an aliphatic or aromatic hydrocarbon such as Solvesso 100(R), toluene or xylene, an alcohol such as n-butanol or isopropanol, an ester such as butyl acetate or ethyl acetate, a ketone such as acetone, methyl isobutyl ketone or methyl ethyl ketone, an ether, an ether-alcohol or an ether-ester, water or a mixture of any of these.

[0028] The primer may be topcoated with any suitable refinish system in order to achieve the desired final color and appearance. For example the primer can be coated with a waterborne basecoat such as Glasurit L-90 available from BASF, followed by application of a solventborne urethane clearcoat such as 923-450 available from BASF. Alternatively the primer can be topcoated with a solventborne basecoat such as Glasurit L-55 available from BASF, followed by application of a solventborne urethane clearcoat such as 923-450 available from BASF. Alternatively it can be topcoated with a solventborne single layer urethane topcoat such as Glasurit 22 line available from BASF.

[0029] The process is particularly suited to, but not limited to, carrying out spot repairs to damaged vehicles. A spot repair refers to a repair of damage that is limited to small area of a vehicle panel, usually less than 50 cm. According to the present invention there is also provided a substrate coated or

partially coated by the process of the invention.

[0030] The invention will now be illustrated by means of the following examples.

5

EXAMPLES:

Example 1

Photoinitiator Solution

[0031] The following ingredients were combined with stirring to form the photoinitiator solution: 8.3 grams Irgacure 819 and 1.7 grams Irgacure 651, both photoinitiator compositions available from Ciba and 90 grams acetone.

10

Example 2

Primer Coating Compositions

[0032] A primer paste formulation was prepared as follows:

Ingredient	Amount (grams)
Difunctional Urethane Acrylate ¹	11.1
Hexafunctional Urethane Acrylate I ²	5.6
Hexafunctional Urethane Acrylate II ³	5.6
Tripropylene glycol diacrylate	7.0
Solvent	6.3
Rheology Control Agent	0.7
Dispersing Agent	0.4
Talc	16.7
Barium Sulfate	16.7
Zinc Phosphate	16.7
Mono-oxide black	0.3
Methyl ethyl ketone	5.7

¹ Ebercyl 8402 from UCB Chemicals

² Ebercyl 8301 from UCB Chemicals

³ Developmental Ebercyl from UCB Chemicals

15

The primer was formed by the addition of the primer paste to the following:

Ingredient	Amount (grams)			
Primer Paste Formulation	174.1	169.5	165.8	155.8
Photoinitiator Solution from Ex.1	10	15	19	30
Acetone	20.0	15.0	11.0	----
Acidic Adhesion Promoter	4.3	4.3	4.3	4.3

20

[0033] The ingredients were mixed and sprayed over cold rolled steel that had been sanded with 220 grit sandpaper. The films were applied in two coats with a one-minute flash dry between coats. After the second coat was applied, the film was flash dried for 3 minutes then cured with an ultraviolet lamp Panacol 450 for 2 minutes where the substrate was at a distance of 10 inches from the light source.

Table 1**Evaluation of Primers for Surface Cure**

[0034] Following curing as set forth above, the panels were subjected to surface tests as follows. (1) Sand was applied to the coated panel, rating is how much of the area was covered by large sand sphere- (100% is completely covered); (2) cotton balls were pressed on to the panel surface- rating is % of surface covered by cotton ball fibers; (3) paper was placed on a panel and a 100 gram weight applied for one minute- passed if paper fell off, failed if paper stuck to the panel. Test results are as follows

Sample	1	2	3	Control*
% photoinitiator	0.5	0.75	0.95	
	% adherence to surface of panel			
Sand	60	40	20	100
Cotton Ball	15	15	15	80
Paper	passed	passed	passed	Failed

* UV Primer Surfacer PPG DS1002

Table 2**Evaluation of Primers for Humidity Resistance and Adhesion**

Sample	1	2	3	Control*
% Initial Adhesion	100	100	99	100
% 7 day Adhesion	100	100	100	100

*UV Primer Surfacer PPG DS1002

[0035] Coatings were prepared according to Example 2 and applied to a cold rolled steel (CRS) substrate in two coats with a one-minute flash dry between

coats. After the second coat was applied, the film was flash dried for 3 minutes then cured under one of the following conditions:

