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(54) Title: CURABLE COMPOSITIONS FOR DISPLAY DEVICES

(57) Abstract: Provided are curable compositions for the preparation of displays. The compositions are particularly suitable as adhesives, sealants, and/or encapsulants for displays. Compositions according to the invention include those having an epoxy resin and a hydroxy-functional compound, wherein the compositions provide good barrier properties after cure.



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CURABLE COMPOSITIONS FOR DISPLAY DEVICES

Field of the Invention

The present invention relates to curable compositions for display devices, *e.g.* liquid crystal displays and organic light emitting diode displays. The present compositions are particularly suitable as adhesives, sealants, and/or encapsulants.

Background

A liquid crystal display ("LCD") typically comprises a liquid crystal material that is housed between two sheets (*e.g.* glass sheets or plastic sheets). Adhesives find several applications in the field of LCD manufacturing. First, an adhesive is typically used to bond the above-mentioned two sheets together and the adhesive acts as a gasket or a sealant to confine liquid crystal material within the display. Generally, a small gap is left in the gasket. The gap is used to introduce the liquid crystal material into the display. After filling the display with liquid crystal material, the gap is sealed with adhesive. Adhesives are also used to bond electrode terminals to the display. Further details about liquid crystal displays and the use of adhesives in liquid crystal displays can be found in the article entitled "Ultraviolet curable adhesive applications on the liquid crystal display" by John M. Dooley, published on pages 13-16 in the December 1993 issue of "European Adhesives and Sealants". Said pages 13-16 are hereby incorporated in their entirety by reference.

The production of LCD's often involves a high temperature silicon deposition. Furthermore, LCD's tend to heat up during use (*e.g.* because of light absorption by polarizers used in the LCD's). Accordingly, sealants and adhesives for LCD's should be resistant to elevated temperatures. Other requirements for adhesives/sealants include good adhesion to the LCD sheets and a low water vapor transmission (water can be detrimental to, for instance, the electrodes).

A low water vapor transmission ("WVT") is particularly important in the field of organic light emitting diodes ("OLED"). Displays based on OLED's are believed to maintain several advantages over LCD's, for instance superior imaging capabilities and a longer battery life. However, OLED's often comprise comparatively unstable organic materials (*e.g.* comparatively unstable conjugated polymers) and highly water sensitive electrodes (*e.g.*

calcium based electrodes). Accordingly, sealants for OLED's should have excellent barrier properties.

5 It is an object of the present invention to provide adhesives/sealants for displays, wherein the adhesives/sealants exhibit, after cure, a low water vapor transmission.

It is an object of the present invention to provide adhesives/sealants for displays, wherein the adhesives/sealants exhibit, after cure, good resistance to high temperatures.

10 Particularly, it is an object of the present invention to provide adhesives/sealants for displays wherein the adhesives/sealants exhibit, after cure, a combination of high temperature resistance, good adhesion, and a low water vapor transmission.

Summary of the Invention

15 The present invention provides curable compositions for the preparation of displays. The compositions are particularly suitable as adhesives, sealants, and/or encapsulants for displays. Compositions according to the invention include those having an epoxy resin and a hydroxy-functional compound, wherein the compositions provide good barrier properties after cure.

20 Detailed Description of the Invention

The present invention provides compositions for display devices (*e.g.* adhesives, sealants, and/or encapsulants for display devices), wherein the composition comprises:

- (i) an epoxy resin; and
- (ii) a hydroxy-functional component.

25

(i) Epoxy Resin

The present compositions contain one or more epoxy resins. Preferably, the compositions will comprise at least one liquid (at room temperature, 23°C) component such that the combination of materials is a liquid. Thus, the epoxide-containing material is preferably a
30 single liquid epoxy material, a combination of liquid epoxy materials, or a combination of liquid epoxy material(s) and solid epoxy material(s) which is soluble in the liquid. However, in certain embodiments, *e.g.* in embodiments where the epoxide material is soluble in other components of the adhesive, the epoxide material may be comprised only of materials that are solid at room temperature.

Examples of suitable epoxy materials include polyglycidyl and poly(methylglycidyl) esters of polycarboxylic acids, or poly(oxiranyl) ethers of polyethers. The polycarboxylic acid can be aliphatic, such as, for example, glutaric acid, adipic acid and the like; cycloaliphatic, such as, for example, tetrahydrophthalic acid; or aromatic, such as, for example, phthalic acid, isophthalic acid, trimellitic acid, or pyromellitic acid. The polyether can be poly(tetramethylene oxide).

Suitable epoxy materials also include polyglycidyl or poly(-methylglycidyl) ethers obtainable by the reaction of a compound having at least one free alcoholic hydroxy group and/or phenolic hydroxy group and a suitably substituted epichlorohydrin. The alcohols can be acyclic alcohols, such as, for example, ethylene glycol, diethylene glycol, and higher poly(oxyethylene) glycols; cycloaliphatic, such as, for example, 1,3- or 1,4-dihydroxycyclohexane, bis(4-hydroxycyclohexyl)methane, 2,2-bis(4-hydroxycyclohexyl)propane, or 1,1-bis(hydroxymethyl)cyclohex-3-ene; or contain aromatic nuclei, such as N,N-bis(2-hydroxyethyl)aniline or p,p'-bis(2-hydroxyethylamino)diphenylmethane.

