INSTRUCTIONS

(a) If Convention application insert "Convention"

662864 (a) CONVENTION

AUSTRALIA

Patents Act 1990

(b) Delete one

REQUEST FOR A (b) STANDARD/~~PETTY~~ PATENT

(c) Insert FULL name(s) of applicant(s)

~~I~~/We being the person(s) identified below as the Applicant, request the grant of a patent to the person identified below as the Nominated Person, for an invention described in the accompanying Standard/~~Petty~~ (b) Complete Patent Specification.

~~I am~~/We are (b) not an opponent or eligible person described in Sections 33-36 of the Act.

[70,71] Applicant and Nominated Person (c):

(d) Insert FULL address(es) of applicant(s)

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(e) Insert TITLE of invention

[54] Invention Title (e) THERMOSETTING COATING SYSTEMS

(f) Insert Names of actual Inventors.

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(Note: The following applies only to Convention applications)

Basic Convention Application Details

(g) Insert number, country and filing date for the/or each basic application

(g) [31] Application No.	[33] Country	Country Code	[32] Date of Application
P 44 01 438.4	Germany	DE	19 January 1994

(h) Signature of applicant(s) (For body corporate see headnote*)

(i) Insert date of signing

(j) Corporate seal if any

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Note: No legalization or other witness required

ENZINGER Hans-Ulrich Dr. SCHULZE Walter-Joachim (h) Signature of Applicant

Zürich, December 06, 1994 (i) Date

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P17/5/91

AUSTRALIA

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NOTICE OF ENTITLEMENT

INSTRUCTIONS
(a) Name of person making statement.

(b) Position of that person.

(c) Name of applicant

(d) Address of applicant

(e) Delete as necessary

(f) Insert details if not covered by (i) or (ii)

(g) Delete as necessary

(h) Delete for non-convention applications

(i) Insert DATE of signing

(j) Signature(s) of person making statement

Note: No legalization or other witness required

I (a) 1) ENZINGER Hans-Ulrich
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2) Holder of Procuration

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State the following:-

1. The nominated person (applicant) is entitled to the grant of a patent
(e) ~~(i) as assignee of the actual inventor(s)~~
(ii) by contract of employment of the actual inventor(s)
or (iii) (f)

2. The nominated person (applicant) is entitled to claim priority from the basic convention application(~~s~~).
(g) (i) as applicants of the said application(~~s~~)
~~(ii) as the assignee of the applicants of the said application(s)~~
~~(iii) with the consent of the applicants of the said application(s)~~

3. The basic convention application(~~s~~) was/~~were~~ the first made in a Convention country in respect of the invention the subject of the application. (h)

Dated (i)

(j) Zürich, December 06, 1994

ENZINGER Hans-Ulrich

Dr. SCHULZE Walter-Joachim

To: The Commissioner of Patents

PHILLIPS ORMONDE AND FITZPATRICK
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P18/3/91

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THERMOSETTING COATING SYSTEMS

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(56) Prior Art Documents
US 4801680
US 4471108

(57) Claim

1. Thermosetting coating system based on carboxyl-functional polyester resins, polyfunctional epoxy compounds and/or β-hydroxyalkylamides as cross linking agents and optionally further conventional additives, the coating system containing as binder resin at least one linear carboxyl-functional polyester resin composed of bifunctional monomer components including a maximum of 10 molar parts of isophthalic acid and at least 90 molar parts of at least one further dicarboxylic acid from the group including aromatic dicarboxylic acids with 8 to 16 carbon atoms, aliphatic dicarboxylic acids with 6 to 22 carbon atoms and cycloaliphatic dicarboxylic acids with 8 to 16 carbon atoms and at least 50 molar parts of at least one branched aliphatic diol with 4 to 12 carbon atoms and a maximum of 50 molar parts of at least one linear aliphatic diol with 2 to 22 carbon atoms and/or at least one cycloaliphatic diol with 6 to 16 carbon atoms.

