

**HIGH-TEMPERATURE AND HIGH-HUMIDITY RESISTANT OPTICALLY
CLEAR ADHESIVE, PREPARATION METHOD THEREFOR, AND
APPLICATION THEREOF**

5 Field of Invention

The invention relates to the technical field of optically clear adhesives, and more particularly to a high-temperature and high-humidity resistant optically clear adhesive (OCA), and its preparation method and applications.

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Background of Invention

With the continuous development of science and technology, the popularization and diversified applications of electronic products have put forward higher demands for material performance. As a key material, optically clear adhesives (OCAs) are widely used in display screens of various products such as smartphones, tablets, electronic watches, and automobiles. As the importance of various devices increases continuously in daily lives and the operating environment becomes increasingly complex, many devices are required to operate under high temperature and high humidity conditions. Therefore, the market demand for the optical performance, adhesion, and durability of OCAs in extreme environments such as high temperature and high humidity is continuously rising. Conventional OCAs are prone to problems, for instance, yellowing, blistering, or delamination in such extreme environments, affecting the display effects and lifespan of the device. In addition, conventional OCAs may contain volatile organic compounds (VOCs) and other harmful substances, which not only pollute the environment but also pose a potential threat to human health. Therefore, it has become a demanding need for the industry to develop an environmentally friendly OCA that can still maintain excellent performance under high temperature and high humidity conditions.

30 In view of this, the present invention is provided.

Summary of Invention

An object of the invention is to provide a high-temperature and high-humidity resistant optically clear adhesive (OCA) and its preparation method and applications. The OCA of the present invention exhibits excellent resistance against high temperature and high humidity, maintaining good optical properties, adhesion performance, and anti-ageing resistance under high-temperature and high-humidity conditions.

In order to achieve the aforementioned object of the present invention, the first aspect of the invention provides a high-temperature and high-humidity resistant OCA, comprising the following components in parts by weight: 40 parts to 55 parts of a soft monomer, 10 parts to 25 parts of a hard monomer, 5 parts to 20 parts of a functional monomer, 5 parts to 15 parts of a thiourea monomer, 3 parts to 15 parts of a polyurethane acrylate prepolymer, 0.2 parts to 2 parts of a photoinitiator, 0.05 parts to 2 parts of a chain transfer agent, 0.1 parts to 1 part of a tackifier coupling agent, 0.1 parts to 1.5 parts of an antioxidant, 0.05 parts to 1 part of a levelling agent, and 0.05 parts to 1.5 parts of a crosslinker;

wherein the polyurethane acrylate prepolymer is primarily prepared by a reaction of a polyol, a polyisocyanate, and a hydroxyl-containing acrylate; the polyol comprises polytetramethylene ether glycol and polycaprolactone diol, and the polyisocyanate comprises isophorone diisocyanate and hexamethylene diisocyanate.

In a specific embodiment of the present invention, the polytetramethylene ether glycol and the polycaprolactone diol in the polyol are present at a molar ratio of 1:(0.5-2).

In a specific embodiment of the present invention, the isophorone diisocyanate and the hexamethylene diisocyanate in the polyisocyanate are present at a molar ratio of 1:(0.5-2).

In a specific embodiment of the present invention, the polyol and the polyisocyanate are present at a molar ratio of 1:(1.2-1.5).

In a specific embodiment of the present invention, the polyol and the hydroxyl-containing acrylate are present at a molar ratio of 1:(0.8-1.2). Furthermore, the hydroxyl-containing

acrylate comprises at least one of 2-hydroxyethyl acrylate, 4-hydroxybutyl acrylate, and hydroxypropyl acrylate.

5 In a specific embodiment of the present invention, the polyurethane acrylate prepolymer is prepared under the action of a catalyst. Furthermore, the catalyst comprises at least one of dibutyltin dilaurate, triethylenediamine, and stannous octoate.

10 In a specific embodiment of the present invention, during the preparation of the polyurethane acrylate prepolymer, the raw materials further comprise a chain extender and/or a filler.

In a specific embodiment of the present invention, the amount of the chain extender is 0.5 wt% to 2 wt% of the total amount of the polyol, the polyisocyanate, and the hydroxyl-containing acrylate. Furthermore, the chain extender is 1,4-butanediol and/or 1,6-hexanediol.

15 In a specific embodiment of the present invention, the amount of the filler is 8 wt% to 12 wt% of the total amount of the polyol, the polyisocyanate, and the hydroxyl-containing acrylate. Furthermore, the filler is at least one of nanocellulose, nanoalumina, and nanosilica; the particle size of the filler is 1 nm to 50 nm.

20 In a specific embodiment of the present invention, the thiourea monomer comprises at least one of 1,3-diallyl 2-thiourea, allyl thiourea, 1-allyl-3-(4-benzylphenyl)thiourea, 1-allyl-3-(pyridin-3-yl)thiourea, 1-allyl-3,3-diethyl-2-thiourea, 1-methallyl-3-methyl-2-thiourea, and N-propylene-N'-2-hydroxyethylthiourea.

25 In a specific embodiment of the present invention, the thiourea monomer and the polyurethane acrylate prepolymer are present at a mass ratio of (0.5-3):1.

30 In a specific embodiment of the present invention, the soft monomer has a glass transition temperature of -60 °C to -10 °C. Furthermore, the soft monomer comprises at least one of ethyl acrylate, 2-ethylhexyl acrylate, lauryl acrylate, butyl acrylate, and isooctyl acrylate.

In a specific embodiment of the present invention, the hard monomer comprises at least one of methyl acrylate, methyl methacrylate, isobornyl acrylate, ethyl methacrylate, styrene, and acrylonitrile.

5 In a specific embodiment of the present invention, the functional monomer comprises at least one of acrylic acid, acrylamide, N, N-dimethylacrylamide, hydroxyethyl acrylate, and hydroxybutyl acrylate.