1. Exposure to daylight in Whitehouse, Ohio on May 6, 2003 at a visible light wavelength of approximately 340-430 nm and intensity of 45-65 mJoules/cm², for time indicated;
2. Exposure to ultraviolet lamp Panacol 450 (UVA only) for time indicated where the substrate was at a distance of 10 inches from the light source;
3. Exposure to a mixture of UVA/B/C under ultraviolet lamp with a D-bulb, 2 passes, at a UVA intensity of 1.6 Joules/cm², a UVB intensity of 0.5 Joules/cm², and a UVC intensity of 0.3 Joules/cm² per pass.

Adhesion was determined by the X-scribe test, the test score indicating the amount of paint remaining following the test.

Example 3

Primer Cured in Outdoor Natural Light

The following ingredients were mixed together to form the primer composition.

Ingredient	Amount (grams)
Difunctional Urethane Acrylate ¹	10.6
Hexafunctional Urethane Acrylate I ²	5.3
Hexafunctional Urethane Acrylate II ³	5.3
Tripropylene glycol diacrylate	6.7
Solvent	6.0
Rheology Control Agent	0.7
Dispersing Agent	0.35
Talc	15.9
Barium Sulfate	15.9
Zinc Phosphate	15.9
Mono-oxide black	0.26
Methyl ethyl ketone	5.4
Acetone	8.6
DS 3194	0.8
Photoinitiator	0.16
Adhesion promoter	2.1

¹ Ebercryn 8402 from UCB Chemicals

² Ebercryn 8301 from UCB Chemicals

³ Developmental Ebercryn from UCB Chemicals

Table 3 Adhesion of Primer to Cold Rolled Steel

Type of Exposure	Exposure Time (minutes)	Initial Adhesion	Post Humidity Adhesion	24 Hour Recovery Adhesion
Outdoor Natural Light	5	95	95	99
	10	99	99	95
	20	95	99	99
UVA lamp	2	95	95	95
	2	99	99	95
UVA/B/C lamp		95	95	95
		95	90	95

CLAIMS:

1. A UV radiation curable primer coating composition comprising
 - a. 5 to 50 % by weight of one or more compounds containing one ethylenically unsaturated free-radically polymerizable group per molecule
 - b. 5 to 50% by weight of one or more compounds containing two or more ethylenically unsaturated free radically polymerizable groups per molecule
 - c. 1.0 to 60% by weight of one or more pigments, fillers and or dyes
 - d. 0.1 to 0.95 % photoinitiators
 - e. 0 to 20% by weight of volatile organic solvent and
 - f. 0.1 to 10% by weight of additives,wherein said coating is curable to a non-tacky surface under a UVA radiation emitting lamp within 2 minutes and in sunlight within 5 minutes.
2. A primer coating composition according to claim 1 wherein the compound A is selected from the group consisting of 1-octene, 1-hexene, 1-decene, vinyl acetate, styrene, alpha-methylstyrene, p-methylstyrene, esters of methacrylic acid and esters of acrylic acid.
3. A primer coating according to claim 1 wherein compound A is selected from butyl acrylate, t-butyl acrylate, isobornyl acrylate, isodecyl acrylate, 2-ethylhexyl acrylate, lauryl acrylate, cyclohexyl acrylate and octyl acrylate.
4. A primer coating composition according to claim 1 wherein compound B is selected from the group consisting of urethane acrylates, diacrylates, triacrylates, polyfunctional acrylates and mixtures thereof.
5. A primer coating according to claim 1 wherein compound B is selected from the group consisting of hexanediol diacrylate, tripropyleneglycol diacrylate, trimethylolpropane triacrylate, alkoxylated

trimethylolpropane triacrylate, pentaerythritol triacrylate, pentaerythritol tetraacrylate, dipentaerythritol hexaacrylate, urethane acrylates and unsaturated polyesters, and mixtures thereof.