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COMPLETE SPECIFICATION
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Invention Title:

THERMOSETTING COATING SYSTEMS

Our Ref : 393587
POF Code: 226745/226745

The following statement is a full description of this invention, including the best method of performing it known to applicant(s):

Thermosetting Coating System

The invention relates to thermosetting coating systems based on linear carboxyl-functional polyester (PES) resins and polyfunctional epoxy compounds and/or β-hydroxyalkylamides, the production and use thereof and to protective layers of these coatings systems.

Thermosetting coating systems generally consist of binder resin and cross linking agent, pigments and additives and/or fillers. Cross linking and therefore setting in heat takes places via polyaddition or polycondensation reactions between functional groups contained in the binder systems. Typical binder systems include epoxy resins/hardeners, polyesters/epoxides, polyesters/isocyanates, polyesters/β-hydroxyalkylamides, acrylates/isocyanates.

The macroscopic properties of cured powder coating films change over time. In the case of polymeric materials, this phenomenon has been known for a long time by the term physical aging (L.C.E. Struik: Physical Aging in Amorphous Polymers and other Materials, published by Elsevier, 1978).

Physical aging is interpreted as the change over time in the macroscopic properties of polymers in the glass stage under constant environmental conditions produced by a state of non-equilibrium in the polymer. In contrast to chemical aging during which irreversible changes such as decomposition reactions or chain breakages occur in the material owing to thermal decomposition or photooxidation, physical aging is reversible.

Changes in the electric and optical characteristics as well as changes in the mechanical properties which are most important for paint films are mentioned as examples of changes due to physical aging. Although this phenomenon is

generally known in the case of paint films, it did not appear to give rise to excessive problems in the past.

Owing to the increased use of organic pigments and the high requirements on powder coatings in precoating metal and coil coating technology, in particular when the coated parts are post formed, this phenomenon and the means for eliminating it are gaining in interest. Another interesting sphere of aplication of powder coatings with improved stability to physical aging are clear coats with higher resistance to environmental stress cracking.

In the aforementioned book "Physical Aging in Amorphous Polymers and other Materials", L.C.E. Struik describes the phenomenon of physical aging in general with reference to polymers. In the past there was little literature dealing with the phenomenon of the physical aging of organic coatings or in particular powder coatings (P.J. Greidanus, Porc. 19th Fatipec Congress, Aachen, 1, 485 (1988) and D.Y. Perera and P. Schutyser: Effect of Physical Aging on Thermal Stress Development in Powder Coatings, XIX International Conference in Organic Coating Science and Technology, Athens, 1993). Only the physical bases and the effects of physical aging are measured and described in these publications but no solution to this problem is proposed.

In the case of powder coatings based on carboxyl-functional polyesters and polyepoxides and/or β-hydroxylalkylamides as cross linking agent, the phenomenon of physical aging can be repressed by increasing the curing temperature, by extending the curing time, by an excess of cross linking agent or by the incorporation of so-called branching agents, i.e. acids or alcohols having functionality greater than 2, into the fundamental polyester resin. An increase in the curing temperature or an extension of the curing time is associated with higher energy consumption and sometimes with colour changes. An excess of cross linking agent should also be rejected for economic reasons and is associated with higher costs. The incorporation of branching agents into the polyester resin is not recommended from a technical point of view as it impairs the flow properties.

The object of the invention is therefore to overcome or

alleviate one or more of the aforementioned disadvantages of the prior art.

This object is achieved with the thermosetting coating system according to claim 1, the process according to claim 12, the use according to claim 13 and by the protective layer according to claim 14.

Advantageous embodiments of the invention are contained in the sub-claims. The object is particularly achieved with a coating system composed of linear carboxyl-functional polyester resins consisting of bifunctional monomer components, the isophthalic acid making up a maximum of 10 molar parts of all acid components and cross linking agents.

It has been found that, in the case of powder coatings based on linear carboxyl-functional polyesters, i.e. those in which the functionality of the acids and alcohols used is lower than or equal to 2, and polyfunctional epoxides and/or β-hydroxyalkylamides as cross linking agents or hardeners, a clear improvement in the resistance to physical aging can surprisingly be achieved.