10 In a specific embodiment of the present invention, the crosslinker comprises at least one of diethylene glycol diacrylate, triethylene glycol diacrylate, pentaerythritol triacrylate, and trimethylolpropane triacrylate.

The second aspect of the present invention provides a preparation method for the high-temperature and high-humidity resistant optically clear adhesive (OCA) provided by the first aspect of the present invention, comprising the following steps: uniformly mixing a soft monomer, a hard monomer, a functional monomer, a thiourea monomer, a polyurethane acrylate prepolymer, and a photoinitiator to obtain a first mixture; irradiating the first mixture with an ultraviolet light for 3 minutes to 5 minutes; then adding a chain transfer agent, a tackifier coupling agent, an antioxidant, a levelling agent, and a crosslinker into the irradiated first mixture, and uniformly mixing to obtain a second mixture; followed by coating the second mixture and curing under the ultraviolet light.

25 In a specific embodiment of the present invention, the OCA has a thickness of 50 μm to 300 μm .

The third aspect of the present invention provides a use of the high-temperature and high-humidity resistant OCA provided in the first aspect of the present invention in display screens.

In comparison with the prior art, the present invention has the following beneficial effects:

30 (1) The OCA of the present invention not only exhibits high light transmittance, but also maintains excellent adhesion, stability, and durability under extreme high temperature and high humidity conditions, thereby preventing bubbling or delamination caused by moisture penetration, and reducing the likelihood of deformation or failure;

- (2) The preparation method for the OCA of the present method omits the use of organic solvents, and is simple to carry out, thereby meeting the requirements of excellent performance and environmental friendliness.

5 Detailed Description of Invention

The technical scheme of the present invention will be clearly and completely described below in conjunction with specific embodiments, but it will be appreciated by those skilled in the art that the following described examples are part of embodiments of the present invention, rather than all embodiments, and are only used to illustrate the present invention, and should not be considered as limiting the scope of the present invention. All other embodiments obtained by those skilled in the art based on the embodiments of the present invention without any inventive effort shall fall within the scope of protection of the present invention. For those conditions not specifically described in the embodiments, conventional conditions or those recommended by the manufacturer are used. For reagents or instruments whose manufacturers are not specifically mentioned, they are conventional products that are commercially available.

The first aspect of the present invention describes a high-temperature and high-humidity resistant optically clear adhesive (OCA), comprising the following components in parts by weight: 40 parts to 55 parts of a soft monomer, 10 parts to 25 parts of a hard monomer, 5 parts to 20 parts of a functional monomer, 5 parts to 15 parts of a thiourea monomer, 3 parts to 15 parts of a polyurethane acrylate prepolymer, 0.2 parts to 2 parts of a photoinitiator, 0.05 parts to 2 parts of a chain transfer agent, 0.1 parts to 1 part of a tackifier coupling agent, 0.1 parts to 1.5 parts of an antioxidant, 0.05 parts to 1 part of a levelling agent, and 0.05 parts to 1.5 parts of a crosslinker; wherein the polyurethane acrylate prepolymer is primarily prepared by a reaction of a polyol, a polyisocyanate, and a hydroxyl-containing acrylate; the polyol comprises polytetramethylene ether glycol and polycaprolactone diol; and the polyisocyanate comprises isophorone diisocyanate and hexamethylene diisocyanate.

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The OCA of the present invention not only exhibits high light transmittance, but also maintains excellent adhesion, stability, and durability under extreme high temperature and

high humidity conditions, thereby preventing bubbling or delamination caused by moisture penetration and reducing the likelihood of deformation or failure.

5 The OCA of the present invention incorporates a certain amount of thiourea monomer and polyurethane acrylate (PUA) prepolymer. The addition of the PUA prepolymer introduces the polyurethane segment with excellent thermal stability and hydrolysis resistance into the OCA system. The acrylate segment polymerizes with various available monomers to form a cross-linked network structure. In combination with the thiourea monomer, which introduces a greater amount of hydrophobic groups and intermolecular hydrogen bonds, the high
10 temperature and high humidity resistance of the OCA is significantly improved, while maintaining high light transmittance of the OCA.

Based on the use of the aforementioned components, the proportions are adjusted in the present invention to ensure high light transmittance and improved resistance against high
15 temperature and high humidity of the OCA. For example, in the high-temperature and high-humidity resistant OCA in different embodiments, the amounts of each component, in parts by weight, are as follows:

The amount of the soft monomer may be 40 parts, 42 parts, 45 parts, 48 parts, 50 parts, 52 parts, 55 parts, or a range formed by any two of these values;

20 The amount of the hard monomer may be 10 parts, 12 parts, 15 parts, 18 parts, 20 parts, 22 parts, 25 parts, or a range formed by any two of these values;

The amount of the functional monomer may be 5 parts, 8 parts, 10 parts, 12 parts, 15 parts, 18 parts, 20 parts, or a range formed by any two of these values;

25 The amount of the thiourea monomer may be 5 parts, 8 parts, 10 parts, 12 parts, 15 parts, or a range formed by any two of these values;

The amount of the PUA prepolymer may be 3 parts, 5 parts, 8 parts, 10 parts, 12 parts, 15 parts, or a range formed by any two of these values;

The amount of the photoinitiator may be 0.2 parts, 0.5 parts, 0.8 parts, 1 part, 1.5 parts, 2 parts, or a range formed by any two of these values;

30 The amount of the chain transfer agent may be 0.05 parts, 0.1 parts, 0.5 parts, 1 part, 1.5 parts, 2 parts, or a range formed by any two of these values;

The amount of the tackifier coupling agent may be 0.1 parts, 0.3 parts, 0.5 parts, 0.8 parts, 1 part, or a range formed by any two of these values;

The amount of the antioxidant may be 0.1 parts, 0.5 parts, 1 part, 1.5 parts, or a range formed by any two of these values;

The amount of the levelling agent may be 0.05 parts, 0.1 parts, 0.5 parts, 0.8 parts, and 1 part, or a range formed by any two of these values; and

5 The amount of the crosslinker may be 0.05 parts, 0.1 parts, 0.5 parts, 1 part, and 1.5 parts, or a range formed by any two of these values.

