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INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(51) International Patent Classification 5 :		(11) International Publication Number:
C09D 123/28, 201/08 C08J 7/04	A1	WO 93/03104
		(43) International Publication Date: 18 February 1993 (18.02.93)

(21) International Application Number: PCT/US92/06286

(22) International Filing Date: 30 July 1992 (30.07.92)

(30) Priority data:
740,352 5 August 1991 (05.08.91) US

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(81) Designated States: CA, JP, KR, European patent (AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LU, MC, NL, SE).

Published
With international search report.

(54) Title: AQUEOUS COATING COMPOSITION

(57) Abstract

Disclosed is a composition comprising: (A) a chlorinated polyolefin, (B) a carboxylic acid functional resin, and (C) water.

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Aqueous Coating Composition

This invention relates to compositions which are useful as primers for enhancing the adhesion of coatings to polyolefin substrates. The compositions of this invention do not contain large quantities of organic solvent. This invention also relates to a process for preparation of these compositions.

The application of paints and other coatings to substrates of polyolefin, such as polypropylene, thermoplastic olefin and polyethylene, is typically difficult because polyolefins are substantially chemically inert.

This problem has previously been overcome by the use of two different approaches which employ adhesion promoting compositions. One approach is to use an adhesion promoting composition as a separate primer coating between the polyolefin substrate and the paint. The primer coating adheres adequately to both the polyolefin and the paint and thereby creates a unitary three component structure with the paint as the outer portion of the structure. Another approach to coating polyolefins has been to use an adhesion promoting composition as an additive along with the paint. This technique is sometimes known in the art as use of a "stir in" adhesion promoter.

Numerous polymeric materials have been investigated as possible components for such adhesion promoters. Chlorinated polyolefins have been found to be very satisfactory as the polymeric component for primer compositions due to their cost and performance. For example, U.S. 3,579,485, U.S. 4,070,421, U.S. 4,966,947, U.S. 4,962,149 and U.S. 4,954,573 disclose chlorinated polyolefins which are entirely satisfactory for the polymeric component of adhesion promoting compositions useful for coating polyolefin substrates.

Even though chlorinated polyolefins have been used to prepare adhesion promoting compositions which are entirely satisfactory, the chlorinated polyolefins had to be formulated with large quantities of an organic solvent, such as xylene or toluene. Use of large quantities of an organic solvent is undesirable because unless elaborate solvent recovery methods are employed application of the primer coating composition results in release of the organic solvent into the atmosphere which can result in both pollution problems and health problems for workers applying the primer coating composition.

We have now discovered an entirely satisfactory water based primer coating composition which preferably does not contain a large quantity of organic solvent.

A composition which is comprised of a chlorinated polyolefin, water and other materials and is reported to be useful as a primer coating for polyolefins is disclosed in WO 9012056.

A composition which is comprised of two types of polymers, water and other materials and is reported to be useful as a primer coating for polyolefins is disclosed in GB 2 272 418.

Broadly, the composition of this invention can be thought of as a three component composition comprising

A) a chlorinated polyolefin having a particular range of molecular weight, softening point and amount of chlorine,

(B) a carboxylic acid functional resin, and

(C) water.

Optionally, an amine, a surfactant and an organic solvent may be present in the composition.

The chlorinated polyolefins useful in this invention can be broadly described as a chlorinated polyolefin having a molecular weight in the range of 9,000 to 150,000, a softening point in the range of 75 to 115°C and an amount of chlorine in the range of 15 to 35 weight percent, based on the weight of the polyolefin. One embodiment of the chlorinated polyolefin useful in this invention is disclosed in U.S. 3,579,485. A chlorinated polyolefin useful in another

embodiment of this invention is the chlorinated polyolefin disclosed in U.S. 3,579,485 reacted with a hydroxyl group containing primary amine to form a chlorinated, imidized polyolefin. These polyolefins are known in the art and are described in U.S. 4,954,573. Thus, since it is within the scope of the invention to use either the chlorinated, non-imidized polyolefin described in U.S. 3,579,485 or the chlorinated, imidized polyolefin described in U.S. 4,954,573 the term "chlorinated polyolefin' is meant to include both of these polymers. In one embodiment of the invention the chlorinated polyolefin can comprise a chlorinated polyolefin having a molecular weight in the range of 100,000 to 150,000, a softening point in the range of 80 to 115°C and an amount of chlorine in the range of 20 to 35 percent, based on the weight of the chlorinated polyolefin. In another embodiment of the invention the chlorinated polyolefin can comprise a chlorinated polyolefin having a molecular eight in the range of 110,000 to 130,000, a softening point in the range of 95 to 105°C and an amount of chlorine in the range of 25 to 30 percent, based on the weight of the chlorinated polyolefin. In a preferred embodiment, the chlorinated polyolefin has a molecular weight in the range of 9,000 to 16,000, a softening point in the range of 80 to 95°C and an amount of chlorine in the range of 18 to 22

percent, based on the weight of the polyolefin. In this invention the molecular weight of the chlorinated polyolefin is average number molecular weight.

In this invention the amount of chlorinated polyolefin in the composition is in the range of 70 to 30 weight percent, preferably in the range of 70 to 40 weight percent, and more preferably in the range of 70 to 45 weight percent, based on the combined weight of the chlorinated polyolefin and the carboxylic acid functional resin.

The carboxylic acid functional resins useful in this invention can comprise any polymer which has a sufficiently high molecular weight to form an admixture with the chlorinated polyolefin and also has sufficient carboxylic acid functionality to be compatible with the chlorinated polyolefin in the sense it will combine with the chlorinated polyolefin and form the primer coating composition of this invention.

According to one theory the carboxylic acid functional resin performs two functions. First, the resin serves as the medium in which the chlorinated polyolefin can be formed into an admixture having a fluid homogenous character. Second, the carboxylic acid functionality of the resin, through neutralization, provides a mechanism for the creating the admixture of the two polymers and the water. Thus, important

characteristics of the resin are that the resin be compatible enough with the chlorinated polyolefin to make a fluid homogenous mixture and that the resin have acid functionality to allow dispersion through amine neutralization of the acid group.

Preferably, the carboxylic acid functional resin is an alkyd resin, a polyester resin or an acrylic resin. More preferably the carboxylic acid functional resin is either an alkyd resin or an acrylic resin.