The thermosetting coating system according to the invention therefore includes a specific saturated polyester as binder resin, which is particularly distinguished in that it is made up of bifunctional aliphatic and/or cycloaliphatic diols and bifunctional aliphatic and/or cycloaliphatic and/or aromatic dicarboxylic acids.

The binder resin contains at least one linear carboxyl-functional polyester resin composed of a maximum of 10 molar parts of isophthalic acid and at least 90 molar parts of at least one further dicarboxylic acid from the group including aromatic dicarboxylic acids with 8 to 16 carbon atoms, aliphatic dicarboxylic acids with 6 to 22 carbon atoms and cycloaliphatic dicarboxylic acids with 8 to 16 carbon atoms. Terephthalic acid is preferred as aromatic dicarboxylic acid, adipic acid, azelaic acid, sebacic acid and dodecanedicarboxylic acid as aliphatic dicarboxylic acid and cyclohexane dicarboxylic acid as cycloaliphatic dicarboxylic acid. In a preferred embodiment, the dicarboxylic acid component of the polyester resins are made up of 3 to 9 molar parts of aliphatic dicarboxylic acids with at least 6 carbon

atoms, adipic acid being particularly preferred, and/or 3 to 9 molar parts of cycloaliphatic dicarboxylic acid, 1,4-cyclohexane dicarboxylic acid being particularly preferred.

The diols of the polyester resins consist, in a proportion of at least 50 molar parts, of at least one branched aliphatic diol with 4 to 12 carbon atoms and, in a maximum proportion of 50 molar parts, of at least one linear aliphatic diol with 2 to 22 carbon atoms and/or at least one cycloaliphatic diol with 6 to 16 carbon atoms. 2,2-Dimethyl-1,3-propane diol is a preferred branched diol.

Epoxy compounds with at least 2 epoxy groups and/or β-hydroxyalkylamides with at least 2 hydroxyalkylamide groups are suitable as a cross linking component. Preferred monomeric polyepoxy compounds include glycidyl ethers of cyanuric or isocyanuric acid or glycidyl esters of polycarboxylic acids. Terephthalic acid, trimellitic acid or mixtures thereof are preferred. Trisglycidylisocyanurate (TGIC) is particularly preferred. Bis [N,N'-di(β-hydroxyethyl] adipamide (Primid XL 552 made by Rohm and Haas) is particularly suitable as β-hydroxyalkylamide compound.

In a preferred embodiment of the coating system, the polyester resin has an acid number of 15 to 100 [mg KOH/g], a maximum OH number of 10 [mg KOH/g] and a glass transition temperature T_g higher than 45°C.

The additives usual for the production and use of powder coatings can additionally be present in the coating system according to the invention.

These are additives from the group comprising accelerators, pigments, fillers, flow-control and degassing agents, heat-, UV- and/or HALS-stabilizers or tribo additives as well as matting additives such as wax if necessary.

The carboxyl-functional polyester resins according to the invention are produced in a known manner by the common heating of all monomeric components in the presence of conventional esterification catalysts to a temperature of about 250°C and removal of the reaction water formed or by a two stage process in which a hydroxyl-functional polyester is formed in the presence of an excess of polyol in a first stage and the polyester is reacted with one or more polybasic

carboxylic acids or their anhydrides to form a carboxyl-functional polyester in a second stage.

The powder coatings according to the invention are preferably produced in the melt by the common extrusion of all constituents of the formulation at temperatures between 90 and 130°C. The extrudate is then cooled, ground and sieved to a particle size smaller than 100 µm. Other processes for the production of the powder coatings are basically also suitable such as mixing of the constituents of the formulation in solution and subsequent precipitation or removal of the solvent by distillation.

The powder coatings according to the invention are applied by processes which are normal with powder coatings, for example using whirl sintering or electrostatic coating, for example by the corona or the tribo system or by the fluidized bed process.