In a specific embodiment of the present invention, the polytetramethylene ether glycol and the polycaprolactone diol in the polyol are present at a molar ratio of 1:(0.5-2), for example,
10 1:0.5, 1:0.8, 1:1, 1:1.2, 1:1.5, 1:1.8, 1:2, or a range formed by any two of these values, thereby improving while balancing the performance of the OCA, including high temperature and high humidity resistance, adhesion strength, etc.

In a specific embodiment of the present invention, the polytetramethylene ether glycol has a
15 number-average molecular weight of 1000 to 3000, while the polycaprolactone diol has a number-average molecular weight of 1000 to 3000.

In a specific embodiment of the present invention, the isophorone diisocyanate and the hexamethylene diisocyanate in the polyisocyanate are present at a molar ratio of 1:(0.5-2),
20 for example, 1:0.5, 1:0.8, 1:1, 1:1.2, 1:1.5, 1:1.8, 1:2, or a range formed by any two of these values, thereby improving while balancing the performance of the OCA, including high temperature and high humidity resistance, adhesion strength, etc.

In a specific embodiment of the present invention, the polyol and the polyisocyanate are
25 present at a molar ratio of 1:(1.2-1.5), for example, 1:1.2, 1:1.3, 1:1.4, 1:1.5, or a range formed by any two of these values.

In a specific embodiment of the present invention, the polyol and the hydroxyl-containing acrylate are present at a molar ratio of 1:(0.8-1.2), for example, 1:0.8, 1:0.9, 1:1, 1:1.1, and
30 1:1.2, or a range formed by any two of these values. Furthermore, the hydroxyl-containing acrylate comprises at least one of 2-hydroxyethyl acrylate, 4-hydroxybutyl acrylate, and hydroxypropyl acrylate.

In a specific embodiment of the present invention, the preparation of the polyurethane acrylate prepolymer is carried out in the presence of a catalyst. Furthermore, the catalyst comprises at least one of dibutyltin dilaurate, triethylenediamine, and stannous octoate. In practical operation, the catalyst is used in a conventional amount. For example, the amount of the catalyst may be 0.02 wt% to 1 wt% based on the total amount of the reactants.

In a specific embodiment of the present invention, the reaction for preparing the polyurethane acrylate prepolymer further comprise a chain extender and/or a filler as raw materials.

In a specific embodiment of the present invention, the amount of the chain extender is 0.5 wt% to 2 wt% based on the total amount of the polyol, the polyisocyanate, and the hydroxyl-containing acrylate. For example, the amount of the chain extender may be 0.5 wt%, 0.8 wt%, 1 wt%, 1.2 wt%, 1.5 wt%, 1.8 wt%, 2 wt%, or a range formed by any two of these values, thereby further improving the cross-linking structure of the OCA and enhancing the high temperature and high humidity resistance. Furthermore, the chain extender is 1,4-butanediol and/or 1,6-hexanediol.

In a specific embodiment of the present invention, the amount of the filler is 8 wt% to 12 wt% based on the total amount of the polyol, the polyisocyanate, and the hydroxyl-containing acrylate. For example, the amount of the filler may be 8 wt%, 9 wt%, 10 wt%, 11 wt%, 12 wt%, or a range formed by any two of these values. This is particularly beneficial for simultaneously ensuring high light transmittance and enhancing high-temperature and high-humidity resistance of the OCA. Furthermore, the filler is at least one of nanocellulose, nanoalumina, and nanosilica. The filler has a particle size of 1 nm to 50 nm.

In a specific embodiment of the present invention, the preparation of the PUA prepolymer comprises steps of: reacting the polyol and the polyisocyanate in the presence of the catalyst at 65 °C to 75 °C for 2 hours to 4 hours, followed by adding the hydroxyl-containing acrylate, the filler, and the chain extender, and continuing the reaction at 65 °C to 75 °C for another 2 hours to 3 hours to obtain the PUA prepolymer.

In a specific embodiment of the present invention, the thiourea monomer comprises at least one of 1,3-diallyl 2-thiourea, allyl thiourea, 1-allyl-3-(4-benzylphenyl)thiourea, 1-allyl-3-

(pyridin-3-yl)thiourea, 1-allyl-3,3-diethyl-2-thiourea, 1-methallyl-3-methyl-2-thiourea, and N-propylene-N'-2-hydroxyethylthiourea.

5 In a specific embodiment of the present invention, the thiourea monomer and the PUA prepolymer are present at a mass ratio of (0.5-3):1, for example, 0.5:1, 1:1, 1.5:1, 2:1, 2.5:1, or 3:1, or more preferably (1-2.5):1, thereby further enhancing the high temperature and high humidity resistance, etc.

10 In a specific embodiment of the present invention, the soft monomer has a glass transition temperature of -60 °C to -10 °C. Furthermore, the soft monomer comprises at least one of ethyl acrylate, 2-ethylhexyl acrylate, lauryl acrylate, butyl acrylate, and isooctyl acrylate.

15 In a specific embodiment of the present invention, the hard monomer comprises at least one of methyl acrylate, methyl methacrylate, isobornyl acrylate, ethyl methacrylate, styrene, and acrylonitrile.

20 In a specific embodiment of the present invention, the functional monomer comprises at least one of acrylic acid, acrylamide, N, N-dimethylacrylamide, hydroxyethyl acrylate, and hydroxybutyl acrylate.

