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CA 2414801 A1 2003/06/22

(21) **2 414 801**

(12) **DEMANDE DE BREVET CANADIEN
CANADIAN PATENT APPLICATION**

(13) **A1**

(22) Date de dépôt/Filing Date: 2002/12/20

(41) Mise à la disp. pub./Open to Public Insp.: 2003/06/22

(30) Priorité/Priority: 2001/12/22 (101 63 826.4) DE

(51) Cl.Int.⁷/Int.Cl.⁷ C09D 175/06, C09D 5/46, C09D 5/44,
C08L 75/06, C08L 31/06

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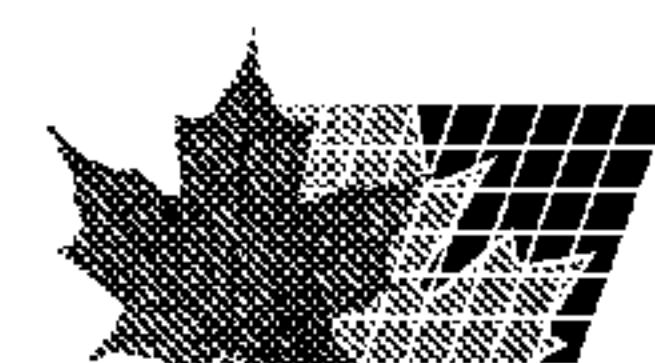
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(54) Titre : COMPOSITIONS DE REVETEMENT EN POUDRE POUVANT ETRE DURCIES SOUS RAYONNEMENT ET
LEUR UTILISATION

(54) Title: RADIATION CURABLE POWDER COATING COMPOSITIONS AND THEIR USE

(57) Abrégé/Abstract:

Disclosed is a radiation curable powder coating composition comprising at least one urethane acrylate having a glass transition temperature of at least 35°C and a melting point of from 50 to 120°C. The resulting coatings are stable to light and weather, have a combination of hardness and flexibility, and have excellent adhesion properties.



23443-798

Abstract:

Disclosed is a radiation curable powder coating composition comprising at least one urethane acrylate having a glass transition temperature of at least 35°C and a
5 melting point of from 50 to 120°C. The resulting coatings are stable to light and weather, have a combination of hardness and flexibility, and have excellent adhesion properties.

23443-798

- 1 -

RADIATION CURABLE POWDER COATING COMPOSITIONS AND THEIR USE

The invention relates to radiation curable powder coating compositions which crosslink to lightfast and weather-stable films.

5 Thermally crosslinkable powder coating materials are widely used in the paint processing industry.

For example, DE-C 27 35 497 describes polyurethane (PU) powder coatings having outstanding weather and heat stability. The crosslinkers whose preparation is described
10 in DE-C 27 12 931 are composed of ϵ -caprolactam-blocked isophorone diisocyanate containing isocyanurate groups. Blocked polyisocyanates containing urethane, biuret or urea groups can also be used.

The disadvantage of these systems lies in the
15 elimination of the blocking agent during the thermal crosslinking reaction. Since the blocking agent may be emitted into the environment, it is necessary to take special precautions to recover the blocking agent upon elimination. Moreover, the reactivity of the crosslinkers is low. Curing
20 temperatures above 170°C are required.

Both disadvantages - emission of the blocking agent and curing at high temperatures - can be avoided by using powder coating materials which are cured by radiation, rather than thermal means. Radiation curable powder coating
25 materials of this kind are described in, for example, U.S. Patent No. 3,485,732, EP 0407826, EP 0636669, WO 99/14254, U.S. Patent No. 3,974,303, U.S. Patent No. 5,639,560 and EP 0934359. EP 0636669 and WO 99/14254 describe two-component, radiation curable powder coating materials based
30 on unsaturated polyester and vinyl ether. These coatings are unsuitable for outdoor use due to severe discoloration.

23443-798

- 2 -

U.S. Patent No. 3,974,303 describes thermoplastic resins containing from 0.5 to 3.5 polymerizable unsaturated groups per 1,000 g molecular weight. The polyurethanes described therein (see Example 1), however, are not weather-
5 stable and are of poor flexibility owing to the absence of polyester groups and low chain length.

U.S. Patent No. 5,639,560 describes radiation curable powder compositions comprising special crystalline polyesters, additionally containing methacrylic end groups,
10 as binders. These powder compositions optionally comprise unsaturated oligomers, including urethane acrylates, in minor amounts, preferably up to 10 percent by weight. However, these radiation curable powder coating formulations have significant disadvantages. They can be ground only at
15 a very low temperature such as minus 80°C and, in general, the resultant coatings are either unstable in outdoor weather, or are inflexible. One serious disadvantage of these compositions is their very low pendulum hardness (according to König) of less than 120 s.