The powder coatings proposed according to the present invention are sufficiently stable in storage and, after cross linking at 140 to 200°C, have very good flow and mechanical properties. They are also distinguished by good weather- and UVB-resistance and particularly good physical aging resistance. The production and the properties of the carboxyl-functional specific polyester resins and of the powder coatings are described hereinafter by way of examples.

Production of the Carboxyl-functional Polyester Resins

Comparison Example

391.9 g (3.76 mol) of 2,2-dimethyl-1,3-propanol and 27.5 g (0.44 mol) of ethylene glycol are placed in a 2-l-esterification reactor provided with temperature sensor, stirrer, reflux column and distillation bridge, and are melted at 140°C under an inert nitrogen atmosphere which is maintained throughout the entire reaction.

515.5 g (3.10 mol) of terephthalic acid, 110.5 g (0.67 mol) of isophthalic acid and 0.1 g of a Sn-containing esterification catalyst are added while stirring and the internal temperature is raised stepwise to 235°C. The reaction is continued until no more distillate is formed and the acid number is less than 10 mg KOH/g.

73.6 g (0.44 mol) of isophthalic acid and 32.4 g (0.22 mol) of adipic acid are then added, and esterificaion is continued until the desired acid number of about 33 mg KOH/g is attained. Part of this second stage is carried out under reduced pressure (< 100 mbar).

Example 1

As in the comparison example, 352.9 g (3.39 mol) of

2,2-dimethyl-1,3-propane diol and 56.2 g (0.91 mol) of ethylene glycol are melted.

639.3 g (3.85 mol) of terephthalic acid and 0.1 g of a Sn-containing esterification catalyst are added while stirring. The reaction is continued until no more distillate is formed and the acid number is less than 10 mg KOH/g.

56.4 g (0.34 mol) of isophthalic acid and 49.6 g (0.34 mol) of adipic acid are then added and esterification is continued until the desired acid number of about 33 mg KOH/g is attained.

Example 2

347.8 g (3.34 mol) of 2,2-dimethyl-1,3-propane diol and 55.4 g (0.89 mol) of ethylene glycol are melted as in the comparison example.

630 g (3.79 mol) of terephthalic acid and 0.1 g of a Sn-containing esterification catalyst are added while stirring. The reaction is continued until no more distillate is formed and the acid number is less than 10 mg KOH/g.

37.1 g (0.22 mol) of isophthalic acid and 32.6 g (0.22 mol) of adipic acid and 38.4 g (0.22 mol) of cyclohexane dicarboxylic acid are then added, and esterification is continued until the desired acid number of about 33 mg KOH/g is attained.

Example 3

366.7 g (3.52 mol) of 2,2-dimethyl-1,3-propane diol and 55.7 g (0.90 mol) of ethylene glycol are melted as in the comparison example.

633.7 g (3.82 mol) of terephthalic acid and 0.1 g of a Sn-containing esterification catalyst are added while stirring. The reaction is continued until no more distillate is formed and the acid number is less than 10 mg KOH/g.

55.9 g (0.34 mol) of isophthalic acid and 49.2 g (0.34 mol) of adipic acid are then added and esterification is continued until the desired acid number of about 22 mg KOH/g is attained.

Example 4

364.5 g (3.50 mol) of 2,2-dimethyl-1,3-propane diol and 55.4 g (0.89 mol) of ethylene glycol are melted as in the comparison example.

630 g (3.79 mol) of terephthalic acid and 0.1 of a Sn-containing esterification catalyst are added while stirring. The reaction is continued until no more distillate is formed and the acid number is less than 10 mg KOH/g.

37.1 g (0.22 mol) of isophthalic acid, 32.6 g (0.22 mol) of adipic acid and 38.4 g (0.22 mol) of cyclohexane dicarboxylic acid are then added and esterification is continued until the desired acid number of about 22 mg KOH/g is attained.

Example 5

Production of the Powder Coatings

The following formulation was used for all powder coatings described in Table 1. DT 3126 made by Ciba was additionally used as accelerator in the powder coatings in Table 2.