The alkyd resins useful in this invention are well known in the art and are polyesterification products of polyhydric alcohols and poly-functional acids where part of the acid component may be a long chain fatty acid from vegetable oil. Examples of poly-functional acids include phthalic anhydride, adipic acid, maleic anhydride, isophthalic acid, sebacic acid, azelaic acid, terephthalic acid, trimellitic anhydride, linoleic acid, linolenic acid, benzoic acid, hexahydrophthalic anhydride, tetrahydrophthalic anhydride, 1,4-cyclohexanedicarboxylic acid or fumaric acid. Examples of polyhydric alcohols include glycerol, trimethylolethane, trimethylolpropane, diethylene glycol, neopentyl glycol, 1,4-cyclohexanedimethanol, 2,2,4-trimethyl-1,3-pentanediol, 1,6-hexanediol, 1,2-propylene glycol, 1,2-propanediol, ethylene glycol, butylene glycol, dipropylene glycol, and

pentaerythritol. The oil which is used to modify the alkyd resin may come from the group of oils which includes tung, linseed, soya, safflower, castor, corn, cotton seed, peralla, sesame, soybean, cocoanut, dehydrated castor and tall oils. The amount of oil modification in resins useful in the invention can be in the range of <45% (short oil alkyds) or in the range of 45 to 65% (medium oil alkyds).

The alkyd resins which are useful in the invention can have a number average molecular weight in the range of 300 to 30,000 grams per mole and an acid number greater than 20. More preferably the acid number of the alkyd resin should be in the range 25 to 150.

Most preferably, the alkyd resin is a medium oil alkyd having 70 percent non-volatile content, a Gardner viscosity in the range of Z to Z2 and an acid value in the range of 50 to 59.

The acrylic resins useful in this invention are well known in the art and are addition polymers which are the reaction products of acrylic monomers which come from a large number of acrylic and methacrylic acids and esters. Examples of acids and esters which can be used include acrylic acid, methacrylic acid, styrene, methyl methacrylate, ethyl acrylate, butyl acrylate, methyl acrylate, ethyl methacrylate, butyl methacrylate, 2-hydroxyethyl acrylate, 2-hydroxyethyl methacrylate,

butyl acrylate, propyl methacrylate, lauryl methacrylate, 2-hydroypropyl methacrylate, 2-ethyl hexyl methacrylate, hexyl methacrylate, cyclohexyl methacrylate, ethoxyethyl acrylate, ethylhexyl acrylate, cyclohexyl acrylate, hexyl acrylate, butadiene, vinyl toluene, vinyl pyrrolidine and 2-hydroxypropyl acrylate.

The acrylic resins useful in this invention have a number average molecular weight in the range of 400 to 40,000 grams per mole and an acid number greater than 20. More preferably the acrylic resin has an acid number in the range of 25 to 150.

In a preferred embodiment, the acrylic resin is a solid acrylic oligomer having an acid value in the range of 220 to 250, a ring and ball softening point in the range of 100 to 120°C and an average number molecular weight in the range of 2000 to 3000.

The polyester resins useful in this invention are well known in the art and are condensation polymers which are the polyesterification product of polyfunctional carboxylic acid and polyfunctional alcohols. Examples of polyfunctional carboxylic acids include phthalic anhydride, isophthalic acid, cyclohexanedicarboxylic acid, adipic acid, azelaic acid, maleic anhydride, sebacic acid, terephthalic acid, trimellitic anhydride, linoleic acid, linolenic acid, benzoic acid, hexahydrophthalic anhydride,

tetrahydrophthalic anhydride, fumaric acid, 1,4-cyclohexanedicarboxylic acid and succinic acid. Examples of polyfunctional alcohols include ethylene glycol, neopentyl glycol, diethylene glycol, 1,6-hexanediol, cyclohexanedimethanol, glycerol, trimethylolethane, trimethylolpropane, butylene glycol, 1,4-cyclohexanedimethanol, 1,2-propylene glycol, 1,2-propanediol, dipropylene glycol, pentaerythritol and 2,2,4-trimethyl-1,3-pentanediol.

Polyesters which are useful in the present invention have a number average molecular weight in the range of 100 to 10,000 grams per mole. Useful polyester resins are those which have a acid number greater than 20. More preferable polyester resins are those which have an acid number in the range 25-150.

The amount of the carboxylic acid functional resin is in the range of 30 to 70 weight percent, preferably in the range of 30 to 60, and more preferably in the range of 30 to 55 weight percent, based on the combined weight of the chlorinated polyolefin and the carboxylic acid functional resin.

Combinations of particular chlorinated polyolefins and particular carboxylic acid functional resins represent a particularly preferred embodiment of this invention. For example, when the preferred chlorinated polyolefin is the non-imidized material, use of the

above described medium oil alkyl having 70 percent non-volatile content, a Gardner viscosity in the range of Z to Z2 and an acid value in the range of 50 to 59 is a particularly preferred embodiment of the invention. Additionally, when the preferred chlorinated polyolefin in the imidized material, use of the above described solid acrylic oligomer having an acid value in the range of 220 to 250, a ring and ball softening point in the range of 100 to 120°C and an average number molecular weight in the range of 2000 to 3000 represents a particularly preferred embodiment of this invention.

The compositions of this invention contain water. The amount of water can vary widely depending on numerous factors, such as the needs of the manufacturer, transportation efficiencies and the needs of the particular customer. An important property of the compositions of this invention is that these compositions can be manufactured using less water than would be typically be required to apply the compositions as a coating and water can be subsequently added by the customer at a later time to prepare a coating composition. This can be done by simply pouring additional water into the composition prepared by the manufacturer without having to consider in detail any particular temperature, pressure or time used to prepare the diluted composition. Thus the manufacturer can

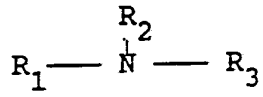
prepare a "concentrated" product which is low in water and ship the concentrated product to the customer without the necessity of incurring the additional cost of also shipping additional amount to customer. A "concentrated" product is also often desired if the adhesion promoter composition is to be used as an additive to the paint in order to prevent excessive dilution of the paint.

Even though the amount of water can vary widely and there is no upper limit on the amount of water there is a lower limit on the amount of water because there must at least be sufficient water in the composition to result in the formation of an admixture of the four components. Although the precise amount of water necessary to form the required admixture can vary, generally there must be at least 40 weight % water in the composition, based on the weight of the total composition, in order to form an admixture with pourable viscosity characteristics.

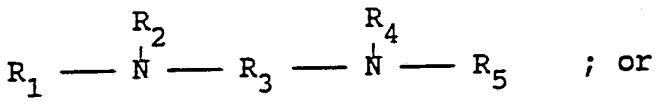
The compositions of this invention can optionally contain an amine. Although the amine can be aromatic or aliphatic, aliphatic amines are preferred. In a preferred embodiment the aliphatic amine can be either a primary, secondary or tertiary amine and optionally contains other oxygen containing functional groups. The

amines useful in this invention generally have a molecular weight in the range of 20 to 200.