Other suitable epoxy compounds include those which may be derived from mono nuclear phenols, such as, for example, resorcinol or hydroquinone, or they may be based on polynuclear phenols, such as, for example, bis(4-hydroxyphenyl)methane (bisphenol F), 2,2-bis(4-hydroxyphenyl)propane (bisphenol A), or on condensation products, obtained under acidic conditions, of phenols or cresols with formaldehyde, such as phenol novolacs and cresol novolacs.

Particularly preferred epoxies include cycloaliphatic epoxies (e.g. cyclohexeneoxide epoxies), bisphenol epoxies (e.g. bisphenol A epoxies or bisphenol F epoxies), and novolac epoxies (e.g. cresolic novolac epoxies or phenolic novolac epoxies). Regarding cycloaliphatic epoxies, it is further preferred that the epoxy compounds comprise at least one cyclohexeneoxide structure, more preferably at least 2 cyclohexeneoxide structures.

Preferred cycloaliphatic diepoxides include bis(4-hydroxycyclohexyl)methane diglycidyl ether, 2,2-bis(4-hydroxycyclohexyl)propane diglycidyl ether, 3,4-epoxycyclohexylmethyl-3,4-epoxycyclohexanecarboxylate, 3,4-epoxy-6-methylcyclohexylmethyl-3,4-epoxy-6-

methylcyclohexanecarboxylate, di(3,4-epoxycyclohexylmethyl)hexanedioate, di(3,4-epoxy-6-methylcyclohexylmethyl)hexanedioate, ethylenebis(3,4-epoxycyclohexanecarboxylate), ethanedioldi(3,4-epoxycyclohexylmethyl) ether, 2-(3,4-epoxycyclohexyl-5,5-spiro-3,4-epoxy)cyclohexane-1,3-dioxane, and combinations thereof.

5

The epoxy materials can have molecular weights which vary over a wide range. Generally, the epoxy equivalent weight, *i.e.* the number average molecular weight divided by the number of reactive epoxy groups, is in the range of 60 to 1000.

- 10 Preferably the compositions of the present invention comprise, relative to the total weight of the composition, at least 10wt%, more preferably at least 20wt%, and most preferably at least 30wt% of cationically curable components. Preferably the compositions of the invention comprise, relative to the total weight of the composition, less than 90 wt%, more preferably less than 80wt%, and most preferably less than 70wt% of cationically curable components.

15

(ii) Hydroxy-functional Component

- The present compositions comprise an hydroxy-functional component. The hydroxy-functional component which can be used in the present invention may be any suitable organic material having a hydroxyl functionality of at least 1, more preferably at least 2, and most preferably at least 3. Preferably the hydroxyl-containing material is aliphatic. Materials comprising more than one hydroxyl group are also referred to as polyols. Materials comprising one hydroxyl group are also referred to as monols.

- 25 Any hydroxy group may be employed for the particular purpose. Preferably the hydroxyl-containing material contains two or more primary or secondary aliphatic hydroxyl. The hydroxyl group may be internal in the molecule or terminal. Monomers, oligomers or polymers can be used. The hydroxyl equivalent weight, *i.e.*, the number average molecular weight divided by the number of hydroxyl groups, is preferably in the range of 31 to 5000.

- 30 Representative examples of hydroxyl-containing materials having a hydroxyl functionality of 1 include alkanols, monoalkyl ethers of polyoxyalkyleneglycols, monoalkyl ethers of alkyleneglycols, and others, and combinations thereof.

Representative examples of useful monomeric polyhydroxy organic materials include alkylene and arylalkylene glycols and polyols, such as 1,2,4-butanetriol, 1,2,6-hexanetriol, 1,2,3-heptanetriol, 2,6-dimethyl-1,2,6-hexanetriol, (2R,3R)-(-)-2-benzyloxy-1,3,4-butanetriol, 1,2,3-hexanetriol, 1,2,3-butanetriol, 3-methyl-1,3,5-pentanetriol, 1,2,3-cyclohexanetriol, 5 1,3,5-cyclohexanetriol, 3,7,11,15-tetramethyl-1,2,3-hexadecanetriol, 2-hydroxymethyltetrahydropyran-3,4,5-triol, 2,2,4,4-tetramethyl-1,3-cyclobutanediol, 1,3-cyclopentanediol, trans-1,2-cyclooctanediol, 1,16-hexadecanediol, 3,6-dithia-1,8-octanediol, 2-butyne-1,4-diol, 1,3-propanediol, 1,4-butanediol, 1,5-pentanediol, 1,6-hexanediol, 1,7-heptanediol, 1,8-octanediol, 1,9-nonanediol, 1-phenyl-1,2-ethanediol, 1,2-cyclohexanediol, 10 1,5-decalindiol, 2,5-dimethyl-3-hexyne-2,5-diol, 2,7-dimethyl-3,5-octadiyne-2,7-diol, 2,3-butanediol, 1,4-cyclohexanedimethanol, and combinations thereof.