20 EP 0934359 describes pulverulant radiation curable mixtures of amorphous and crystalline polyesters containing terminal methacrylate groups. The powder coating materials produced thereof do have a higher glass transition temperature than the crystalline methacrylates of U.S.
25 Patent No. 5,639,560. However, cooling is still required for their grinding. Moreover, the hardness and adhesion of these coatings are less than satisfactory.

It is an object of the present invention to find radiation curable powder coating compositions which are
30 stable on storage at 35°C and which also, after curing of the films, lead to coatings that have high adhesion, a combination of hardness and flexibility, and are stable in

23443-798

- 3 -

outdoor weather. Moreover, the powder coating materials should be comminuted with little or no refrigerant.

Surprisingly it has been found that these objects may be achieved by means of a radiation curable powder
5 coating composition comprising, as binder, at least one amorphous urethane acrylate having a glass transition point (T_g) of at least 35°C, and a melting point of 50 to 120°C.

The invention provides radiation curable powder coating composition comprising:

- 10 A) binders composed of at least one amorphous urethane acrylate having a glass transition temperature of at least 35°C and a melting point of from 50 to 120°C; and
- B) auxiliaries or additives.

The invention likewise provides for the use of
15 compositions comprising:

- A) binders composed of at least one amorphous urethane acrylate having a glass transition temperature of at least 35°C and a melting point of from 50 to 120°C; and
- B) auxiliaries or additives,

20 for producing radiation curable powder coating compositions.

The invention further provides a process for producing radiation curable powder coating compositions by mixing:

- A) binders composed of at least one amorphous urethane
25 acrylate having a glass transition temperature of at least 35°C and a melting point of from 50 to 120°C; and
- B) auxiliaries or additives,

23443-798

- 4 -

wherein upper temperature limits of between 120°C and 130°C are maintained in heatable kneading devices, particularly extruders.

The invention also provides processes for
5 producing coatings by applying radiation curable powder coating compositions comprising:

- A) binders composed of at least one amorphous urethane acrylate having a glass transition temperature of at least 35°C and a melting point of from 50 to 120°C; and
 - 10 B) auxiliaries or additives,
- to substrates, and curing the coating compositions.

Urethane acrylates for the purposes of this invention are composed of a hydroxyl-containing polyester to which urethane groups and acrylate groups are attached by
15 reaction with polyisocyanates and acrylate-containing alcohols. These urethane acrylates have a Tg of at least 35°C and a melting point of 50 to 120°C.

The preparation methods of the amorphous urethane acrylates of the invention are generally known. Preferably
20 they are prepared by reacting amorphous hydroxyl-containing polyesters (A.1) having a Tg of 35 to 80°C with polyisocyanates (A.2) and compounds (A.3) containing at least one alcohol group and at least one polymerizable acrylate group. The resulting urethane acrylates contain
25 both urethane groups and terminal acrylate groups.

The amorphous hydroxyl-containing polyesters (A.1) having a Tg of 35 to 80°C are prepared, typically, by polycondensation of appropriate dicarboxylic acids and polyols. Condensation takes place conventionally in an inert
30 gas atmosphere at temperatures from 100 to 260°C, preferably

23443-798

- 5 -

from 130 to 220°C, in the melt or in an azeotropic procedure, as described, for example, in Methoden der Organischen Chemie (Houben-Weyl), Volume 14/2, pages 1 to 5, 21 to 23, 40 to 44, Georg Thieme Verlag, Stuttgart, 1963, or in C.R. Martens, 5 Alkyd Resins, pages 51 to 59, Reinhold Plastics Appl. Series, Reinhold Publishing Comp., New York, 1961. The carboxylic acids preferred for preparing polyesters may be aliphatic, cycloaliphatic, aromatic and/or heterocyclic in nature and may be substituted by halogen atoms and/or unsaturated. Examples 10 of preferred carboxylic acids include: succinic, adipic, suberic, azelaic, sebacic, phthalic, terephthalic, isophthalic, trimellitic, pyromellitic, tetrahydrophthalic, hexahydrophthalic, hexahydroterephthalic, dichlorophthalic, tetrachlorophthalic, endomethylenetetrahydrophthalic, glutaric 15 acid and 1,4-cyclohexanedicarboxylic acid, along with their anhydrides and/or esters. Particular preference is given to isophthalic acid, terephthalic acid, hexahydroterephthalic acid and 1,4-cyclohexanedicarboxylic acid.