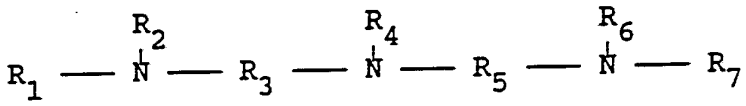
A highly preferred group of amines are primary, secondary and tertiary aliphatic amines having a functionality of 1 to 3 which can be generally represented by the general formulae:



; or



; or



wherein $R_1 = R_7$ are independently selected from H or straight or branched chain alkyl, hydroxyalkyl, or alkoxylalkyl groups of 1 to 20 carbon atoms; $R_1 - R_7$ can additionally include a substituted alkyl group, i.e., where one or more of the carbons in the radical is replaced with or has substituted thereon another functionality, e.g., an amine, ether, hydroxy or -mercapto moiety, e.g., tris-(3-aminopropyl) amine.

Another group of highly preferred amines within the above classes are those primary, secondary or tertiary aliphatic amines of the above Formulae in which $R_1 - R_7$ is specifically substituted with or contains one or more hydroxyl (-OH) functionalities.

Another group of preferred amines can be represented by the formulae:

$$R_8—(NH_2)_n \quad \text{or} \quad R_9—(\overset{R_9}{\overset{|}{N}}H)_n \quad \text{or} \quad R_8—(R_{10}—\overset{R_9}{\overset{|}{N}}—R_{11})$$

wherein n is 1 or 2 and R_8, R_9, R_{10} and R_{11} are independently selected from straight or branched chain alkyl, hydroxyalkyl or alkoxyalkyl groups of 1 to 20 carbon atoms. These chains may also be substituted with another functionality as described above.

Yet another group which comprise amines preferred in the practice of the present invention are primary, secondary and tertiary aliphatic amines with an amine functionality of 1–3 which also contain one or more ether or alkoxy linkages. Such materials are sometimes referred to as poly(oxyalkylene)diamines. Ethoxylated or propoxylated materials are particularly preferred.

Exemplary amines preferred for use in the present invention include:

2–amino–1–butanol;

4–amino–1–butanol;

2–aminoethanethiol;

2–aminoheptane;

2–amino–1–hexanol;

6–amino–1–hexanol;

allylamine;

2-amino-3-methyl-1-butanol;

2-amino-2-methyl-1,3-propanediol;

2-amino-2-methyl-1-propanol;

2-amino-1-pentanol;

5-amino-1-pentanol;

3-amino-1-propanol;

ammonium hydroxide

amylamine;

butylamine;

N,N'-bis(2-aminoethyl)-1,3-propanediamine;

N,N'-bis(3-aminopropyl)-1,3-propanediamine;

1,3-bis(dimethylamino)-2-propanol;

1-[N,N-bis(2-hydroxyethyl)amino]-2-propanol;

N,N'-bis(2-hydroxyethyl)ethylenediamine;

decylamine;

1,4-diaminobutane;

1,10-diaminodecane;

1,12-diaminododecane;

1,7-diaminoheptane;

1,3-diamino-2-hydroxypropane;

3,3'-diamino-N-methyldipropylamine;

1,2-diamino-2-methylpropane;

1,9-diaminononane;

1,8-diaminooctane;

1,5-diaminopentane;

1,2-diaminopropane;

1,3–diaminopropane;

dibutylamine;

3–(dibutylamino)propylamine;

diethanolamine;

diethylamine;

5–diethylamino–2–pentanol;

3–(diethylamino)–1,2–propanediol;

1–diethylamino–2–propanol;

3–diethylamino–1–propanol;

3–diethylaminopropylamine;

diethylenetriamine;

N,N–diethylethanolamine;

N,N–diethylethylenediamine;

N,N–diethylmethylamine;

N,N'–diethyl–1,3–propanediamine;

diisobutylamine;

diisopropanolamine;

diisopropylamine;

2–(diisopropylamino)ethanol;

3–diisopropylamino–1,2–propanediol;

N,N–diisopropylethylamine;

1–dimethylamino–2–propanol;

2–dimethylaminoethanol

3–dimethylamino–1–propanol;

3–dimethylaminopropylamine;

1,3–dimethylbutylamine;

3,3–dimethylbutylamine;

N,N–dimethylethanolamine;

N,N–dimethylethylamine;

N,N–dimethylethylenediamine;

N,N–dimethyl–N'–ethylethylenediamine;

N,N'–dimethyl–1–,6–hexanediamine;

2,5–dimethyl–2,5–hexanediamine;

1,5–dimethylhexylamine;

2,2–dimethyl–1,3–propanediamine;

(±)–1,2–dimethylpropylamine;

dipropylamine;

dodecylamine;

ethanolamine;

3–ethoxypropylamine;

ethylamine;

2–(ethylamino)ethanol;

N–ethylbutylamine;

2–ethylbutylamine;

N–ethyldiethanolamine;

ethylenediamine;

hexamethylenediamine;

1,6–hexanediamine;

hexylamine;

isoamylamine;

isopropylamine;

N–isopropylethylenediamine;

N'-isopropyl-2-methyl-1,2-propanediamine;

N,N,N',N'-tetramethyl-1,4-butanediamine;

N,N,N',N'-tetramethyldiaminomethane;

N,N,N',N'-tetramethylethylenediamine;

N,N,N',N'-tetramethyl-1,6-hexanediamine;

N,N,N',N'-tetramethyl-1,3-propane-diamine;

N,N,2,2-tetramethyl-1,3-propanediamine;

tributylamine;

tridecyamine;

triethanolamine;

triethylamine;

triisooctylamine;

triisopropyanolamine;

trimethylamine;

methylamine;

2-(methylamino)ethanol;

N-Methylbutylamine;

1-methylbutylamine;

2-methylbutylamine;

N-methyldiethanolamine;

N-methylethylenediamine;

N-methyl-1,3-propanediamine;

morpholine

nonylamine;

octylamine;

teri-octylamine;

propylamine;

2-(propylamino)ethanol;

1-tetradecylamine; and

tris(3-aminopropyl)amine. Mixtures of such materials may also be employed.

Preferred amines are ammonium hydroxide, dimethylethanolamine, triethylamine, diethanolamine, ethanolamine, dimethylamine, diethylamine, piperazine, morpholine and 2-amino-2-methyl-1-propanol. When the carboxylic acid functional resin is an alkyd resin a particularly preferred amine is ammonium hydroxide. When the carboxylic acid functional resin is an acrylic a preferred amine is dimethylethanolamine.

The amount of amine can be in the range of 0 to 20 weight percent, preferably in the range of 10 to 20 weight percent, based on the combined weights of the chlorinated polyolefin and carboxylic acid functional resin.