Representative examples of useful oligomeric and polymeric hydroxyl-containing materials include polyoxyethylene and polyoxypropylene glycols and triols of molecular weights from 15 about 200 to about 10,000; polytetramethylene glycols of varying molecular weight; poly(oxyethylene-oxybutylene) random or block copolymers; copolymers containing pendant hydroxy groups formed by hydrolysis or partial hydrolysis of vinyl acetate copolymers, polyvinylacetal resins containing pendant hydroxyl groups; hydroxy-terminated polyesters and hydroxy-terminated polylactones; hydroxy-functionalized polyalkadienes, 20 such as polybutadiene; aliphatic polycarbonate polyols, such as an aliphatic polycarbonate diol; and hydroxy-terminated polyethers, and combinations thereof.

Preferred hydroxyl-containing monomers include 1,4-cyclohexanedimethanol and aliphatic and cycloaliphatic monohydroxy alkanols. Preferred hydroxyl-containing oligomers and 25 polymers include hydroxyl and hydroxyl/epoxy functionalized polybutadiene, polycaprolactone diols and triols, ethylene/butylene polyols, and monohydroxyl functional monomers. Preferred examples of polyether polyols are polypropylene glycols of various molecular weights and glycerol propoxylate-B-ethoxylate triol. Also preferred are linear and branched polytetrahydrofuran polyether polyols available in various molecular weights, such 30 as in the range of 150-4000 g/mol, preferably in the range of 150-1500 g/mol, more preferably in the range of 150-750 g/mol.

Especially preferred polyols include (i) polyester polyols, (ii) polyols comprising one or more caprolactone residues, and (iii) C₁-C₁₀ glycols (*e.g.* ethylene glycol, propylene glycol, or

butylene glycol). Particularly preferred are polyester polyols comprising caprolactone residues, such as the trimethylolpropane triester with caprolactone.

5 In one embodiment, the compositions preferably comprise, relative to the total weight of the composition, at least 1 wt% of one or more hydroxy-functional compounds. In another embodiment, the compositions preferably comprise, relative to the total weight of the composition, at least 5 wt%, and most preferably at least 10 wt% of one or more hydroxy-functional compounds. Furthermore, the compositions preferably comprise, relative to the total weight of the composition, at most 60 wt% of one or more hydroxy-functional
10 compounds, more preferably at most 40 wt%, and most preferably at most 25 wt%. C₁-C₁₀ glycols are preferably present in an amount of at most 10 wt%, more preferably at most 5 wt% relative to the total weight of the composition.

Preferably the ratio of the number of epoxide equivalents to the number of hydroxyl
15 equivalents in the present compositions (hereinafter also referred to as "EH ratio" or "EHR") is at least 1.5, more preferably at least 1.65, most preferably at least 1.8. If the EH ratio is below 1.5, the resistance of the composition to yellowing and/or degradation may decrease. The EH ratio is preferably below 100, more preferably below 20, even more preferably below 5, and most preferably about 2.

20 In one embodiment, the ratio of the combined weight of epoxy-functional components and hydroxy-functional components in the composition to the number of hydroxyl groups in the composition (hereinafter also referred to as "Weight Per Hydroxy" or "WPH") is at least 350. In another embodiment, the WPH is at least 400, and in an even further embodiment the
25 WPH is at least 500. Increased WPH values may increase the flexibility of the compositions (when cured).

Preferably the combined weight of epoxy resins and hydroxy-functional components makes up, relative to the total weight of the composition, at least 70 wt% of the composition, more
30 preferably at least 80 wt%, even more preferably at least 90 wt%, and most preferably at least 95 wt%.

(iii) Adhesion Promoter

The present compositions preferably comprise a suitable adhesion promoter or mixture of adhesion promoters. Suitable adhesion promoters include silane adhesion promoters.

Examples of silane adhesion promoters include acrylate-functional silanes; amino-functional silanes; mercapto-functional silanes; methacrylate-functional silanes; acrylamido-functional silanes; allyl-functional silanes; epoxy-functional silanes; and vinyl-functional silanes. The adhesion promoters preferably are methoxy- or ethoxy-substituted as well. Preferably the present adhesives comprise an epoxy-functional silane adhesion promoter, more preferably an epoxy-functional trialkoxy silane adhesion promoter, most preferably a 3-glycidoxypentyltrimethoxysilane adhesion promoter. Commercial examples of a 3-glycidoxypentyltrimethoxysilane adhesion promoter include Z-6040 from Dow Corning.

The present compositions preferably comprise, relative to the total weight of the composition, 0.1-10 wt% of adhesion promoter, more preferably 0.1-5 wt%, most preferably 0.25-3 wt%.