25 In a specific embodiment of the present invention, the photoinitiator comprises, but is not limited to, at least one of benzophenone, benzoin dimethyl ether, methyl benzoylformate, 2-benzyl-2-dimethylamino-1-(4-morpholinophenyl)-1-butanone, 2-hydroxy-2-methyl-1-phenylpropan-1-one, 2,4,6-trimethylbenzoyl-diphenylphosphine oxide, 1-hydroxy-cyclohexyl benzophenone, 2,4-diethylthioxanthone, and α -hydroxyisobutyrophenone.

30 In a specific embodiment of the present invention, the chain transfer agent comprises, but is not limited to, at least one of thioglycolic acid, dodecyl mercaptan, isooctyl mercaptoacetate, 2-mercaptoethanol, isooctyl 3-mercaptopropionate, and pentaerythritol tetrakis(3-mercaptopbutyrate).

In a specific embodiment of the present invention, the tackifier coupling agent comprises, but is not limited to, at least one of vinyltriethoxysilane, dimethyldimethoxysilane,

vinyltrimethoxysilane, 3-(methacryloyloxy)propyltrimethoxysilane, methyltrimethoxysilane, and methyltriethoxysilane.

5 In a specific embodiment of the present invention, the antioxidant comprises, but is not limited, to at least one of tetrakis[β -(3,5-di-tert-butyl-4-hydroxyphenyl)propionate]pentaerythritol ester, 2,6-di-tert-butyl-p-cresol, 2,2'-methylenebis-(4-methyl-6-tert-butylphenol), 1,1,3-tris(2-methyl-4-hydroxy-5-tert-butylphenyl)butane, 4,4'-thiobis(6-tert-butyl-3-methylphenol), and N,N-diphenyl-p-phenylenediamine.

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In a specific embodiment of the present invention, the levelling agent comprises, but is not limited to, at least one of Troysol S366, Modaflow 2100, TEGO Glide 435, BYK370, BYK333, TEGO Flow ZFS 460, and Perenol S71 UV.

15 In a specific embodiment of the present invention, the crosslinker comprises at least one of diethylene glycol diacrylate, triethylene glycol diacrylate, pentaerythritol triacrylate, and trimethylolpropane triacrylate.

20 The second aspect of the present invention provides a method for preparing the high-temperature and high-humidity resistant optically clear adhesive described in the first aspect of the present invention, comprising the following steps: uniformly mixing a soft monomer, a hard monomer, a functional monomer, a thiourea monomer, a polyurethane acrylate prepolymer, and a photoinitiator to obtain a first mixture; irradiating the first mixture with an ultraviolet light for 3 minutes to 5 minutes; adding a chain transfer agent, a tackifier coupling agent, an antioxidant, a levelling agent, and a crosslinker to the irradiated first mixture, and uniformly mixing to obtain a second mixture; and coating the second mixture and curing with ultraviolet light.

25 In a specific embodiment of the present invention, the wavelength of the ultraviolet light may be 280 nm to 420 nm.

30 In a specific embodiment of the present invention, in the curing step with the ultraviolet light, the energy is in the range of 2500 mj/cm² to 3500 mj/cm², for example, 2500 mj/cm², 2800

mj/cm², 3000 mj/cm², 3200 mj/cm², 3500 mj/cm², or a range formed by any two of these values.

5 In a specific embodiment of the present invention, the OCA has a thickness of 50 μm to 300 μm, for example, 50 μm, 100 μm, 150 μm, 200 μm, 250 μm, 300 μm, or a range formed by any two of these values. The specific thickness range may be adjusted according to actual requirements, while ensuring that both high light transmittance and high temperature and high humidity resistance are maintained within this thickness range.

10 In a specific embodiment of the present invention, the curing with the ultraviolet light after coating comprises steps of: coating the second mixture on the release surface of a heavy release film, adhering a light release film thereto, and then curing it with the ultraviolet light.

15 In practical operation, the coating may be carried out by conventional coating methods, either manually or by using a coating machine.

In a specific embodiment of the present invention, the heavy release film and the light release film, independently, have a thickness of 50 μm to 100 μm, for example, 50 μm, 60 μm, 75 μm, 80 μm, 90 μm, 100 μm, or a range formed by any two of these values.

20 In a specific embodiment of the present invention, the heavy release film has a release force of 20 gf/in to 40 gf/in, while the light release film has a release force of 3 gf/in to 10 gf/in.

25 The third aspect of the present invention provides a use of the high-temperature and high-humidity resistant OCA provided in the first aspect of the present invention in display screens.

Example 1:

30 The present example provides a high-temperature and high-humidity resistant OCA, comprising the following components in parts by weight: 45 parts of lauryl acrylate, 19 parts of isobornyl acrylate, 15 parts of hydroxybutyl acrylate, 15 parts of allyl thiourea, 5 parts of PUA prepolymer, 0.3 parts of methyl benzoylformate as the photoinitiator, 0.09 parts of dodecyl mercaptan, 0.2 parts of vinyltriethoxysilane, 0.11 parts of tetrakis[β-(3,5-di-tert-

butyl-4-hydroxyphenyl)propionate]pentaerythritol ester as the antioxidant, 0.2 parts of Modaflow 2100 as the levelling agent, and 0.1 parts of triethylene glycol diacrylate.