The surfactants useful in this invention include both non-ionic and cationic surfactants. By the term "non-ionic surfactant" we mean a surfactant which contains no positively or negatively charged functional groups. By the term "cationic surfactant" we mean a surfactant which ionizes in water to produce a large positively charged ion and a small negatively charged ion.

In this invention the non-ionic surfactants can have a molecular weight of up to 500 or even higher and can include polymeric materials. The non-ionic surfactants include materials which contain groups of varying polarity whereby one part of the molecule is hydrophilic and the other part of the molecule is hydrophobic. Examples of such materials include polyethyleneoxy polyols and ethoxylated alkyl phenols. Particularly preferred classes of non-ionic surfactants include alkyl phenoxy poly(ethyleneoxy) alcohols, primary ethoxylated alcohols and secondary ethoxylated alcohols. Preferably the non-ionic surfactant is an alkyl phenoxy poly(ethyleneoxy) alcohol having 22 to 215 carbon atoms.

The cationic surfactants useful in this invention have a molecular weight in the range of 100 to 1000 and can be broadly be described as alkoxylated amines. Preferably the cationic surfactant is an alkoxylated fatty amine or an akloxylated tallow amine.

The amount of surfactant is broadly in the range of 1 to 30 weight percent, based on the combined weight of the chlorinated polyolefins and carboxylic acid functional resin.

The compositions can contain other materials in major amounts. For example, the compositions of this invention can contain materials typically used in the

paint industry to prepare paint formulations, such as thickeners, wetting agents and flow aids, pigments, resins and solvents.

The compositions can contain organic solvents as long as the amount of organic solvent is not large. Thus, the compositions of this invention preferably contain less than 50%, more preferably less than 25% organic solvent based on the weight of the composition.

The compositions of this invention can be used as aa adhesion promoting composition according to two different approaches. Selection of the preferred approach depends on numerous factors, such as the specific characteristics of the paint, the particular substrate to be painted, the nature and extent of the other materials to be used in the paint formulation and other factors. In accordance with one approach, the coating compositions of this invention can be used as a separate primer coat by preferably diluting the manufactured composition with water, adding any desired additives and then applying the diluted composition to a polyolefin substrate to form a coating using conventional means, such as spraying, brushing or dipping. After the composition has been applied as a coating and the coating has dried a paint is applied over the primer coating. The primer coating adheres to both the polyolefin and the paint and thereby creates a

unitary three component structure with the paint as the outer portion of the structure. In accordance with the other approach, the compositions of this invention can be used as a so-called "stir-in" paint additive. In this approach the composition is added admixed with the paint and the resulting admixture is applied to the polyolefin substrate to form a single coating which adheres to the polyolefin substrate.

The compositions of this invention are admixtures which have a particle size suitably small to make the admixtures useful in coating embodiments. Therefore, the compositions of this invention include not only what some authorities call "emulsions" and "dispersions" but include as well as all other physical forms in which the various components can be become admixed. For example, some authorities characterize water containing admixtures wherein the particle size in the range of 0.1 to 10 microns as a "emulsions". Other authorities regard water containing admixtures wherein the particle size is greater than 10 microns as a "dispersion". While both of these types of admixtures are within the scope of this invention, the invention is not limited to these or any other kind of particular admixture and includes all possible types of admixtures regardless of physical form as long as the particle size is small

enough for the admixtures to have utility in coating applications.

While the compositions of this invention are particularly useful as adhesion promoters to enhance the adhesion of paints to polyolefin substrates it is within the scope of the invention for the compositions to be used by themselves as a paint to form a final protective coating which not only protects the substrate but also is decorative as a result of the addition of pigments.

The compositions of this invention are prepared by a novel process comprised of two steps. First an admixture of the chlorinated polyolefin and carboxylic acid functional resin is prepared and then an admixture is prepared composed of the admixture of the polyolefin and resin and water.

The chlorinated polyolefin and carboxylic acid functional resin can be prepared into an admixture by methods well known in the art by preparing admixtures of polymeric materials. If the chlorinated polyolefin and carboxylic acid functional resins are both solids they can be admixed in a double screw extruder or can be powdered and the mixed powders extruded in a single screw extruder. Additionally the admixture can be prepared by combining the resin and chlorinated polyolefin on a hot roll mill such as a hot three roll mill. The admixture can be cooled and then pulverized

to a powder prior to addition of the other materials.

When the chlorinated polyolefin is a solid and the carboxylic acid functional resin is a liquid or solution, the admixture can be prepared by heating the mixture above the softening point of the chlorinated polyolefin and stirring the chlorinated polyolefin and the resin together. The chlorinated polyolefin can be mixed with the resin by applying high-shear mixing.

The admixture of water, and optionally an amine and surfactant, and the admixture of the chlorinated polyolefin and carboxylic acid functional resin can be prepared by methods well known in the art for preparing admixtures of polymers and water. According to one method the admixture is prepared by subjecting the materials to high shear in a vessel equipped with a means to create high shear, such as a stirrer. According to another method, a heated admixture of the chlorinated polyolefin and the carboxylic acid functional resin is combined with a heated admixture of water and amine using agitation techniques well known in the art.

In the following examples compositions of the invention were prepared from a chlorinated polyolefin, a carboxylic acid functional resin, water and optionally an amine, a surfactant and an organic solvent. In some examples, the chlorinated polyolefin and the carboxylic

acid functional resin were introduced into a vessel equipped with means to agitate the materials. Agitation was started and the contents of the vessel were heated at a specific temperature for a specific period of time. In other examples the chlorinated polyolefin and the carboxylic acid functional resin were combined into the admixture using a standard three roll mill. Water and optionally an amine, a surfactant and an organic solvent were then added and an admixture formed by agitation with high shear. Storage stability tests of selected compositions were conducted by visual inspection in glass jars after 2 to 8 weeks at 25°C. Paint adhesion tests of selected compositions were conducted in accordance with ASTM D 3359-83, Method A. Paint adhesion tests were conducted by applying the composition to polypropylene and to a polyolefin which had been modified with an elastomeric polymer. These materials are generally designated in the trade as "thermoplastic olefins" and are abbreviated "TPO". The composition was applied by spraying and then dried at room temperature. A commercially available automotive paint system, was applied and baked 30 minutes at 250°F.

Example 1

This example illustrates preparation of a composition of the invention using a particular

chlorinated polyolefin, a particular carboxylic acid functional resin, water and an amine.