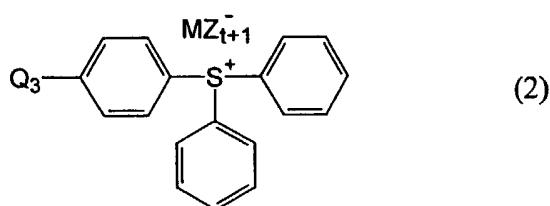
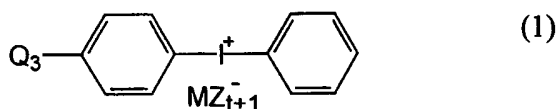
(iv) Cationic Photoinitiator

The present compositions preferably comprise a cationic photoinitiator. In the compositions according to the invention, any suitable type of photoinitiator that, upon exposure to actinic radiation, forms cations that initiate the reactions of the cationically polymerizable compounds, such as epoxy material(s), can be used. There are a large number of known and technically proven cationic photoinitiators that are suitable. They include, for example, onium salts with anions of weak nucleophilicity. Examples are halonium salts, iodosyl salts or sulfonium salts, such as are described in published European patent application EP 153904 and WO 98/28663, sulfoxonium salts, such as described, for example, in published European patent applications EP 35969, 44274, 54509, and 164314, or diazonium salts, such as described, for example, in U.S. Patents 3,708,296 and 5,002,856. All eight of these disclosures are hereby incorporated in their entirety by reference. Other cationic photoinitiators are metallocene salts, such as described, for example, in published European applications EP 94914 and 94915, which applications are both hereby incorporated in their entirety by reference.

A survey of other current onium salt initiators and/or metallocene salts can be found in "UV Curing, Science and Technology", (Editor S. P. Pappas, Technology Marketing Corp., 642 Westover Road, Stamford, Conn., U.S.A.) or "Chemistry & Technology of UV & EB

Formulation for Coatings, Inks & Paints", Vol. 3 (edited by P. K. T. Oldring), and both books are hereby incorporated in their entirety by reference.

Preferred initiators include diaryl iodonium salts, triaryl sulfonium salts, or the like. Typical photo-polymerization initiators are represented by the following formulae (1) and (2):



wherein

Q₃ represents a hydrogen atom, an alkyl group having 1 to 18 carbon atoms, or an alkoxy group having 1 to 18 carbon atoms;

M represents a metal atom, for instance antimony;

Z represents a halogen atom, for instance fluorine; and

t is the valent number of the metal, for example 6 in the case of antimony.

Preferably, the present compositions comprise, relative to the total weight of the adhesive, 0.1-15 wt% of one or more cationic photoinitiators, more preferably 1-10 wt%.

(v) Antioxidant

The present compositions preferably comprise an antioxidant. Any suitable antioxidant may be used. Preferred antioxidants include hindered phenol antioxidants, for instance octadecyl-3-(3',5'-di-tert-butyl-4'-hydroxyphenyl) propionate, thiodiethylene bis (3,5-di-tert-butyl-4-hydroxy) hydrocinnamate, butylated paracresol-dicyclopentadiene copolymer; and tetrakis [methylene (3,5-di-tert-butyl-4-hydroxyhydro-cinnamate)] methane. Commercial examples include Irganox 1010 and Irganox 1035 from Ciba Geigy.

Preferably the present compositions comprise, relative to the total weight of the composition, 0.1-5 wt% of antioxidant, more preferably 0.1-2 wt%.

(vi) Free Radically Polymerizable Components, and

(vii) Free Radical Photoinitiators

The present compositions may comprise free radically polymerizable components, for instance allyl-, acrylate- or methacrylate functional components, and free radical photoinitiators, for instance acetophenone or benzil ketal free radical photoinitiators.

However, their presence is generally not preferred, for instance because their presence tends to result in cured compositions having a comparatively low high temperature resistance to yellowing. Preferably the present compositions comprise, relative to the total weight of the composition, less than 30 wt% of free radical polymerizable components, more preferably less than 15 wt%, even more preferably less than 5 wt%, and most preferably the present compositions are absent free radical polymerizable components. The present compositions preferably comprise, relative to the total weight of the composition, less than 2 wt% of free radical photoinitiators, more preferably less than 1 wt%, and most preferably the present compositions are absent free radical photoinitiators.

(viii) Additives

The present compositions may further comprise any suitable additive. Examples of additives include, for instance, inert inorganic materials (e.g. silicon dioxides or nanoclays), surfactants, and the like. Silicon dioxides and nanoclays may aid in further improving the barrier properties of the present composition, for instance further reducing the water vapor transmission and/or permeance of the cured composition. If present, silicon dioxides and nanoclays are preferably used in amounts of 0.1-10 wt%, more preferably 0.1-5 wt%, and most preferably 0.1-3 wt% relative to the total weight of the composition.

Applications

The present compositions are suitable in the preparation of displays, e.g. as adhesives, sealants (for instance side sealants), and/or encapsulants for displays.

For instance, the present compositions are suitable in the fabrication of LCD's. LCD's generally comprise a liquid crystal material that is housed between two sheets, for instance glass sheets or plastic sheets. The present compositions may be used to bond the two sheets together, and the compositions may act as a gasket or a (side) sealant to confine liquid crystal material within the display. Generally, a small gap is left in the gasket. The gap is used to introduce the liquid crystal material into the display. After filling the display with liquid

crystal material, the present compositions may be used to seal the gap. The present compositions may also be used to bond electrode terminals to the display.

Further examples of displays in which the present compositions may be used include organic
5 light emitting diode (OLED) displays. The present compositions are particularly suitable as encapsulants or (side-) sealants for OLED's to protect the organic light emitting layer and/or the electrodes in the OLED's from oxygen and, particularly, water.