Among them, the preparation method of the PUA prepolymer comprises steps of: adding 100 g of polycaprolactone diol with a number-average molecular weight of 2000 and 100 g of polytetramethylene ether glycol with a number-average molecular weight of 2000 into an anaerobic reactor; adding in polyisocyanate, such that the total molar ratio of polycaprolactone diol and polytetramethylene ether glycol to polyisocyanate is 1:1.3; heating up to 70 °C; adding in 0.2 g of dibutyltin dilaurate, and reacting for 2.5 hours under continuous stirring; adding in 11.612 g of 2-hydroxyethyl acrylate, nanocellulose (with average particle size of 10 nm), and 1,4-butanediol, and continue reacting for 2 h with continuous stirring at 70 °C; cooling to room temperature, and storing away from light after sealing. Particularly, the polyisocyanate comprises the hexamethylene diisocyanate and the isophorone diisocyanate at a molar ratio of 1:1; the added amount of the nanocellulose is 10 wt% based on the total amount of polycaprolactone diol, polytetramethylene ether glycol, polyisocyanate, and 2-hydroxyethyl acrylate; and the added amount of the 1,4-butanediol is 1 wt% based on the total amount of polycaprolactone diol, polytetramethylene ether glycol, polyisocyanate, and 2-hydroxyethyl acrylate.

The present example provides a method for preparing a high-temperature and high-humidity resistant OCA, comprising the following steps of: uniformly mixing lauryl acrylate as the soft monomer, isobornyl acrylate as the hard monomer, hydroxybutyl acrylate as the functional monomer, allyl thiourea as the thiourea monomer, the PUA prepolymer, and the photoinitiator; irradiating the resulting mixture with an ultraviolet light at a wavelength of 365 nm for 4 minutes; then adding dodecyl mercaptan as the chain transfer agent, vinyltriethoxysilane as the tackifier coupling agent, tetrakis[β -(3,5-di-tert-butyl-4-hydroxyphenyl)propionate]pentaerythritol ester as the antioxidant, Modaflow as the levelling agent, and triethylene glycol diacrylate as the crosslinker, and mixing them uniformly to obtain a mixture, coating the mixture on the release surface of a heavy release film with a thickness of 75 μm (release force at 30 gf/in), followed by adhering a light release film (release force at 5 gf/in) thereon, and then curing the coated film under ultraviolet light at a wavelength of 365 nm with a total energy of 3000 mj/cm^2 . An OCA with a thickness of 200 μm is obtained.

Example 2:

The present example refers to the high-temperature and high-humidity resistant OCA and the preparation method thereof as described in Example 1, with the only differences being the amounts of the components and the type of the thiourea monomer.

The high-temperature and high-humidity resistant OCA of the present example, comprises the following components in parts by weight: 47 parts of lauryl acrylate, 17 parts of isobornyl acrylate, 15 parts of hydroxybutyl acrylate, 12 parts of 1-allyl-3-(4-benzylphenyl)thiourea, 8 parts of PUA prepolymer, 0.3 parts of methyl benzoylformate as the photoinitiator, 0.09 parts of dodecyl mercaptan, 0.2 parts of vinyltriethoxysilane, 0.11 parts of tetrakis[β -(3,5-di-tert-butyl-4-hydroxyphenyl)propionate]pentaerythritol ester as the antioxidant, 0.2 parts of Modaflow 2100 as the levelling agent, and 0.1 parts of triethylene glycol diacrylate.

Example 3:

The present example refers to the high-temperature and high-humidity resistant OCA and the preparation method thereof as described in Example 1, with the only differences being the amounts of the components and the type of the thiourea monomer.

The high-temperature and high-humidity resistant OCA of the present example, comprises the following components in parts by weight: 50 parts of lauryl acrylate, 20 parts of isobornyl acrylate, 14 parts of hydroxybutyl acrylate, 10 parts of 1-allyl-3-(4-benzylphenyl)thiourea, 5 parts of PUA prepolymer, 0.3 parts of methyl benzoylformate as the photoinitiator, 0.09 parts of dodecyl mercaptan, 0.2 parts of vinyltriethoxysilane, 0.11 parts of tetrakis[β -(3,5-di-tert-butyl-4-hydroxyphenyl)propionate]pentaerythritol ester as the antioxidant, 0.2 parts of Modaflow 2100 as the levelling agent, and 0.1 parts of triethylene glycol diacrylate.

Example 4:

The present example refers to the high-temperature and high-humidity resistant OCA and the preparation method thereof as described in Example 1, with the only differences being the amounts of the components and the type of the thiourea monomer.

The high-temperature and high-humidity resistant OCA of the present example, comprising the following components in parts by weight: 45 parts of lauryl acrylate, 20 parts of isobornyl acrylate, 14 parts of hydroxybutyl acrylate, 10 parts of 1-allyl-3-(4-benzylphenyl)thiourea, 10 parts of PUA prepolymer, 0.3 parts of methyl benzoylformate as the photoinitiator, 0.09 parts of dodecyl mercaptan, 0.2 parts of vinyltriethoxysilane, 0.11 parts of tetrakis[β -(3,5-di-tert-butyl-4-hydroxyphenyl)propionate]pentaerythritol ester as the antioxidant, 0.2 parts of Modaflow 2100 as the levelling agent, and 0.1 parts of triethylene glycol diacrylate.

10 Example 5:

The present example refers to the high-temperature and high-humidity resistant OCA and the preparation method thereof as described in Example 4, with the only differences being the amounts of the thiourea monomer and the PUA prepolymer.

15 The amount of 1-allyl-3-(4-benzylphenyl)thiourea as the thiourea monomer used in the present example is 5 parts by weight, while the amount of PUA prepolymer used is 15 parts by weight.

20 Example 6:

The present example refers to the high-temperature and high-humidity resistant OCA and the preparation method thereof as described in Example 4, with the only differences being the amounts of the thiourea monomer and the PUA prepolymer.

25 The amount of 1-allyl-3-(4-benzylphenyl)thiourea as the thiourea monomer used in the present example is 15 parts by weight, while the amount of the PUA prepolymer used is 5 parts by weight.

30 Comparative Example 1:

Comparative Example 1 refers to the high-temperature and high-humidity resistant OCA and the preparation method thereof as described in Example 1, with the only differences being

the amounts of the components and the omission of the thiourea monomer and the PUA prepolymer.