Parts by Weight

948	Binder
15	Resiflow PV 88 [1)]
7	Benzoin
30	Organic Pigment

[1)] Flow-control agent based on polyacrylate, commercial product made by Worlee-Chemie GmbH

The constituents of the formulation are mixed dry in a Henschel mixer at 700 rpm for 30 seconds and are then extruded on a Buss co-kneader (PLK 46) with a barrel temperature of 100°C, cooled screw and a screw speed of 150 rpm. The extrudate is cooled, ground and sieved to smaller than 90 µm.

Painting tests were carried out on aluminium plates (Q-panel AL-36 5005 H 14/08 (0.8 mm)) with a stoving temperature of 180°C and a stoving time of 10 minutes. The paint film thickness is about 90 µm.

Table 1 shows the changes over time in the impact as test criterion for the physical aging after storage in an air conditioned room at 23°C and 50% relative atmospheric humidity.

Table 2 shows the changes over time in the impact as test criterion for physical aging after storage in an oven at 50°C.

TABLE 1 STORAGE IN AIR CONDITIONED ROOM (23°C/50% RELATIVE ATMOSPHERIC HUMIDITY)

POLYESTER	CROSS LINKING AGENT	MIXING RATIO CoPES CROSS LINKING AGENT	REVERSE IMPACT 0 DAYS	1 DAY	2 DAYS	7 DAYS	30 DAYS
CoPES from Comparison Example	Primid XL 552	95 : 5	> 140	120	80	40	< 10
CoPES from Example 1	Primid XL 552	95 : 5	> 140	> 140	> 140	> 140	> 140
CoPES from Example 2	Primid XL 552	95 : 5	> 140	> 140	> 140	> 140	> 140
CoPES from Example 3	Primid XL 552	96.5 : 3.5	> 140	> 140	> 140	> 140	> 140
CoPES from Example 4	Primid XL 552	96.5 : 3.5	> 140	> 140	> 140	> 140	> 140

Reverse impact, ASTM D 2794, ball diameter 5/8" [inch*pound]

TABLE 2 STORAGE IN THE OVEN (50 °C)

Polyester	DT 3126 Percentage of Total Formulation	Cross Linking Agent	Mixing Ratio CoPES/Cross Linking Agent	IMPACT 0 Days	10 Days	20 Days	30 Days	40 Days	50 Days
CoPES from Comparison Example	0.5	TGIC	93 : 7	> 140	100	80	50	< 10	< 10
	3.0	XB 910	91.3 : 8.7	> 140	90	70	30	< 10	< 10
CoPES from Example 1	0.5	TGIC	93 : 7	> 140	> 140	> 140	> 140	> 140	> 140
	3.0	XB 910	91.3 : 8.7	> 140	> 140	> 140	> 140	> 140	> 140
CoPES from Example 2	0.5	TGIC	93 : 7	> 140	> 140	> 140	> 140	> 140	> 140
	3.0	XB 910	91.3 : 8.7	> 140	> 140	> 140	> 140	> 140	> 140
CoPES from Example 3	0.5	TGIC	95 : 5	> 140	> 140	> 140	> 140	> 140	> 140
	3.0	XB 910	93.5 : 6.5	> 140	> 140	> 140	> 140	> 140	> 140
CoPES from Example 4	0.5	TGIC	95 : 5	> 140	> 140	> 140	> 140	> 140	> 140
	3.0	XB 910	93.5 : 6.5	> 140	> 140	> 140	> 140	> 140	> 140

Reverse Impact, ASTM D 2794, ball diameter 5/8" [inch*pound]

The claims defining the invention are as follows:

1. Thermosetting coating system based on carboxyl-functional polyester resins, polyfunctional epoxy compounds and/or β-hydroxyalkylamides as cross linking agents and optionally further conventional additives, the coating system containing as binder resin at least one linear carboxyl-functional polyester resin composed of bifunctional monomer components including a maximum of 10 molar parts of isophthalic acid and at least 90 molar parts of at least one further dicarboxylic acid from the group including aromatic dicarboxylic acids with 8 to 16 carbon atoms, aliphatic dicarboxylic acids with 6 to 22 carbon atoms and cycloaliphatic dicarboxylic acids with 8 to 16 carbon atoms and at least 50 molar parts of at least one branched aliphatic diol with 4 to 12 carbon atoms and a maximum of 50 molar parts of at least one linear aliphatic diol with 2 to 22 carbon atoms and/or at least one cycloaliphatic diol with 6 to 16 carbon atoms.