Accordingly, preferred compositions include those that provide good adhesion to substrates
10 and good barrier properties. Also, because the production of LCD's often involves a high temperature silicon deposition step, it is preferred that the compositions for displays have good high temperature resistance. Furthermore, in particular from an integrity and aesthetic point of view, it is preferred that the compositions provide good scratch resistance.

Consequently, preferred compositions according to the present invention include those
15 having (when cured) one or more of the following properties:

- (i) a water vapor transmission, as measured according to the test method set forth herein, of less than $10 \text{ g/hr}\cdot\text{m}^2$, preferably less than $5 \text{ g/hr}\cdot\text{m}^2$, more preferably less than $1.5 \text{ g/hr}\cdot\text{m}^2$, and most preferably less than $0.5 \text{ g/hr}\cdot\text{m}^2$;
- (ii) a water vapor permeance, as measured according to the test method set forth herein, of
20 less than $0.06 \text{ g/Pa}\cdot\text{hr}\cdot\text{m}^2$, preferably less than $0.03 \text{ g/Pa}\cdot\text{hr}\cdot\text{m}^2$, more preferably less than $0.01 \text{ g/Pa}\cdot\text{hr}\cdot\text{m}^2$, and most preferably less than $0.001 \text{ g/Pa}\cdot\text{hr}\cdot\text{m}^2$;
- (iii) an adhesion to glass, as determined at 50% humidity and 23°C by a 180° peel test at a peeling rate of 0.1 inch/min, of at least 20 g/in and below 1000 g/in, more preferably at least 40 g/in, and most preferably at least 60 g/in;
- 25 (iv) a hardness of at least H, more preferably at least 3H, and most preferably at least 6H.

A process for preparing a display according to the present invention includes curing the present composition. The curing may be effected by any suitable means, preferably by radiation (*e.g.* electron beam radiation or, preferably, UV radiation) and/or heat. Preferably
30 the process includes curing the adhesive by UV radiation.

The present displays may be used in a wide variety of articles, for instance in computers (*e.g.* in computer monitor screens or laptop screens), televisions, cameras (*e.g.* camcorders),

watches, calculators, cell phones, telephones, pagers, palm pilots, stereos (*e.g.* in car stereo displays) etc.

Examples

- 5 The following examples are given as particular embodiments of the invention and to demonstrate the practice and advantages thereof. It is to be understood that the examples are given by way of illustration and are not intended to limit the specification or the claims that follow in any manner.
- 10 Examples 1-21
- Compositions were prepared, cured, and tested. The composition formulations and test results are set forth in the following Tables 1-4 (for details on sample preparation and test methods, please see the "Test Method" section below). All weight percentages in Tables 1-4 are relative to the total weight of the composition. Details about the ingredients used in the
- 15 compositions are set forth in the following glossary:

Glossary

Name	Description
Araldite GY 282	Bisphenol F Epoxy available from Ciba Resins
CN 816	Acrylic oligomer available from Sartomer
CN 817	Acrylic oligomer available from Sartomer
CN 818	Acrylic oligomer available from Sartomer
DEN 438	Phenolic epoxy novolac resin available from Dow Chemical
Ebecryl 745	Acrylic oligomer available from UCB Chemicals
Ebecryl 754	Acrylic oligomer available from UCB Chemicals
Ebecryl 767	Acrylic oligomer available from UCB Chemicals
Irgacure 1173	Free radical photoinitiator available from Ciba Geigy
Irganox 1010	Antioxidant available from Ciba Geigy
Irganox 1035	Antioxidant available from Ciba Geigy
SR1010	cationic photoinitiator: Triarylsulfonium hexafluoroantimonate (50% in propylene carbonate) available from Sartomer
SR1011	cationic photoinitiator: Triarylsulfonium hexafluorophosphate (50% in propylene carbonate) from Sartomer
Surfynol 420	ethoxylated acetylenic diols (>65% ethoxylated 2,4,7,9-tetramethyl-5-decyn-4,7-diol) available from Air Products
Uvacure 1500	3,4-epoxycyclohexyl-methyl-3,4-epoxycyclohexane carboxylate available from UCB Chemicals
Uvacure 1501	Cycloaliphatic diepoxide available from UCB Chemicals
Uvacure 1502	Cycloaliphatic diepoxide available from UCB Chemicals
Uvacure 1530	mixture of aliphatic polyol and 3,4-epoxycyclohexyl-methyl-3,4-epoxycyclohexane carboxylate (EHR = 2, WPH = 370), available from UCB Chemicals.
Uvacure 1531	mixture of aliphatic polyol and 3,4-epoxycyclohexyl-methyl-3,4-epoxycyclohexane carboxylate (EHR = 2, WPH = 446), available from UCB Chemicals.
Uvacure 1532	mixture of aliphatic polyol and 3,4-epoxycyclohexyl-methyl-3,4-epoxycyclohexane carboxylate (EHR = 2, WPH = 555), available from UCB Chemicals.
Uvacure 1533	modified cycloaliphatic epoxide available from UCB Chemicals.
Uvacure 1534	mixture of aliphatic polyol and modified cycloaliphatic epoxy (EHR = 1.4, WPH = 375), available from UCB Chemicals.
UVI 6974	mixture of triarylsulfonium hexafluoroantimonate salts, available from Union Carbide
UVI 6990	mixture of triarylsulfonium hexafluorophosphate salts, available from Union Carbide
Z-6040	3-glycidoxypolytrimethoxy silane, available from Dow Corning
Zeothix 177	silicon dioxide, available from J. M. Huber Corporation