5 The high-temperature and high-humidity resistant OCA of Comparative Example 1, comprises the following components in parts by weight: 55 parts of lauryl acrylate, 25 parts of isobornyl acrylate, 19 parts of hydroxybutyl acrylate, 0.3 parts of methyl benzoylformate as the photoinitiator, 0.09 parts of dodecyl mercaptan, 0.2 parts of vinyltriethoxysilane, 0.11 parts of tetrakis[β -(3,5-di-tert-butyl-4-hydroxyphenyl)propionate]pentaerythritol ester as the antioxidant, 0.2 parts of Modaflow 2100 as the levelling agent, and 0.1 parts of triethylene glycol diacrylate.

Comparative Example 2:

15 Comparative Example 2 refers to the high-temperature and high-humidity resistant OCA and the preparation method thereof as described in Example 1, with the only differences being the amounts of the components and the type of PUA prepolymer.

20 The high-temperature and high-humidity resistant OCA of Comparative Example 2, comprises the following components in parts by weight: 50 parts of lauryl acrylate, 20 parts of isobornyl acrylate, 14 parts of hydroxybutyl acrylate, 10 parts of 1-allyl-3-(4-benzylphenyl)thiourea, 5 parts of PUA prepolymer, 0.3 parts of methyl benzoylformate as the photoinitiator, 0.09 parts of dodecyl mercaptan, 0.2 parts of vinyltriethoxysilane, 0.11 parts of tetrakis[β -(3,5-di-tert-butyl-4-hydroxyphenyl)propionate]pentaerythritol ester as the antioxidant, 0.2 parts of Modaflow 2100 as the levelling agent, and 0.1 parts of triethylene glycol diacrylate.

30 The preparation method of the PUA prepolymer refers to that as described in Example 1, with the following differences: the 100 g of the polycaprolactone diol with a number-average molecular weight of 2000 and the 100 g of the polytetramethylene ether glycol with a number-average molecular weight of 2000 in Example 1 are replaced with 200 g of the polycaprolactone diol with a number-average molecular weight of 2000; and the hexamethylene diisocyanate and the isophorone diisocyanate in Example 1 are replaced with an equimolar hexamethylene diisocyanate.

Comparative Example 3:

Comparative Example 3 refers to the high-temperature and high-humidity resistant OCA and the preparation method thereof as described in Example 1, with the only difference being the amounts of the components and the type of PUA prepolymer.

The high-temperature and high-humidity resistant OCA of Comparative Example 3, comprises the following components in parts by weight: 50 parts of lauryl acrylate, 20 parts of isobornyl acrylate, 14 parts of hydroxybutyl acrylate, 10 parts of 1-allyl-3-(4-benzylphenyl)thiourea, 5 parts of PUA prepolymer, 0.3 parts of methyl benzoylformate as the photoinitiator, 0.09 parts of dodecyl mercaptan, 0.2 parts of vinyltriethoxysilane, 0.11 parts of tetrakis[β -(3,5-di-tert-butyl-4-hydroxyphenyl)propionate]pentaerythritol ester as the antioxidant, 0.2 parts of Modaflow 2100 as the levelling agent, and 0.1 parts by weight of triethylene glycol diacrylate.

The preparation method of the PUA prepolymer refers to that as described in Example 1, with the following differences: the 100 g of polycaprolactone diol with a number-average molecular weight of 2000 and the 100 g of polytetramethylene ether glycol with a number-average molecular weight of 2000 in Example 1 are replaced with 200 g of polytetramethylene ether glycol with a number-average molecular weight of 2000; and the hexamethylene diisocyanate and the isophorone diisocyanate in Example 1 are replaced with an equimolar hexamethylene diisocyanate.

Comparative Example 4:

Comparative Example 4 refers to the high-temperature and high-humidity resistant OCA and the preparation method thereof as described in Example 1, with the only differences being the amounts of the components and the type of PUA prepolymer.

The high-temperature and high-humidity resistant OCA of Comparative Example 4, comprises the following components in parts by weight: 50 parts of lauryl acrylate, 20 parts of isobornyl acrylate, 14 parts of hydroxybutyl acrylate, 10 parts of 1-allyl-3-(4-benzylphenyl)thiourea, 5 parts of PUA prepolymer, 0.3 parts of methyl benzoylformate as

the photoinitiator, 0.09 parts of dodecyl mercaptan, 0.2 parts of vinyltriethoxysilane, 0.11 parts of tetrakis[β -(3,5-di-tert-butyl-4-hydroxyphenyl)propionate]pentaerythritol ester as the antioxidant, 0.2 parts of Modaflow 2100 as the levelling agent, and 0.1 parts of triethylene glycol diacrylate.

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The preparation method of the PUA prepolymer refers to that as described in Example 1, with the difference that the hexamethylene diisocyanate and the isophorone diisocyanate in Example 1 are replaced with an equimolar hexamethylene diisocyanate.

10 Comparative Example 5:

Comparative Example 5 refers to the high-temperature and high-humidity resistant OCA and the preparation method thereof as described in Example 1, with the differences being the amounts of the components, and the omission of the PUA prepolymer.

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The high-temperature and high-humidity resistant OCA of Comparative Example 5, comprises the following components in parts by weight: 53 parts of lauryl acrylate, 21 parts of isobornyl acrylate, 15 parts of hydroxybutyl acrylate, 10 parts of 1-allyl-3-(4-benzylphenyl)thiourea, 0.3 parts of methyl benzoylformate as the photoinitiator, 0.09 parts of dodecyl mercaptan, 0.2 parts of vinyltriethoxysilane, 0.11 parts of tetrakis[β -(3,5-di-tert-butyl-4-hydroxyphenyl)propionate]pentaerythritol ester as the antioxidant, 0.2 parts of Modaflow 2100 as the levelling agent, and 0.1 parts of triethylene glycol diacrylate.