Examples of suitable polyols include ethylene 20 glycol, 1,2- and 1,3-propylene glycol, 1,4- and 2,3-butylene glycol, di- β -hydroxyethylbutanediol, 1,5-pentanediol, 1,6-hexanediol, 1,8-octanediol, decanediol, dodecanediol, neopentyl glycol, cyclohexanediol, 3(4),8(9)-bis(hydroxymethyl)tricyclo[5.2.1.0^{2,6}]decane (digidol), 25 1,4-bis(hydroxymethyl)cyclohexane, 2,2-bis(4-hydroxycyclohexyl)propane, 2,2-bis[4-(β -hydroxyethoxy)phenyl]propane, 2-methylpropane-1,3-diol, 2-methylpentane-1,5-diol, 2,2,4(2,4,4)-trimethylhexane-1,6-diol, glycerol, trimethylolpropane, trimethylolethane, 30 hexane-1,2,6-triol, butane-1,2,4-triol, tris(β -hydroxyethyl)isocyanurate, pentaerythritol, mannitol, sorbitol, diethylene glycol, triethylene glycol, tetraethylene glycol, dipropylene glycol, polypropylene glycols, polybutylene glycols, xylylene

23443-798

- 6 -

glycol, and neopentyl glycol hydroxypivalate. Preference is given to monoethylene glycol, neopentyl glycol, dicitol, cyclohexanedimethanol, trimethylolpropane, and glycerol.

Subsequent or additional branching of the
5 polyesters with polyisocyanates is possible. In such cases it is advantageous to use, for example, the isophoronediiisocyanato isocyanurate.

Amorphous polyesters thus prepared preferably have an OH number of 15 to 150 mg KOH/g, a Tg of 35 to 80°C, and
10 an acid number of less than 5 mg KOH/g. It is also possible to use mixtures of amorphous polyesters.

Examples of polyisocyanates (A.2) include diisocyanates of aliphatic, (cyclo)aliphatic or cycloaliphatic structure. Representative examples of the polyisocyanates are
15 2-methylpentamethylene 1,5-diisocyanate, hexamethylene diisocyanate, trimethylhexamethylene 1,6-diisocyanate, especially the 2,2,4- and 2,4,4- isomer and technical-grade mixtures of both isomers, 4,4'-methylene-bis(cyclohexyl isocyanate), norbornane diisocyanate, and 3,3,5-trimethyl-1-
20 isocyanato-3-isocyanatomethylcyclohexane (IPDI). In addition, other suitable polyisocyanates include those obtained by reacting polyisocyanates with themselves by way of isocyanate groups, such as isocyanurates, formed by reaction of three isocyanate groups. The polyisocyanates may likewise contain
25 biuret or allophanate groups. In such instances IPDI is especially suitable.

Suitable polymerizable compounds (A.3) containing at least one free OH group and one polymerizable acrylate group include, for example, hydroxyethyl acrylate (HEA),
30 hydroxypropyl acrylate, and glycerol diacrylate. Hydroxyethyl acrylate (HEA) is especially preferred.

23443-798

- 7 -

For preparing the amorphous urethane acrylate from the amorphous OH-containing polyesters (A.1), polyisocyanates (A.2), and compounds (A.3), the polyisocyanate is introduced as an initial charge, dibutyltin laurate (DBTL) as catalyst
5 and IONOL*CP (Shell) as polymerization inhibitor are added, and the polyester is added in an NCO:OH ratio of 2.5-1.5:1. After the end of the addition, the reaction is completed at 100 to 140°C. Thereafter, component (A.3), e.g., hydroxyethyl acrylate, in a residual-NCO:OH ratio of 1.0-1.1:1 is added to
10 the reaction product and the reaction is completed at 80 to 140°C so as to give an NCO content of less than 0.1%. Also possible is a prior reaction of a polyisocyanate, e.g. IPDI, with component (A.3) and the addition of this NCO-containing precursor to the hydroxyl-containing polyester.

15 Accelerated electron beams are suitable for the radiation curing of the powder coating composition of the invention. The electron beams generate free radicals from the powder coating composition, in numbers sufficient to ensure extremely rapid polymerization of the reactive
20 acrylate groups. It is preferred to use radiation doses of from 5 to 15 Mrad.