Table 1

Ingredients	Ex. 1	Ex. 2
Araldite GY282 (epoxy), wt%	45.0	46.0
DEN 438 (epoxy), wt%	51.0	48.0
UVI 6974 (cationic photoinitiator), wt%	2.0	2.0
Ethylene Glycol (polyol), wt%	2.0	2.0
Zeothix 177 (silicon dioxide), wt%	--	2.0
Properties		
Viscosity (cps)	32,570	32,560
Water Vapor Transmission (g/hr·m ²)	0.20	0.10
Water Vapor Permeance (g/Pa·hr·m ²)	0.0015	0.00075

Table 2

Ingredient	Ex. 3	Ex. 4	Ex. 5	Ex. 6
Uvacure 1531 (epoxy/polyol mixture), wt%	97.0	97.0	--	--
Uvacure 1532 (epoxy/polyol mixture), wt%	--	--	97.0	--
Uvacure 1530 (epoxy/polyol mixture), wt%	--	--	--	96.5
Irganox 1010 (antioxidant), wt%	0.5	--	--	--
Irganox 1035 (antioxidant), wt%	--	0.5	0.5	0.5
SR1010 (cationic photoinitiator), wt%	--	2.0	--	2.0
SR1011 (cationic photoinitiator), wt%	2.0	--	2.0	--
Z-6040 (adhesion promoter), wt%	0.5	0.5	0.5	1.0
Properties				
Water Vapor Transmission (g/hr·m ²)	2.7	2.1	4.1	1.1
Water Vapor Permeance (g/Pa·hr·m ²)	0.020	0.016	0.030	0.008
Adhesion to Glass and Mylar	excellent	excellent	excellent	excellent

Table 3

Ingredient	Ex. 7	Ex. 8	Ex. 9	Ex. 10	Ex. 11	Ex. 12	Ex. 13
Ebecryl 745, wt% (acrylic oligomer)	97	--	--	--	--	--	--
Ebecryl 754, wt% (acrylic oligomer)	--	97	--	--	--	--	--
Ebecryl 767, wt% (acrylic oligomer)	--	--	97	--	--	--	--
CN 816, wt% (acrylic oligomer)	--	--	--	97	--	--	--
CN 817, wt% (acrylic oligomer)	--	--	--	--	97	--	--
CN 818, wt% (acrylic oligomer)	--	--	--	--	--	97	--
Araldite GY282 (epoxy), wt%	--	--	--	--	--	--	63
DEN 438 (epoxy), wt%	--	--	--	--	--	--	35
Surfynol 420, wt%	--	--	--	--	--	--	1.0
UVI 6974 (cationic photoinitiator), wt%	--	--	--	--	--	--	1.0
Irgacure 1173, wt% (free radical photoinitiator)	3	3	3	3	3	3	--
Properties							
Yellowing rating after 8hrs @ 300°C	3	3	3	4	4	4	1
Yellowing rating after 8hrs @ 250°C	3	3	3	4	4	4	1

Table 4

Ingredient	Ex. 14	Ex. 15	Ex. 16	Ex. 17	Ex. 18	Ex. 19	Ex. 20	Ex. 21
Uvacure 1500 (epoxy)	97	--	--	--	--	--	--	--
Uvacure 1501 (epoxy)	--	97	--	--	--	--	--	--
Uvacure 1502 (epoxy)	--	--	97	--	--	--	--	--
Uvacure 1530 (epoxy/polyol mixture, EHR = 2), wt%	--	--	--	97	--	--	--	--
Uvacure 1531 (epoxy/polyol mixture, EHR = 2), wt%	--	--	--	--	97	--	--	--
Uvacure 1532 (epoxy/polyol mixture, EHR = 2), wt%	--	--	--	--	--	97	--	--
Uvacure 1533 (epoxy)	--	--	--	--	--	--	97	--
Uvacure 1534 (epoxy/polyol mixture, EHR = 1.4), wt%	--	--	--	--	--	--	--	97
UVI 6990 (cationic photoinitiator)	3	3	3	3	3	3	3	3
Properties								
Yellowing rating after 8hrs @ 300°C	3	3	3	2	2	2	4	4
Yellowing rating after 8hrs @ 250°C	2	2	2	1	1	2	4	4

* The product obtained by curing the formulation of example 19 was more flexible than the product obtained by curing the formulation of example 18, which in turn was more flexible than the product obtained by curing the formulation of example 17 and the product obtained by curing the formulation of example 13.

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Test Methods

(i) Water Vapor Transmission (WVT) and Water Vapor Permeance (WVP).

For each composition in Table 1 and Table 2, the following procedure was followed:

A 3 mil drawdown of the composition was cast on a 9x12 inch glass plate and fully cured

10 with UV radiation to prepare a film of cured composition.