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Comparative Example 6:

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Comparative Example 6 refers to the high-temperature and high-humidity resistant OCA and the preparation method thereof as described in Example 1, with the differences being the amounts of the components, and the omission of the PUA prepolymer.

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The high-temperature and high-humidity resistant OCA of Comparative Example 6, comprises the following components in parts by weight: 55 parts of lauryl acrylate, 23 parts of isobornyl acrylate, 16 parts of hydroxybutyl acrylate, 5 parts of PUA prepolymer, 0.3 parts of methyl benzoylformate as the photoinitiator, 0.09 parts of dodecyl mercaptan, 0.2 parts of

vinyltriethoxysilane, 0.11 parts of tetrakis[β -(3,5-di-tert-butyl-4-hydroxyphenyl)propionate]pentaerythritol ester as the antioxidant, 0.2 parts of Modaflow 2100 as the levelling agent, and 0.1 parts of triethylene glycol diacrylate.

5 Experimental Example:

The following performance tests were conducted on the OCAs prepared in the different examples and comparative examples, with the test results shown in Table 1.

10 Optical performance test: According to the adhered structure of a 7-inch glass cover/OCA/matching ITO sheet, the test is carried out according to GB/T 2410-2008 "Determination of light transmittance and haze of transparent plastics".

15 Gel content test: The OCA is cut into small pieces of similar sizes, with half of them post-cured at 5000 mj/cm². The OCA is wrapped around a glass rod, and its initial mass is recorded as Ms before the OCA-wrapped glass rod is placed into a beaker filled with 250 mL of ethyl acetate. The solvent is filtered with a 300-mesh filter after 24 hours, while the remaining OCA is dried in an oven at 120°C for 30 minutes. The remaining weight is recorded as Me, and the gel content (G) is calculated by the following formula: $G = Me/Ms$.

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Table 1: The performance test results for the respective OCA

Number	Light transmittance (%)	Haze	Yellowness	Gel content before UV curing (%)	Gel content after UV curing (%)
Example 1	93.1	0.12	0.13	61.07	74.54
Example 2	93.0	0.11	0.13	61.55	73.66
Example 3	93.2	0.13	0.14	61.32	74.59
Example 4	92.9	0.14	0.15	62.51	75.03
Example 5	93.1	0.13	0.15	61.77	74.61
Example 6	93.0	0.14	0.14	62.02	74.89
Comparative Example 1	93.2	0.11	0.14	60.01	72.15

Comparative Example 2	92.8	0.13	0.13	59.92	72.33
Comparative Example 3	93.0	0.12	0.15	60.18	72.45
Comparative Example 4	92.9	0.13	0.13	60.48	72.19
Comparative Example 5	93.1	0.13	0.14	59.92	71.94
Comparative Example 6	93.2	0.12	0.13	59.61	71.68

From the test results in Table 1, it can be seen that the OCAs of the present invention have better optical performances and cross-linking effects.

- 5 The following performance tests were further conducted on OCAs prepared in the different examples and comparative examples, with the test results shown in Table 2.

Adhesion test: The light release films of each OCA are torn off before sticking them on a 50 μm transparent PET film, respectively. A manual sampler is used to cut them into samples with a width of 25.4 mm and a length of 200 mm. After tearing off the heavy release film, each sample is stuck on a SUS 304 steel plate. A 2 kg roller is used to roll on each sample back and forth 3 times at a speed of 600 mm/min. The samples are respectively placed in environments of 25 °C, 60 °C, 90 °C, 60 °C/90 %RH, and 85 °C/85 %RH for 20 min, and then the 180° peeling adhesion is tested using a tensile testing machine.

Table 2: The peeling force test results of the respective OCA under different conditions

Number	25 °C	60 °C	90 °C	60 °C/90% RH	85 °C/85% RH
Example 1	19.891	16.911	15.294	8.684	8.754
Example 2	19.855	16.864	14.985	8.786	8.591
Example 3	19.532	16.684	14.799	8.631	8.031
Example 4	20.064	17.042	15.943	9.451	9.355
Example 5	19.521	15.941	14.024	8.015	7.482
Example 6	19.423	15.152	13.551	7.647	7.035

Comparative Example 1	18.011	15.565	11.864	5.551	5.612
Comparative Example 2	18.300	15.661	11.994	5.612	5.431
Comparative Example 3	18.401	15.812	12.862	5.785	5.881
Comparative Example 4	18.641	16.266	13.011	6.032	6.154
Comparative Example 5	17.791	15.001	12.041	5.175	4.674
Comparative Example 6	17.045	14.456	11.784	4.978	4.048

From the test results in Table 2, it can be seen that the OCAs of the present invention have excellent resistance to high temperature and high humidity, can maintain relatively better cohesion under high temperature and high humidity conditions, and have greater adhesion.

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The ageing resistance of the OCAs prepared in the different examples and comparative examples was further tested, with the test results shown in Table 3.

10 Aging resistance test: Each OCA is adhered to the glass cover plate, and after defoaming, it is treated using a LED light with a total energy of 3000 mj/cm². Each OCA is then placed in environments of 25 °C, 90 °C, 110 °C, 85 °C/85 %RH, and 110 °C/90 %RH for 1000 h, respectively. Within this period, the samples are observed for the presence of bubbles or other phenomena. If there are bubbles, the sample is marked as ×; otherwise, it is marked as √.

15 Table 3: The ageing resistance test results of the respective OCA

Number	25 °C – 1000 h	90 °C – 1000 h	110 °C – 1000 h	85 °C/85% RH – 1000 h	110 °C/90% RH – 1000 h
Example 1	√	√	√	√	√
Example 2	√	√	√	√	√
Example 3	√	√	√	√	√
Example 4	√	√	√	√	√
Example 5	√	√	√	√	√
Example 6	√	√	√	√	√
Comparative Example 1	√	×	×	×	×
Comparative Example 2	√	√	×	√	×

Comparative Example 3	√	√	×	√	×
Comparative Example 4	√	√	√	√	×
Comparative Example 5	√	√	×	×	×
Comparative Example 6	√	√	×	×	×

From the test results in Table 3, it can be seen that the OCAs of the present invention have excellent ageing resistance without bubble reappearance and adhesive failure in high temperature and high humidity environments.