An admixture was prepared composed of 50 grams of a chlorinated, non-imidized polyolefin which has a molecular weight in the range of 9,000 to 16,000, a softening point in the range of 80 to 95°C and an amount of chlorine in the range of 18 to 22 percent, based on the weight of the polyolefin, and 50 grams of a carboxylic acid functional resin which is a medium oil alkyd resin (70% solids) having an acid number in the range of 50 to 59. The admixture was heated for 2 hours at 105°C. 10 grams of ammonium hydroxide (28%) and 200 grams of water were then added and an admixture formed by high shear agitation for 10 minutes. The resultant composition was diluted with sufficient additional water to result in a primer coating composition typical of that used in the trade and paint adhesion tests were conducted with the following results.

Polypropylene	100% adhesion
TPO	100% adhesion

Example 2

This example illustrates preparation of a composition of the invention using different amounts of the same materials used in Example 1.

An admixture was prepared composed of 30 grams polyolefin and 70 grams of alkyd resin. The admixture was heated for 1 hour at 121°C. 8 grams of ammonium hydroxide (28%) and 150 grams of water were then added and an admixture formed by high shear agitation for 10 minutes. The result of a stability test at 25°C for 8 weeks was slight settling. The composition was diluted with sufficient additional water to result in a primer coating composition typical of that used in the trade and paint adhesion tests were conducted with the following results.

Polypropylene	100% adhesion
TPO	100% adhesion

Example 3

This example illustrates preparation of a composition of the invention using a different amine than used in Example 1.

An admixture was prepared composed of 50 grams polyolefin and 50 grams of alkyd resin. The admixture was heated for 2 hours at 121°C. 4 grams of dimethylethanolamine and 200 grams of water were then added and an admixture formed by high shear agitation for 10 minutes. The result of a stability test at 25°C for 8 weeks was no change. The composition was diluted with sufficient additional water to result in a primer

coating composition typical of that used in the trade and paint adhesion tests were conducted with the following results.

Polypropylene	100% adhesion
TPO	100% adhesion

Example 4

This example illustrates preparation of a composition of the invention using a different amount of the same materials used in Example 3.

An admixture was prepared composed of 70 grams polyolefin and 30 grams of alkyd resin. The admixture was heated for 12 hours at 121°C. 2 grams of dimethylethanolamine and 200 grams of water were then added and an admixture formed by high shear agitation for 10 minutes. The result of a stability test at 25°C for 8 weeks was slight settling. The composition was diluted with sufficient additional water to result in a primer coating composition typical of that used in the trade and paint adhesion tests were conducted with the following results.

Polypropylene	100% adhesion
TPO	100% adhesion

Example 5

This example illustrates preparation of a composition of the invention using different amounts of the same materials used in Example 1.

An admixture was prepared composed of 25 grams polyolefin and 25 grams of alkyd resin. The admixture was heated for 2 hours at 121°C. The 10g ammonium hydroxide (28%) and 100g water were then added and an admixture formed by high shear agitation for 10 minutes. The result of a stability test at 25°C for 6 weeks was slight settling. The composition was diluted with sufficient additional water to result in a primer coating composition typical of that used in the trade and paint adhesion tests were conducted with the following results.

Polypropylene	100% adhesion
TPO	100% adhesion

Example 6

This example illustrates preparation of a composition of the invention using different amounts of the same materials used in Example 5.

An admixture was prepared composed of 35 grams polyolefin and 15 grams of alkyd resin. The admixture was heated for 2 hours at 121°C. 5 grams of ammonium hydroxide (28%) and 100 grams of water were then added

and an admixture formed by high shear agitation for 10 minutes. The result of a stability test at 25°C for 6 weeks was heavy settling. The composition was diluted with sufficient additional water to result in a primer coating composition typical of that used in the trade and paint adhesion tests were conducted with the following results.

Polypropylene	20% adhesion
TPO	100% adhesion

Example 7

This example illustrates preparation of a composition of the invention using different amounts of the same materials used in Example 5.

An admixture was prepared composed of 25 grams polyolefin and 25 grams of alkyd resin. The admixture was heated for 2 hours at 121°C. 1 gram of ammonium hydroxide (28°) and 100 grams of water were then added and an admixture formed by high shear agitation for 10 minutes. The result of a stability test at 25°C for 6 weeks was heavy settling. The composition was diluted with sufficient additional water to result in a primer coating composition typical of that used in the trade and paint adhesion tests were conducted with the following results.

Polypropylene	0% adhesion
TPO	40% adhesion

Example 8

This example illustrates preparation of a composition of the invention using a different carboxylic acid functional resin than used in Example 7.

An admixture was prepared composed of 50 grams polyolefin and 50 grams of a chain stopped alkyd resin (70% solids) with 35 acid number. The admixture was heated for 10 minutes at 121°C. 8 grams of ammonium hydroxide (28%) and 100 grams of water were then added and an admixture formed by high shear agitation for 10 minutes. The result of a stability test at 25°C for 8 weeks was heavy settling. The composition was diluted with sufficient additional water to result in a primer coating composition typical of that used in the trade and paint adhesion tests were conducted with the following results.

Polypropylene	100% adhesion
TPO	100% adhesion

Example 9

This example illustrates preparation of a composition of the invention using a different carboxylic acid functional resin than used in Example 8.

An admixture was prepared composed of 30 grams polyolefin and 70 grams of an acrylic resin (65% solids) with 90 acid number. The admixture was heated for 2 hours at 121°C. 6 grams of ammonium hydroxide (28%) and 100 grams of water were then added and an admixture formed by high shear agitation for 10 minutes. The composition was diluted with sufficient additional water to result in a primer coating composition typical of that used in the trade and paint adhesion tests were conducted with the following results.

Polypropylene	100% adhesion
TPO	100% adhesion

Example 10

This example illustrates preparation of a composition of the invention using a chlorinated polyolefin, a carboxylic acid functional resin, an amine, a surfactant and water.

An admixture was prepared composed of 50 grams polyolefin and 50 grams of an alkyd resin with 85 acid number. The admixture was heated for 2 hours at 121°C. 4 grams of dimethylethanolamine, 1 gram of a nonyl phenoxy poly(ethyleneoxy) alcohol having an HBL value in the range of 13 to 14 and 200 grams of water were then added and an admixture formed by high shear agitation for 10 minutes. The result of a stability test for 2

weeks and 25°C was heavy settling. The composition was diluted with sufficient additional water to result in a primer coating composition typical of that used in the trade and paint adhesion tests were conducted with the following results.

Polypropylene	100% adhesion
TPO	100% adhesion

Example 11

This example illustrates preparation of a composition of the invention similar to that prepared in Example 10 except a different carboxylic acid functional resin was used.