A test dish (Payne cup, 3 inch diameter, ¾ inch depth) was filled with a ½ inch thick layer of desiccant (anhydrous calcium chloride, particle size 0.6-2.36 mm). The opening of the test dish was then covered with part of the film of cured composition, and the film was secured on the test dish with a sealing ring. In addition, the outer edges of the film were tightly wrapped

15 with Parafilm™ (paraffinic film). The thus assembled test dish was weighed and then immediately placed in controlled environment (95% R.H. environment, 23°C). At one hour intervals for a total period of 6 hours, the test dish was removed from the controlled environment, weighed, gently shaken (to mix the desiccant particles) and immediately

replaced in the controlled environment. After these 6 hours, the test dish was placed in the controlled environment for an additional 18 hours, after which the test dish was once more weighed.

The datapoints were plotted in a graph, with the horizontal axis being time in hours and the vertical axis being the weight change (i.e. determined weight minus weight right before initial placement in the controlled environment). A curve was inscribed through the data points, which curve tended to become an upward straight line. The slope of this straight line was determined (in g/hr). The water vapor transmission was then calculated by multiplying the test area (i.e. the test dish opening area in m^2) by this slope. The water vapor permeance was then calculated by dividing the water vapor transmission by 133.3 Pa (i.e. the vapor pressure difference). See also ASTM Method E 96-80, which is hereby incorporated in its entirety by reference.

(ii) Yellowing

For each composition in Table 3 and Table 4, the following procedure was followed:

A first sample was prepared by covering a glass slide with a 5 micron thick layer of the composition. Subsequently, the composition was cured by UV radiation. A second sample was prepared in an identical manner as the first sample. One of the samples was then placed in an oven at 250°C (air atmosphere), whereas the other sample was placed in an oven at 300°C (air atmosphere). 8 hours after being placed in the oven, the samples were removed and visually (naked eye) graded for yellowing and burns. The following scale was used to grade the samples:

Grade	Description
1	Clear, very little yellowing or very little burn black
2	Clear, very little yellowing, and slight peripheral burn black
3	Yellowing and peripheral burn black
4	Peripheral and internal burn black

A composition having a rating of 1 or 2 after exposure for 8 hrs to 300°C is considered to have a substantial resistance to yellowing.

Having described specific embodiments of the present invention, it will be understood that many modifications thereof will readily be apparent to those skilled in the art, and it is intended therefore that this invention is limited only by the spirit and scope of the following claims.

What is claimed is:

1. A curable composition for displays, said composition comprising:
 - (i) an epoxy resin; and
 - 5 (ii) a hydroxy-functional compound;wherein said composition has substantial resistance to yellowing.
2. A curable composition for displays, said composition comprising:
 - (i) an epoxy resin; and
 - 10 (ii) a hydroxy-functional compound;wherein said composition has a ratio of epoxy equivalents to hydroxy equivalents of at least 1.5
3. A curable composition for displays, said composition comprising:
 - 15 (i) an epoxy resin;
 - (ii) a hydroxy-functional compound; and
 - (iii) 0-30 wt% of free radically polymerizable components.
4. A curable composition for displays, said composition comprising:
 - 20 (i) an epoxy resin;
 - (ii) a hydroxy-functional compound; and
 - (iii) at least one compound selected from the group consisting of nanoclays and silicon dioxide.
- 25 5. The composition according to any one of claims 1-4, wherein said composition is an adhesive, sealant, and/or encapsulant for displays.
6. The composition according to any one of claims 1-5, wherein said epoxy resin is selected from the group consisting of cycloaliphatic epoxy resins, epoxy novolacs, and epoxy
30 bisphenols.
7. The composition according to any one of claims 1-6, wherein said epoxy resin comprises at least two cyclohexene oxide structures.

8. The composition according to any one of claims 1-7, wherein said hydroxy-functional compound is a polyol.
9. The composition according to any one of claims 1-8, wherein said hydroxy-functional
5 component is selected from the group consisting of polyester polyols, polyols comprising one or more caprolactone residues, and C₁-C₁₀ glycols.
10. The composition according to any one of claims 1-9, wherein said epoxy resin and
said polyol make up, relative to the total weight of said composition, at least 70 wt% of said
10 composition.
11. The composition according to any one of claims 1-9, wherein said epoxy resin and
said polyol make up, relative to the total weight of said adhesive, at least 90 wt% of the
adhesive.
- 15 12. The composition according to any one of claims 1-11, wherein said composition has a
ratio of epoxy equivalents to hydroxy equivalents of at least 1.8.
13. The composition according to any one of claims 1-12, wherein said composition
20 further comprises a silane adhesion promoter.
14. The composition according to claim 13, wherein said adhesion promoter is an epoxy-
functional silane adhesion promoter.
- 25 15. The composition according to claim 13, wherein said adhesion promoter is a 3-
glycidoxypropyltrimethoxysilane adhesion promoter.
16. The composition according to any one of claims 1-15, wherein said composition
comprises, relative to the total weight of said composition, 0-5 wt% of free radically
30 polymerizable components.
17. The composition according to any one of claims 1-16, wherein said composition is
absent free radically polymerizable components.

18. The composition according to any one of claims 1-15, wherein said composition comprises, relative to the total weight of said composition, 0-5 wt% of components selected from the group consisting of acrylate-functional components, methacrylate-functional components, and allyl-functional components.