In the case of UV curing, further necessary starting materials include UV initiators, which are known in principle from conventional liquid UV-curing systems, (see,
25 for example, EP 633912). These are substances, which upon irradiation with UV light, break down into free radicals and initiate the polymerization. Examples of suitable UV initiators include 2,2'-diethoxyacetophenone, hydroxycyclohexyl phenyl ketone, benzophenone, 2-hydroxy-2-methyl-1-phenylpropan-1-one, xanthone, thioxanthone and
30 benzil dimethyl ketal. UV initiators of this kind are available commercially, e.g., IRGACURE* 184 or DEGACURE*
*Trade-mark

23443-798

- 7a -

1173 from Ciba. The fraction of the photoinitiator in the overall system is approximately 0.5 to 5% by weight.

The use of initiators such as thioxanthenes, phosphine oxides, metallocenes, tertiary aminobenzenes or
5 tertiary aminobenzophenones, which break down into free radicals on exposure to visible light is also possible.

Optional additives are acrylate or methacrylate compounds, such as the triacrylate of tris(2-hydroxyethyl) isocyanurate (SR* 386, Sartomer), and adhesion promoters,
10 which may be used in minor fractions of 0 to 20% by weight in order to modify the coating properties.

Further additives customary in the case of powder coating materials are leveling agents, light stabilizers, and devolatilizers. These may be used at 0 to 5% by weight.
15 Also possible is the use of pigments and extenders, e.g., metal oxides such as titanium dioxide, and metal hydroxides, sulfates, sulfides, carbonates, silicates, talc, carbon black, etc., in weight fractions of 0 to 40%.

In order to prepare the ready-to-use powder coating
20 composition the ingredients are mixed. Homogenization of the ingredients may take place in suitable apparatus, such as heatable kneading devices, for example, but preferably by extrusion, with recommended upper temperature limits of 120 to 130°C. Following cooling to room temperature and
25 appropriate size reduction, the extrudate is ground to a ready-to-spray powder form without addition of refrigerants. Application of the ready-to-spray powder to appropriate substrates may take place in accordance with the known methods, such as by electrostatic or tribostatic powder
30 spraying, fluidized bed sintering or electrostatic fluid-bed sintering, for example. Examples of suitable

*Trade-mark

23443-798

- 7b -

substrates include untreated and pretreated metallic substrates, wood, wood materials, plastics, glass and paper.

The coatings produced from the powder coating compositions of the invention are stable to light and weathering; have a combination of flexibility and hardness; and possess good adhesion. The invention accordingly further provides for the use of the radiation curable compositions for producing coatings and also to a process for producing coatings by using the powder coating compositions.

The invention is illustrated by the following examples:

Examples

1. Preparation procedure for the amorphous hydroxyl-containing polyester

Acid components and alcohol components are admixed with 0.2 percent by mass of n-butyltin trioctanoate and heated at 190°C under nitrogen and with stirring in an apparatus provided with a distillation column. As water is separated out, this temperature is slowly raised to 230°C. After about 98% of the theoretical amount of water has been removed by distillation, the product is cooled and tested for OH number (OHN in mg KOH/g) and acid number (AN in mg KOH/g).

In this way, the following polyester is prepared:

A1: amorphous polyester composed of IPA 100%, MEG 20%, CHDM 40% and NPG 40%, OHN 41 mg KOH/g, AN 0.5 mg KOH/g, Tg 53°C

IPA: isophthalic acid, MEG: monoethylene glycol, CHDM: 1,4-cyclohexanedimethanol, NPG: neopentyl glycol.

23443-798

- 8 -

2. Preparation of the amorphous urethane acrylate A1U

260.0 g of the polyester A1 (OHN 41) are melted and added in portions at 120°C with vigorous stirring to a mixture of 83.3 g of a 1:1 adduct of IPDI and hydroxyethyl acrylate, 0.7 g of IONOL CP and 0.2 g of DBTL. After about one hour of stirring, the NCO content is below 0.1% and the hot reaction mixture is poured from the flask onto a sheet. As soon as the reaction mass has solidified and cooled, it is mechanically size-reduced and ground. The Tg of this product is 45°C.