An admixture was prepared composed of 50 grams polyolefin and 50 grams of a acrylic resin (60% solids) with 90 acid number. The admixture was heated for 2 hours at 121°C. 4 grams of dimethylethanolamine, 2 grams of an alkyl phenoxy poly(ethyleneoxy) alcohol and 200 grams of water were then added and an admixture formed by high shear agitation for 10 minutes. The result of a stability test for 2 weeks and 25°C was no change. The composition was diluted with sufficient additional water to result in a primer coating composition typical of that used in the trade and paint adhesion tests were conducted with the following results.

Polypropylene	100% adhesion
TPO	100% adhesion

Example 12

This example illustrates preparation of a composition of the invention similar to that prepared in Example 10 except a different carboxylic acid functional resin was used.

An admixture was prepared by subjecting to high shear 50 grams polyolefin and 50 grams of an acrylic resin (70% solids) with acid number 45. 4 grams of dimethylethanolamine, 2 grams of an alkyl phenoxy poly(ethyleneoxy) alcohol and 200 grams of water were then added and an admixture formed by high shear agitation for 10 minutes. The result of a stability test for 2 weeks and 25°C was heavy settling. The composition was diluted with sufficient additional water to result in a primer coating composition typical of that used in the trade and paint adhesion tests were conducted with the following results.

Polypropylene	20% adhesion
TPO	100% adhesion

Example 13

This example illustrates preparation of a composition of the invention composed of a chlorinated

polyolefin, a carboxylic acid functional resin, an amine and water.

An admixture was prepared by subjecting to high shear 50 grams polyolefin and 50 grams of an acrylic resin (80% solids) with acid number 35. 4 grams of dimethylethanolamine and 200 grams of water were then added and an admixture formed by high shear agitation for 10 minutes. The result of a stability test for 2 weeks and 25°C was heavy settling. The composition was diluted with sufficient additional water to result in a primer coating composition typical of that used in the trade and paint adhesion tests were conducted with the following results.

Polypropylene	20% adhesion
TPO	100% adhesion

Example 14

This example illustrates preparation of a composition of the invention similar to Example 12 except that a different carboxylic acid functional resin was used.

An admixture was prepared by subjecting to high shear 50 grams polyolefin and 50 grams of an alkyd resin (80% solids) with acid number 34. 4 grams of dimethylethanolamine, 4 grams of an alkyl phenoxy poly(ethylenoxy) alcohol and 250 grams of water were

then added and an admixture formed by high shear agitation for 10 minutes. The result of a stability test for 2 weeks and 25°C was heavy settling. The composition was diluted with sufficient additional water to result in a primer coating composition typical of that used in the trade and paint adhesion tests were conducted with the following results.

Polypropylene	0% adhesion
TPO	100% adhesion

Example 15

This example illustrates preparation of a composition of the invention similar to Example 14 except that a different carboxylic acid functional resin was used.

An admixture was prepared by subjecting to high shear 50 grams polyolefin and 50 grams of a saturated polyester resin (75% solids) with acid number 68. 4 grams of dimethylethanolamine, 2 grams of an alkyl phenoxy poly(ethyleneoxy) alcohol and 200 grams of water were then added and an admixture formed by high shear agitation for 10 minutes. The result of a stability test for 2 weeks and 25°C was reversible phase separation.

Example 16

This example illustrates preparation of a composition of the invention similar to Example 15 except that a different carboxylic acid functional resin was used.

An admixture was prepared by subjecting to high shear 50 grams polyolefin and 50 grams of an saturated polyester resin (85% solids in xylene) with an acid number in the range of 24 to 34. 4 grams of dimethylethanolamine, 2 grams of an alkyl phenoxy poly(ethyleneoxy) alcohol and 200 grams of water were then added and an admixture formed by high shear agitation for 10 minutes. The result of a stability test for 2 weeks and 25°C was reversible phase separation.

Example 17

This example illustrates preparation of a composition of the invention composed of a chlorinated polyolefin, a carboxylic acid functional resin, an amine, a surfactant, an organic solvent and water.

An admixture was prepared by subjecting to high shear 50 grams polyolefin and 50 grams of an acrylic modified polyester resin (50% solids) with acid value in the range of 58 to 66. 4 grams of dimethylethanolamine, 4 grams of an alkyl phenoxy poly(ethyleneoxy) alcohol, 4

grams of xylene and 200 grams of water were then added and an admixture formed by high shear agitation for 10 minutes. The result of a stability test for 2 weeks and 25°C was slight settling. The composition was diluted with sufficient additional water to result in a primer coating composition typical of that used in the trade and paint adhesion tests were conducted with the following results.

Polypropylene	100% adhesion
TPO	100% adhesion

Example 18

This example illustrates preparation of a composition of the invention similar to Example 16 except that a different carboxylic acid functional resin was used.

An admixture was prepared by subjecting to high shear 50 grams polyolefin and 50 grams of an acrylic resin (70% solids) with acid number. 4 grams of dimethylethanolamine, 2 grams of an alkyl phenoxy poly(ethyleneoxy) alcohol and 200 grams of water were then added and an admixture formed by high shear agitation for 10 minutes. The result of a stability test for 2 weeks and 25°C was heavy settling. The composition was diluted with sufficient additional water to result in a primer coating composition typical of

that used in the trade and paint adhesion tests were conducted with the following results.

Polypropylene	90% adhesion
TPO	100% adhesion

Example 19

This example illustrates preparation of a composition of the invention composed of a chlorinated polyolefin, a carboxylic acid functional resin, an amine, a surfactant, an organic solvent and water.

An admixture was prepared by combining on a 3 roll mill 13 grams of an imidized chlorinated polyolefin which has a molecular weight in the range of 9,000 to 16,000, a softening point in the range of 80 to 95°C and an amount of chlorine in the range of 18 to 22 percent, based on the weight of the polyolefin, 13 grams of a solid acrylic resin with acid number 230. The admixture was cooled and pulverized into granules and the granules heated at 121°C for 1 hour. 15 grams of ammonium hydroxide, 1 gram of a non-ionic nonyl phenoxy(ethylene oxy) alcohol with HLB of 18.6, 10 grams of xylene and 150 grams of water were then added and an admixture formed by high shear agitation for 20 minutes. The composition was diluted with sufficient additional water to result in a primer coating composition typical of

that used in the trade and paint adhesion tests were conducted with the following results.

Polypropylene	100% adhesion
TPO	100% adhesion

Example 20

This example illustrates preparation of a composition of the invention composed of a chlorinated polyolefin, a carboxylic acid functional resin, an amine, an organic solvent and water.