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19. The composition according to any one of claims 1-18, wherein said composition is absent a free radical photoinitiator.

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20. The composition according to any one of claims 1-19, wherein said composition comprises a cationic photoinitiator.

21. The composition according to any one of claims 1-20, wherein said composition comprises an antioxidant.

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22. The composition according to any one of claims 1-21, wherein said composition comprises silicon dioxide.

23. The composition according to any one of claims 1-22, wherein said composition comprises a nanoclay.

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24. A composition for displays, said composition consisting essentially of:

- (i) one or more epoxy resins;
- (ii) one or more hydroxyfunctional components;
- (iii) one or more adhesion promoters;
- (iv) one or more cationic photoinitiators;
- (v) one or more antioxidants; and
- (vi) optionally one or more components selected from the group consisting of silicon dioxide and nanoclays.

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30 25. The composition of claim 24, wherein said one or more epoxy resins includes an epoxy resin selected from the group consisting of cycloaliphatic epoxy resins, epoxy novolacs, and epoxy bisphenols.

26. The composition according to any one of claims 24-25, wherein said one or more hydroxyfunctional components consist essentially of one or more polyols.

27. The composition according to any one of claims 24-26, wherein said one or more
5 adhesion promoters include an epoxy-functional silane adhesion promoter.

28. The composition according to any one of claims 1-27, wherein said composition, after cure, has a water vapor transmission of less than $10 \text{ g/hr}\cdot\text{m}^2$.

10 29. The composition according to any one of claims 1-27, wherein said composition, after cure, has a water vapor transmission of less than $1.5 \text{ g/hr}\cdot\text{m}^2$.

30. The composition according to any one of claims 1-29, wherein said composition, after cure, has a water vapor permeance of less than $0.06 \text{ g/Pa}\cdot\text{hr}\cdot\text{m}^2$.

15 31. The composition according to any one of claims 1-29, wherein said composition, after cure, has a water vapor permeance of less than $0.01 \text{ g/Pa}\cdot\text{hr}\cdot\text{m}^2$.

32. The composition according to any one of claims 1-31, wherein said composition, after
20 cure, has an adhesion to glass of at least 20 g/in .

33. The composition according to any one of claims 1-32, wherein said composition, after cure, has a hardness of at least H.

25 34. A curable composition for displays, said composition having substantial resistance to yellowing and, after cure, the following combination of properties:

- (a) a water vapor transmission of less than $5 \text{ g/hr}\cdot\text{m}^2$;
- (b) water vapor permeance of less than $0.01 \text{ g/Pa}\cdot\text{hr}\cdot\text{m}^2$;
- (c) an adhesion to glass of at least 40 g/in ; and
- 30 (d) a hardness of at least H.

35. A process for preparing a display, said process comprising curing the composition according to any one of claims 1-34.

36. A liquid crystal display prepared by the process of claim 35.
37. An organic light emitting diode display prepared by the process of claim 35.
- 5 38. A computer, television, camera, watch, calculator, cell phone, telephone, pager, palm pilot, or stereo comprising a display prepared by the process of claim 35.

INTERNATIONAL SEARCH REPORT

International Application No
PCT/NL 02/00528

A. CLASSIFICATION OF SUBJECT MATTER

IPC 7 C08G59/62 C08G59/40 C08L63/00 G02F1/133 G09F9/35

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC 7 C08G C08L G02F G09F

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

EPO-Internal, WPI Data, PAJ

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	WO 01 44342 A (MAEDA SUNAO ;KITAMURA TADASHI (JP); MITSUI CHEMICALS INC (JP)) 21 June 2001 (2001-06-21) paragraphs '0027!', '0043!', '0047!'-'0087!', '0089!'-'0160!', '0243!', '0252!', '0253!'; examples 1-27; tables 1,10 -& EP 1 153 952 A 14 November 2001 (2001-11-14) --- -/--	1, 4-8, 13-15, 17, 19, 22, 28-36, 38



Further documents are listed in the continuation of box C.



Patent family members are listed in annex.

* Special categories of cited documents:

- *A* document defining the general state of the art which is not considered to be of particular relevance
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- *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 when the document is taken alone
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- *&* document member of the same patent family

Date of the actual completion of the international search

17 September 2002

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INTERNATIONAL SEARCH REPORT

International Application No

PCT/NL 02/00528

C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

Category °	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US 6 174 932 B1 (TAYLOR DONALD ET AL) 16 January 2001 (2001-01-16) column 2, line 20-37, 41 column 3, line 5-12, 22-31, 51-58 column 4, line 55, 56 column 7, line 15 examples 1-11 ---	1-12, 16-22, 28-34
X	WO 93 15124 A (MINNESOTA MINING & MFG) 5 August 1993 (1993-08-05) page 5-9 page 11, line 6, 7 page 11, line 10 page 11, line 15; examples 1-13 ---	1-9, 12-20, 22, 28-34
X	US 5 837 398 A (ADAMS JOHN E ET AL) 17 November 1998 (1998-11-17) column 3, line 63-66 column 4-5 example 1 ----	1-10, 12, 16-20, 28-34
A	US 5 145 889 A (WADA YUSUKE ET AL) 8 September 1992 (1992-09-08) the whole document -----	

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Int'l Application No

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