3. Preparation of inventive polymers and powder coating compositions of examples 1 and 2 and of the comparative experiment Z

100 parts of the amorphous urethane acrylate A1U are admixed with 0.7 part of BYK^{*} 361 (leveling agent, BYK Chemie), 1.0 part of Worlée Add^{*} 900 (devolatilizer, Worlée-Chemie) and 1 part of EBECRYL^{*} 170 (adhesion promoter, UCB). The size-reduced ingredients are intimately mixed in an edge runner mill and then homogenized in an extruder at not more than 130°C. After cooling, the extrudate is fractionated and ground to a particle size < 100 µm using a pinned-disk mill, with (in the case of the comparative experiment) and without (in the case of the inventive example) the addition of refrigerants (liquid nitrogen or dry ice). The powder thus prepared is applied using an electrostatic powder spraying unit at 60 kV to degreased and optionally pretreated aluminum panels and MDF boards (medium density fiberboards). It is then melted under IR irradiation and cured by means of electron beams (15 Mrad).

4. Comparative example Z

For comparative example Z the following formulation was used:

300 g UVECOAT^{*} 2100 (UCB Chemicals)
 86 g UVECOAT^{*} 9010 (UCB Chemicals)
 8 g IRGACURE^{*} 184 (Ciba Specialty Chemicals)
 4 g RESIFLOW^{*} PV 5 (Worlée-Chemie)
 2 g WORLÉE^{*} Add 900 (Worlée-Chemie)

The parameters are summarized in Table 1:

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23443-798

- 9 -

Table 1

<i>Experiment</i>	<i>A1U</i>	<i>Substrate</i>	<i>Grindable without refrig.¹</i>	<i>HK² [sec]</i>	<i>EC³ [mm]</i>	<i>CC⁴</i>
1	100	Aluminum	Yes	228	6.5	0
2	100	MDF	Yes	204	-	0
Comparative Z		Standard steel	No	88	6.5	0

- 5 1: Grindability without use of refrigerant
 2: Hardness according to König (DIN 53 157)
 3: Erichsen cupping (DIN 53 156)
 4: Cross-cut(DIN 53153, ISO 2409)
 (Scale 0 (no loss of adhesion) to 5 (total loss of adhesion))

10

Only the inventive experiments 1 and 2 are grindable without addition of refrigerant and, moreover, exhibit a combination of high hardness, flexibility, and excellent adhesion in the coating.

15

Comparative example Z has weaknesses, in particular, in the grindability of the powder and in the hardness of the coating.

23443-798

- 10 -

CLAIMS:

1. A radiation curable powder coating composition comprising:

I. a binder composed of at least one amorphous urethane acrylate having a glass transition temperature of at least 35°C and a melting point of from 50 to 120°C; and

II. at least one member selected from the group consisting of auxiliaries and additives.

2. The composition of claim 1, wherein the amorphous urethane acrylate is synthesized by reacting the following components:

(A.1) at least one amorphous hydroxyl-containing polyester having a glass transition temperature of from 35 to 80°C,

(A.2) at least one polyisocyanate, and

(A.3) at least one compound containing at least one alcohol group and at least one polymerizable acrylate group.

3. The composition of claim 2, wherein the polyester (A.1) is prepared by reacting at least one monomeric dicarboxylic acid with at least one monomeric polyol.

4. The composition of claim 3, wherein the dicarboxylic acid is selected from the group consisting of succinic acid, adipic acid, suberic acid, azelaic acid, sebacic acid, phthalic acid, terephthalic acid, isophthalic acid, trimellitic acid, pyromellitic acid, tetrahydrophthalic acid, hexahydrophthalic acid, hexahydroterephthalic acid, dichlorophthalic acid,

23443-798

- 11 -

tetrachlorophthalic acid, endo-methylenetetrahydrophthalic acid, glutaric acid, 1,4-cyclohexanedicarboxylic acid, anhydrides thereof and esters thereof.

5. The composition of claim 3, wherein the
5 dicarboxylic acid is selected from the group consisting of isophthalic acid, terephthalic acid, hexahydroterephthalic acid and 1,4-cyclohexanedicarboxylic acid.

6. The composition of any one of claims 3 to 5,
wherein the polyol is selected from the group consisting of
10 ethylene glycol, 1,2- and 1,3-propylene glycol, 1,4- and 2,3-butylene glycol, di- β -hydroxyethylbutanediol, 1,5 pentanediol, 1,6-hexanediol, 1,8-octanediol, decanediol, dodecanediol, neopentyl glycol, cyclohexanediol, 3(4),8(9)-bis(hydroxymethyl)tricyclo[5.2.1.0^{2,6}]decane (digidol),
15 1,4-bis(hydroxymethyl)cyclohexane, 2,2-bis(4-hydroxycyclohexyl)propane, 2,2-bis[4-(β -hydroxyethoxy)phenyl]propane, 2-methylpropane-1,3-diol, 2-methylpentane-1,5-diol, 2,2,4(2,4,4)-trimethylhexane-1,6-diol, glycerol, trimethylolpropane, trimethylolethane,
20 hexane-1,2,6-triol, butane-1,2,4-triol, tris(β -hydroxyethyl)isocyanurate, pentaerythritol, mannitol, sorbitol, diethylene glycol, triethylene glycol, tetraethylene glycol, dipropylene glycol, polypropylene glycols, polybutylene glycols, xylylene glycol, and neopentyl glycol
25 hydroxypivalate.