An admixture was prepared by combining at 66°C 50 grams of a chlorinated polyolefin having a molecular weight in the range of 100,000 to 150,000, a softening point in the range of 80 to 115°C and an amount of chlorine in the range of 20 to 35 percent, based on the weight of the polyolefin, 50 grams of a carboxylic acid functional resin which is an alkyd resin (70% solids) having an acid number in the range of 50 to 59, and 50 grams of xylene. 10 grams of dimethylethanolamine and 250 grams of water were then added and an admixture formed by high shear agitation for 10 minutes. The result of a stability test for 3 weeks and 25°C was slight settling. The composition was diluted with sufficient additional water to result in a primer coating composition typical of that used in the trade

and paint adhesion tests were conducted with the following results.

Polypropylene	0% adhesion
TPO	70% adhesion

Example 21

This example illustrates preparation of a composition of the invention composed of a chlorinated polyolefin, a carboxylic acid functional resin, an amine, an organic solvent and water.

An admixture was prepared by combining at 66°C 50 grams of a chlorinated polyolefin having a molecular weight in the range of 110,000 to 130,000, a softening point in the range of 95 to 105°C and an amount of chlorine in the range of 25 to 30 percent, based on the weight of the polyolefin, 50 grams of a carboxylic acid functional resin which is an alkyd resin (70% solids) having an acid number in the range of 50 to 59, and 50 grams of xylene. 10 grams of dimethylethanolamine and 250 grams of water were then added and an admixture formed by high shear agitation for 10 minutes. The result of a stability test for 3 weeks and 25°C was no change. The composition was diluted with sufficient additional water to result in a primer coating composition typical of that used in the trade and paint

adhesion tests were conducted with the following results.

Polypropylene	0% adhesion
TPO	100% adhesion

CLAIMS

1. A composition comprising

(A) from 70 to 30 weight percent, based on the combined weight of components (A) and (B), of a chlorinated polyolefin having a molecular weight in the range of 9,000 to 150,000, a softening point in the range of 75 to 115°C, and an amount of chlorine in the range of 15 to 35 weight percent, based on the weight of the polyolefin, and

(B) from 30 to 70 weight percent, based on the combined weight of components (A) and (B), of a carboxylic acid functional resin,

(C) from 0 to 20 weight percent, based on the combined weight of components (A) and (B), of a primary, secondary or tertiary amine having a molecular weight in the range of 20 to 200, and

(D) water.

2. The composition claim 1 wherein the chlorinated polyolefin has a molecular weight in the range of 9,000 to 16,000, a softening point in the range of 80 to 95°C and an amount of chlorine in the range of 18 to 22 percent.

3. The composition of claim 1 wherein the chlorinated polyolefin is a chlorinated, imidized polyolefin has a molecular weight in the range of 9,000 to 16,000, a softening point in the range of 80 to 95°C and an amount of chlorine in the range of 18 to 22 percent.

4. The composition of claim 1 wherein the carboxylic acid functional resin in selected from the group consisting of alkyd resins, polyester resins and acrylic resins.

5. The composition of claim 4 wherein the alkyl resin is a medium oil alkyd having 70 percent non-volatile content, a Gardner viscosity in the range of Z to Z2 and an acid number in the range of 50 to 59.

6. The composition of claim 4 wherein the acrylic resin is a solid acrylic oligomer having an acid value of 220 to 250, a ring and ball softening point in the range of 100 to 120°C and an average number molecular weight in the range of 2000 to 3000.

7. The composition of claim 1 wherein the amine is selected from the group consisting of ammonium hydroxide, dimethylethanolamine, triethylamine, diethanolamine, ethanolamine, dimethylamine, diethylamine, piperazine, morpholine and 2-amino-2-methyl-1-propanol.

8. The composition of claim 1 wherein the composition further contains

(E) from 1 to 30 weight percent, based on the combined weight of components (A) and (B), of a surfactant.

9. The composition of claim 8 wherein the surfactant is a non-ionic surfactant selected from the group consisting of alkyl phenoxy poly(ethyleneoxy) alcohols, primary ethoxylated alcohols and secondary ethoxylated alcohols.

10. The composition of claim 8 wherein the surfactant is a cationic surfactant selected from the group consisting of alkoxylated fatty amines and alkoxylated tallow amines.

11. The composition of claim 1 wherein the amount of component (A) is in the range of 70 to 40 weight

percent and the amount of component (B) is in the range of 30 to 60 weight percent.

12. A composition comprising

(A) from 70 to 45 weight percent, based on the combined weight of components (A) and (B), of a chlorinated, non-imidized polyolefin having a molecular weight in the range of 9,000 to 16,000, a softening point in the range of 80 to 95°C and an amount of chlorine in the range of 18 to 22 percent, based on the weight of the polyolefin,

(B) from 30 to 55 weight percent, based on the combined weight of components (A) and (B), of a medium oil alkyd having 70 percent non-volatile content, a Gardner viscosity in the range of Z to Z2 and an acid number in the range of 50 to 59,

(C) from 10 to 20 weight percent, based on the combined weight of components (A) and (B), of ammonium hydroxide, and

(D) water.

13. A composition comprising

(A) from 70 to 45 weight percent, based on the combined weight of components (A) and (B), of

a chlorinated, imidized polyolefin having a molecular weight in the range of 9,000 to 16,000, a softening point in the range of 80 to 95°C and an amount of chlorine in the range of 18 to 22 percent, based on the weight of the polyolefin,

(B) from 30 to 55 weight percent, based on the combined weight of components (A) and (B), of a solid acrylic oligomer having an acid value of 220 to 250, a ring and ball softening point in the range of 100 to 120°C and an average number molecular weight in the range of 2000 to 3000,

(C) from 10 to 20 weight percent, based on the combined weight of components (A) and (B), of dimethylethanolamine, and

(D) water.

14. A polyolefin article coated with the composition of Claim 1.

15. A process comprising

(A) preparing an admixture of

(1) from 70 to 30 weight percent, based on the combined weight of components (1) and (2), of a chlorinated polyolefin having a

molecular weight in the range of 9,000 to 250,000, a softening point in the range of 75 to 115°C, and an amount of chlorine in the range of 15 to 35 weight percent, based on the weight of the polyolefin, and

(2) from 30 to 70 weight percent, based on the combined weight of components (1) and (2), of a carboxylic acid functional resin, and

(B) preparing an admixture of

(1) the admixture prepared in step (A),

(2) from 0 to 20 weight percent, based on the weight of the admixture prepared in step (A), of a primary , secondary or tertiary amine having a molecular weight in the range of 20 to 200, and

(3) water.

16. The process of claim 14 wherein the chlorinated polyolefin has a molecular weight in the range of 9,000 to 16,000, a softening point in the range of 80 to 95°C and an amount of chlorine in the range of 18 to 22 percent.

17. The process of claim 14 wherein the chlorinated polyolefin is a chlorinated, imidized polyolefin has a molecular weight in the range of 9,000 to 16,000, a softening point in the range of 80 to 95°C and an amount of chlorine in the range of 18 to 22 percent.