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In summary, the light transmittance of the OCA of the present invention is more than 90% and the haze is less than 1. In a high temperature and high humidity environment (temperature at 100 °C to 150 °C, humidity at 60% to 90%), the absence of bubble reappearance and adhesive failure is maintained for 1000 hours, thereby showing excellent resistance of the OCA against high temperature and high humidity.

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Finally, it should be noted that the above examples are only used to illustrate the technical solutions of the present invention, rather than to limit it. Although the present invention has been described in detail with reference to the aforementioned examples, those skilled in the art should understand that they can still modify the technical solutions described in the aforementioned examples, or replace some or all of the technical features therein with equivalents. However, these modifications or replacements do not cause the essence of the corresponding technical solutions to deviate from the scope of the technical solutions of the examples of the present invention.

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Claims

1. A high-temperature and high-humidity resistant optically clear adhesive, comprising the following components in parts by weight:
 - 40 parts to 55 parts of a soft monomer;
 - 10 parts to 25 parts of a hard monomer;
 - 5 parts to 20 parts of a functional monomer;
 - 5 parts to 15 parts of a thiourea monomer;
 - 3 parts to 15 parts of a polyurethane acrylate prepolymer;
 - 0.2 parts to 2 parts of a photoinitiator;
 - 0.05 parts to 2 parts of a chain transfer agent;
 - 0.1 parts to 1 part of a tackifier coupling agent;
 - 0.1 parts to 1.5 parts of an antioxidant;
 - 0.05 parts to 1 part of a levelling agent; and
 - 0.05 parts to 1.5 parts of a crosslinker, wherein

the polyurethane acrylate prepolymer is primarily prepared by a reaction of a polyol, a polyisocyanate, and a hydroxyl-containing acrylate;

the polyol comprises polytetramethylene ether glycol and polycaprolactone diol, the polytetramethylene ether glycol and the polycaprolactone diol in the polyol are present at a molar ratio of 1:(0.5-2);

the polyisocyanate comprises isophorone diisocyanate and hexamethylene diisocyanate, the isophorone diisocyanate and the hexamethylene diisocyanate in the polyisocyanate are present at a molar ratio of 1:(0.5-2);

the thiourea monomer comprises at least one of 1,3-diallyl 2-thiourea, allyl thiourea, 1-allyl-3-(4-benzylphenyl)thiourea, 1-allyl-3-(pyridin-3-yl)thiourea, 1-allyl-3,3-diethyl-2-thiourea, 1-methallyl-3-methyl-2-thiourea, and N-allyl-N'-2-hydroxyethylthiourea; and

the functional monomer comprises at least one of acrylic acid, acrylamide, N,N-dimethylacrylamide, hydroxyethyl acrylate, and hydroxybutyl acrylate.
2. The high-temperature and high-humidity resistant optically clear adhesive according to claim 1, comprising at least one of the following characteristics:
 - (1) the polyol and the polyisocyanate are present at a molar ratio of 1:(1.2-1.5);

- (2) the polyol and the hydroxyl-containing acrylate are present at a molar ratio of 1:(0.8-1.2);
 - (3) the hydroxyl-containing acrylate comprises at least one of 2-hydroxyethyl acrylate, 4-hydroxybutyl acrylate, and hydroxypropyl acrylate;
 - (4) the polyurethane acrylate prepolymer is prepared in the presence of a catalyst, wherein the catalyst comprises at least one of dibutyltin dilaurate, triethylenediamine, and stannous octoate; and
 - (5) the reaction for preparing the polyurethane acrylate prepolymer further comprises a chain extender and/or a filler.
3. The high-temperature and high-humidity resistant optically clear adhesive according to claim 1, wherein the thiourea monomer and the polyurethane acrylate prepolymer are present at a mass ratio of (0.5-3):1.
4. The high-temperature and high-humidity resistant optically clear adhesive according to claim 1, comprising at least one of the following characteristics:
 - (1) the soft monomer comprises at least one of ethyl acrylate, 2-ethylhexyl acrylate, lauryl acrylate, and butyl acrylate;
 - (2) the hard monomer comprises at least one of methyl acrylate, methyl methacrylate, isobornyl acrylate, ethyl methacrylate, styrene, and acrylonitrile; and
 - (3) the crosslinker comprises at least one of diethylene glycol diacrylate, triethylene glycol diacrylate, pentaerythritol triacrylate, and trimethylolpropane triacrylate.
5. A method for preparing the high-temperature and high-humidity resistant optically clear adhesive according to any one of claims 1 to 4, comprising the steps of:

uniformly mixing a soft monomer, a hard monomer, a functional monomer, a thiourea monomer, a polyurethane acrylate prepolymer, and a photoinitiator to obtain a first mixture;

irradiating the first mixture with an ultraviolet light for 3 minutes to 5 minutes;

adding a chain transfer agent, a tackifier coupling agent, an antioxidant, a levelling agent, and a crosslinker to the irradiated first mixture, and uniformly mixing to obtain a second mixture; and

coating the second mixture and curing with ultraviolet light.

6. The method according to claim 5, wherein the optically clear adhesive has a thickness of 50 μm to 300 μm .
7. Use of a high-temperature and high-humidity resistant optically clear adhesive according to any one of claims 1 to 4, or a high-temperature and high-humidity resistant optically clear adhesive prepared by a preparation method according to any one of claims 5 to 6, in display screens.