- 5 6. A primer coating according to claim 1 wherein compound B is selected from the group consisting of di- and poly-functional urethane acrylates.
7. A primer coating composition according to claim 1 wherein the pigment to binder ratio is between 0.8 and 2.0.
- 10 8. A primer coating composition according to claim 1 wherein the pigment to binder ratio is between 1.2 and 1.8.
- 15 9. A primer coating composition according to claim 1 wherein the photoinitiator comprises a compound selected from the group consisting of acyl phosphine oxides and benziketals.
- 20 10. A primer coating composition according to claim 1 wherein said coating is cured by 5 minute exposure to outdoor light having an intensity of 45-65 mJoules/cm² and demonstrates 95% post humidity test adhesion.
- 25 11. A process for applying a primer coating composition to a substrate comprising
- A. applying a UV radiation curable primer to a substrate;
- B. curing the primer with a source selected from the group consisting of one or more UV lamps having a UV-B:UV-A ratio of 1:1 or less, and natural outdoor light having a wavelength between 320 and 430 nm, and mixtures thereof,
- 30 wherein the UV radiation curable primer comprises
- a. 5 to 50 % by weight of one or more compounds containing one ethylenically unsaturated free-radically polymerizable group per molecule,

- b. 5 to 50% by weight of one or more compounds containing two or more ethylenically unsaturated free radically polymerizable groups per molecule,
- c. 1.0 to 60% by weight of one or more pigments, fillers and or dyes,
- 5 d. 0.1 to 0.95 % photoinitiators,
- e. 0 to 20% by weight of volatile organic solvent and
- f. 0.1 to 10% by weight of additives.
12. A process according to claim 11 wherein the coating applied comprises compound A is selected from the group consisting of 1-octene, 1-hexene, 1-decene, vinyl acetate, styrene, alpha-methylstyrene, p-methylstyrene, esters of methacrylic acid and esters of acrylic acid and mixtures thereof.
- 10
13. A process according to claim 11 wherein the coating applied comprises compound A selected from butyl acrylate, t-butyl acrylate, isobornyl acrylate, isodecyl acrylate, 2-ethylhexyl acrylate, lauryl acrylate, cyclohexyl acrylate and octyl acrylate and mixtures thereof.
- 15
14. A process according to claim 11 wherein the coating applied comprises compound B selected from the group consisting of urethane acrylates, urethane diacrylates, tri- and polyfunctional urethane acrylates and mixtures thereof.
- 20
15. A process according to claim 11 wherein the coating applied comprises a pigment to binder ratio between 0.8 and 2.0.
- 25
16. A process according to claim 11 wherein the coating applied comprises a pigment to binder ratio between 1.2 and 1.8.
- 30
17. A process according to claim 11 wherein a UV light source is applied wherein the UVA intensity is from 0.8 to 1.6 Joules/cm², the UVB

intensity is from .001 to 0.5 Joules/cm² and the UVC intensity is from .001 to 0.3 Joules/cm².

- 5
18. A process according to claim 11 wherein the coating is cured under natural light conditions, said light providing an intensity of 5-100 mJoules/ cm².
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19. A process according to claim 11 wherein the substrate to which the coating is applied to a substrate comprising an automotive vehicle.
20. A process according to claim 11 wherein the coating process comprises application of the primer coating in the repair of an automotive vehicle.

INTERNATIONAL SEARCH REPORT

International Application No

PCT/US 03/37223

A. CLASSIFICATION OF SUBJECT MATTER

IPC 7 C09D4/00 C08F2/48 B05D3/06

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)

IPC 7 C09D C08F B05D

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 practical, search terms used)

EPO-Internal, PAJ

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	GB 2 283 975 A (NAT STARCH CHEM CORP) 24 May 1995 (1995-05-24) examples	1-5
A	WO 01/74499 A (FENN DAVID ROBERT ; HARVEY IAIN W (GB); SKORUPSKI HENRYK (GB); WALT) 11 October 2001 (2001-10-11) cited in the application page 1, paragraph 1; claims 1-22	1-20
A	WO 92/17337 A (RED SPOT PAINT & VARNISH) 15 October 1992 (1992-10-15) claims 1,16	1,11
A	US 5 559 163 A (DAWSON WILLIAM R ET AL) 24 September 1996 (1996-09-24) claim 1	1
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☒ Further documents are listed in the continuation of box C.

☒ Patent family members are listed in annex.

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In  ional Application No

PCT/US 03/37223

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