18. The process of claim 14 wherein the carboxylic acid functional resin in selected from the group consisting of alkyd resins, polyester resins and acrylic resins.

19. The process of claim 17 wherein the alkyl resin is a medium oil alkyd having 70 percent non-volatile content, a Gardner viscosity in the range of Z to Z2 and an acid number in the range of 50 to 59.

20. The process of claim 17 wherein the acrylic resin is a solid acrylic oligomer having an acid value in the range of 220 to 250, a ring and ball softening point in the range of 100 to 120°C and an average number molecular weight in the range of 2000 to 3000.

21. The process of claim 14 wherein the amine is selected from the group consisting of ammonium

hydroxide, dimethylethanolamine, triethylamine, diethanolamine, ethanolamine, dimethylamine, diethylamine, piperazine, morpholine and 2-amino-2-methyl-1-propanol.

22. The process of claim 14 wherein the admixture prepared in step (B) further comprises

(4) from 1 to 30 weight percent, based on the combined weight of the admixture prepared in step (A), of a surfactant.

23. The process of claim 21 wherein the surfactant is a non-ionic surfactant selected from the group consisting of alkyl phenoxy poly(ethyleneoxy) alcohols, primary ethoxylated alcohols and secondary ethoxylated alcohols.

24. The process of claim 21 wherein the surfactant is a cationic surfactant selected from the group consisting of alkoxylated fatty amines and alkoxylated tallow amines.

25. The process of claim 14 wherein the amount of component (1) in step (A) is in the range of 70 to 40 weight percent and the amount of component (2)

is step (A) is in the range of 30 to 60 weight percent.

26. A process comprising

(A) preparing an admixture of

(1) from 70 to 45 weight percent, based on the combined weight of components (1) and (2), of a chlorinated, non-imidized polyolefin having a molecular weight in the range of 9,000 to 16,000, a softening point in the range of 80 to 95°C and an amount of chlorine in the range of 18 to 22 percent, based on the weight of the polyolefin, and

(2) from 30 to 55 weight percent, based on the combined weight of components (1) and (2), of a medium oil alkyd having 70 percent non-volatile content, a Gardner viscosity in the range of Z to Z2 and an acid number in the range of 50 to 59, and

(B) preparing an admixture of

(1) the admixture prepared in step (A),

(2) from 10 to 20 weight percent, based on the weight of the admixture prepared in step (A), of ammonium hydroxide, and

(3) water.

27. A process comprising

(A) preparing an admixture of

(1) from 70 to 45 weight percent, based on the combined weight of components (1) and (2), of a chlorinated, imidized polyolefin having a molecular weight in the range of 9,000 to 16,000, a softening point in the range of 80 to 95°C and an amount of chlorine in the range of 18 to 22 percent, based on the weight of the polyolefin, and

(2) from 30 to 55 weight percent, based on the combined weight of components (1) and (2), of a solid acrylic oligomer having an acid value of 220 to 250, a ring and ball softening point in the range of 100 to 120°C and an average number molecular weight in the range of 2000 to 3000, and

(B) preparing an admixture of

(1) the admixture prepared in step (A),

(2) from 10 to 20 weight percent, based on the weight of the admixture prepared in step (A), of dimethylethanolamine, and

(3) water.

INTERNATIONAL SEARCH REPORT

International Application No PCT/US 92/06286

I. CLASSIFICATION OF SUBJECT MATTER (if several classification symbols apply, indicate all)[6]

According to International Patent Classification (IPC) or to both National Classification and IPC

Int.Cl. 5 C09D123/28; C09D201/08; C08J7/04

II. FIELDS SEARCHED

Minimum Documentation Searched[7]

Classification System	Classification Symbols
Int.Cl. 5	C09D ; C08J

Documentation Searched other than Minimum Documentation to the Extent that such Documents are Included in the Fields Searched[8]

III. DOCUMENTS CONSIDERED TO BE RELEVANT[9]

Category °	Citation of Document,[11] with indication, where appropriate, of the relevant passages[12]	Relevant to Claim No.[13]
X	EP,A,0 325 861 (MITSUI PETROCHEM.) 2 August 1989 see column 1, line 5 - line 10 see column 4, line 15 - line 30 see claims 1,2; examples 2,6	1,4,14, 18
A	WO,A,9 012 056 (PPG INDUSTRIES) 18 October 1990 cited in the application see page 8, line 15 - line 21 see page 6, line 20 - line 31 see page 10 - page 11; examples see claims 1,5,9	1,4,8, 14,18
A	WO,A,8 911 494 (EASTMAN KODAK) 30 November 1989 cited in the application see page 11, line 14 - page 13, line 18 see page 14, line 1 - line 21	1-3,14, 17,21

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° Special categories of cited documents :[10]

"A" document defining the general state of the art which is not considered to be of particular relevance

"E" earlier document but published on or after the international filing date

"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)

"O" document referring to an oral disclosure, use, exhibition or other means

"P" document published prior to the international filing date but later than the priority date claimed

"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step

"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.

"&" document member of the same patent family

IV. CERTIFICATION

Date of the Actual Completion of the International Search	Date of Mailing of this International Search Report
02 NOVEMBER 1992	11. 11. 92
International Searching Authority	**Signature of Authorized Officer**
EUROPEAN PATENT OFFICE	ENGEL S.L.

III. DOCUMENTS CONSIDERED TO BE RELEVANT (CONTINUED FROM THE SECOND SHEET)

Category °	Citation of Document, with indication, where appropriate, of the relevant passages	Relevant to Claim No.
A	GB,B,539 416 (STANDARD OIL) 10 September 1941 see page 6, right column, paragraph 2; example 1 see claims 1,5,11,12	1,4,5, 14,21

ANNEX TO THE INTERNATIONAL SEARCH REPORT ON INTERNATIONAL PATENT APPLICATION NO. US 9206286 SA 63293

This annex lists the patent family members relating to the patent documents cited in the above-mentioned international search report. The members are as contained in the European Patent Office EDP file on
The European Patent Office is in no way liable for these particulars which are merely given for the purpose of information. 02/11/92

Patent document cited in search report	Publication date	Patent family member(s)		Publication date
EP-A-0325861	02-08-89	JP-A-	1174579	11-07-89
WO-A-9012056	18-10-90	DE-A-	3910901	11-10-90
		EP-A-	0466743	22-01-92
WO-A-8911494	30-11-89	US-A-	4966947	30-10-90
		EP-A-	0452311	23-10-91
		JP-T-	3504392	26-09-91
		US-A-	4954573	04-09-90
GB-B-539416		None		