7. The composition of any one of claims 3 to 5,
wherein the polyol is selected from the group consisting of monoethylene glycol, neopentyl glycol, digidol, cyclohexanedimethanol, trimethylolpropane, and glycerol.

30 8. The composition of any one of claims 2 to 7,
wherein the polyisocyanate (A.2) is selected from the group

23443-798

- 12 -

consisting of an aliphatic diisocyanate, a (cyclo)aliphatic diisocyanate, a cycloaliphatic diisocyanate, an isocyanurate and mixtures thereof.

9. The composition of claim 8, wherein the
5 diisocyanate is selected from the group consisting of 2-methylpentamethylene 1,5-diisocyanate, hexamethylene diisocyanate, trimethylhexamethylene 1,6-diisocyanate, especially the 2,2,4- and 2,4,4- isomer and technical-grade mixtures of both isomers, 4,4'-methylene-bis(cyclohexyl
10 isocyanate), norbornane diisocyanate, and 3,3,5-trimethyl-1-isocyanato-3-isocyanatomethylcyclohexane.

10. The composition of any one of claims 2 to 7, wherein the polyisocyanate (A.2) is selected from the group consisting of isophorone diisocyanate, hexamethylene
15 diisocyanate, trimethylhexamethylene diisocyanate, dicyclohexylmethyl diisocyanate, 2,2,6-trimethyl-1,4-diisocyanatocyclohexane and norbornane diisocyanate.

11. The composition of any one of claims 2 to 7, wherein the polyisocyanate contains biuret or allophanate
20 groups.

12. The composition of any one of claims 2 to 11, wherein the compound (A.3) is selected from the group consisting of hydroxyethyl acrylate (HEA), hydroxypropyl acrylate, and glycerol diacrylate.

25 13. The composition of any one of claims 2 to 12, wherein the compound (A.3) is hydroxyethyl acrylate.

14. The composition of any one of claims 1 to 13, wherein the urethane acrylate has an NCO content of less than 0.1%.

23443-798

- 13 -

15. The composition of any one of claims 1 to 14, wherein the auxiliaries and additives are selected from the group consisting of UV initiators, leveling agents, light stabilizers, devolatilizers, pigments, fillers, adhesion
5 promoters, acrylate-containing compounds, methacrylate-containing compounds and mixtures thereof.

16. The composition of claim 15, wherein the UV initiators comprise 2,2'-diethoxyacetophenone, hydroxycyclohexyl phenyl ketone, benzophenone, 2-hydroxy-2-
10 methyl-1-phenylpropan-1-one, xanthone, thioxanthone and benzil dimethyl ketal.

17. The composition of claim 15 or 16, wherein the UV initiators are present in an amount of 0.5 to 5% by weight.

18. The composition of any one of claims 15 to 17, wherein the additives comprise acrylate compounds, methacrylate compounds and adhesion promoters.

19. A radiation curable powder coating composition comprising:

A) a binder composed of an amorphous urethane acrylate
20 prepared from the reaction of:

(A.1) an amorphous hydroxyl-containing polyester prepared by the reaction of isophthalic acid with ethylene glycol, cyclohexanedimethanol and neopentyl glycol.

(A.2) 3,3,5-trimethyl-1-isocyanato-3-
25 isocyanatomethylcyclohexane; and

(A.3) hydroxyethyl acrylate;

and

23443-798

- 14 -

B) additives comprising a leveling agent, a devolatizer and an adhesion promoter.

20. A process for producing the radiation curable powder coating composition as defined in any one of claims 1 to 19, which comprises mixing the binder with the member selected from auxiliaries and additives, while maintaining an upper temperature limit of between 120°C and 130°C, in a heatable kneading device.

21. The process of claim 20, wherein the heatable kneading device is an extruder.

22. A process for coating a substrate, which comprises:

applying the powder coating composition as defined in any one of claims 1 to 15 to the substrate, and

15 then curing the powder coating composition.

23. The process of claim 22, wherein the curing is conducted by accelerated electron beams.

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