2. Coating system according to claim 1, wherein the additives are selected from the group including accelerators, pigments, fillers, flow-control agents, degassing agents, heat-stabilizers, UV-stabilizers, HALS-stabilizers, matting additives and tribo additives.

3. Coating system according to either one of claims 1 or 2, wherein the cross linking agent is a monomeric epoxy compound.

4. Coating system according to any one of claims 1 to 3, wherein the cross linking agent is a glycidyl ester of a polycarboxylic acid.

5. Coating system according to any one of claims 1 to 3, wherein the cross linking agent is a glycidyl ether of cyanuric acid or isocyanuric acid.

6. Coating system according to any one of claims 1 to 4, wherein the polycarboxylic acid is trimellitic acid or terephthalic acid.

7. Coating system according to claim 1, wherein the cross linking agent is a β-hydroxyalkylamide with at least 2 hydroxyalkylamide groups.

8. Coating system according to any one of claims 1 to 7, wherein the branched aliphatic diol is 2,2-dimethyl-1,3-

propane diol.

9. Coating system according to any one of claims 1 to 8, wherein the dicarboxylic acid is selected from the group including terephthalic acid, adipic acid, azelaic acid, sebacic acid, dodecanedicarboxylic acid or cyclohexane dicarboxylic acid.

10. Coating system according to any one of the preceding claims, wherein the content of aliphatic dicarboxylic acid and/or of cycloaliphatic dicarboxylic acid as a dicarboxylic acid is 3 to 9 molar parts.

11. Coating system according to any one of the preceding claims, wherein the carboxyl-functional polyester resin has an acid number of 15 to 100 mg KOH/g and a maximum OH number of 10 mg KOH/g and a glass transition temperature higher than 45°C.

12. Process for the production of thermosetting coating systems based on linear carboxyl-functional polyester resins, according to any one of claims 1 to 11 wherein the binder resin, the cross linking agent of which there is at least one from the group including polyfunctional epoxy compounds and β-hydroxyalkylamides and optionally further conventional additives are mixed in the melt at 90 to 130°C, extruded, discharged, granulated, ground and sieved to a particle size of smaller than 100 µm.

13. Use of the thermosetting coating system according to any one of claims 1 to 11 for the production of coverings or protective layers by fluidized bed process or electrostatic spraying.

14. Protective layer ~~which can be~~ produced from the coating system according to any one of claims 1 to 11.

15. A thermosetting coating system substantially as hereinbefore described with reference to any one of Examples 1 to 4.

16. A process according to claim 12 substantially as hereinbefore described with reference to Example 5.

DATED: 9 January, 1995

PHILLIPS ORMONDE & FITZPATRICK

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ABSTRACT

The invention relates to a thermosetting coating system based on carboxyl-functional polyester resins, polyfunctional epoxy compounds and/or β-hydroxyalkylamides and optionally further conventional additives, the coating system containing as binder resin at least one linear carboxyl-functional polyester resin composed of a maximum of 10 molar parts of isophthalic acid and at least 90 molar parts of at least one further dicarboxylic acid from the group including aromatic dicarboxylic acids with 8 to 16 carbon atoms, aliphatic dicarboxylic acids with 6 to 22 carbon atoms and cycloaliphatic dicarboxylic acids with 8 to 16 carbon atoms and at least 50 molar parts of at least one branched aliphatic diol with 4 to 12 carbon atoms and a maximum of 50 molar parts of at least one linear aliphatic diol with 2 to 22 carbon atoms and/or at least one cycloaliphatic diol with 6 to 16 carbon atoms.