

Claims

1. A method for silane coupling agent compounding modification and dispersing optimization for photovoltaic-grade submicron silica micro-powder, comprising the following steps:

5 drying a photovoltaic-grade submicron silica micro-powder under an inert atmosphere for 2-4 h, cooling to a room temperature, and then placing in a high-shear mixing reactor, wherein a surface hydroxyl content index of the photovoltaic-grade submicron silica micro-powder is 1.5–3.0 mmol/g;

10 adding a first silane coupling agent solution and a second silane coupling agent solution sequentially, wherein after the first silane coupling agent reacts for 1.5-2.5 h, the second silane coupling agent is added; and after the first silane coupling agent is reacted completely and a temperature is lowered to 70°C, the second silane coupling agent is added;

15 then adding a dispersion additive solution and continuing stirring at 70-90°C for 1.0-2.0 h;

removing a solvent through a reduced-pressure distillation after a reaction is completed, drying an obtained solid in a vacuum drying oven at 60°C for 6-12 h to obtain a modified silica micro-powder;

20 wherein the first silane coupling agent is 3-(2,3-epoxypropoxy)propyltrimethoxysilane, and a solution thereof is prepared by mixing the coupling agent with anhydrous ethanol at a mass ratio of 1:(8-12); the second silane coupling agent is N-(2-aminoethyl)-3-aminopropylmethyldimethoxysilane, and a solution thereof is prepared by mixing the coupling agent with anhydrous isopropanol at a mass ratio of 1:(10-15);

25 the dispersion additive is a polyether-modified polysiloxane, and the solution thereof is prepared by mixing the additive with anhydrous n-hexane at a mass ratio of 1:(20-30);

30 the polyether-modified polysiloxane has a number average molecular weight of 2000–4000 and an HLB value of 8-10, polyether chain segments thereof are copolymerized from ethylene oxide and propylene oxide in a molar ratio of 3:1, and an end-capping group is methyl; and

a reaction chamber of the high-shear mixing reactor is made of 316L stainless steel, and an inner wall is subjected to electrolytic polishing treatment to a surface roughness $Ra \leq 0.4 \mu\text{m}$.

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2. The method for silane coupling agent compounding modification and dispersing optimization for photovoltaic-grade submicron silica micro-powder according to claim 1, wherein an addition amount of the first silane coupling agent solution accounts for 1.5-3.0 wt% of a mass of the silica micro-powder, and an addition means is to add dropwise to a reaction system at a constant rate by a metering pump within 10-15 min, and a
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- system temperature is maintained at $65 \pm 2^\circ\text{C}$ during a dropwise addition process.
3. The method for silane coupling agent compounding modification and dispersing optimization for photovoltaic-grade submicron silica micro-powder according to claim 1, wherein an addition amount of the second silane coupling agent solution accounts for
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- 0.8-1.5 wt% of a mass of silica micro-powder, and after a reaction of the first silane coupling agent is completed and a temperature is cooled to 70°C , the second silane coupling agent solution is injected into the reactor in a form of a spray through a bottom feed port, wherein the spray has a pressure of 0.2-0.4 MPa, and an atomization particle size controlled within the range of 20–50 μm .
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4. The method for silane coupling agent compounding modification and dispersing optimization for photovoltaic-grade submicron silica micro-powder according to claim 1, wherein the high-shear mixing reactor is equipped with a double-layer jacketed temperature control system, an inner layer is a the reaction chamber and an outer layer
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- is charged with thermal oil for temperature regulation; and three layer of serrated dispersion discs are installed inside the reaction chamber, an upper disc is 5 cm away from a liquid level, a middle disc is located at a center of the liquid level, and a lower disc is 3 cm away from a bottom, an spacing between adjacent discs is equal, and a rotating shaft is installed vertically.
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5. The method for silane coupling agent compounding modification and dispersing

optimization for photovoltaic-grade submicron silica micro-powder according to claim 1, wherein the reduced-pressure distillation is carried out in a rotary evaporator until no condensate is produced, wherein a water bath temperature is 50-60°C, a vacuum degree is -0.095 to -0.098 MPa, and a distillation time is 40-60 min.

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6. The method for silane coupling agent compounding modification and dispersing optimization for photovoltaic-grade submicron silica micro-powder according to claim 1, wherein the vacuum drying oven is equipped with a porous stainless steel tray inside, the tray has a pore diameter of 0.5 mm and a porosity of 40%, and a paving thickness of the silica micro-powder is not more than 5 mm; and turning-over is performed on a material every 2 h during the drying process; and the turning-over is carried out by a means of a pulse purging with clean nitrogen gas at an air pressure of 0.1 MPa, each purging lasts for 10 s, and the purging is carried out 3 times in total.

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7. The method for silane coupling agent compounding modification and dispersing optimization for photovoltaic-grade submicron silica micro-powder according to claim 1, wherein the anhydrous ethanol, the anhydrous isopropanol and the anhydrous n-hexane are subjected to a molecular sieve dehydration treatment before use and are filtered through a 0.22 μm polytetrafluoroethylene membrane; and a metal ion content is confirmed by an ICP-MS detection, and the contents of sodium, iron and other metal ions and chloride ions are all below 0.1ppb.

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8. The method for silane coupling agent compounding modification and dispersing optimization for photovoltaic-grade submicron silica micro-powder according to claim 1, wherein the particle size D50 of the photovoltaic-grade submicron silica micro-powder is 0.4-0.6 μm , and the number average molecular weight of the polyether-modified polysiloxane is 2500-3500.

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9. A modified photovoltaic-grade submicron silica micro-powder according to any one of claims 1-8, wherein the surface thereof is grafted with a compound coating layer of 3-(2,3-epoxypropoxy)propyltrimethoxysilane and N-(2-aminoethyl)-3-

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aminopropylmethyldimethoxysilane, with a coating rate of 5-10 wt%, a sodium/iron/chloride ion content of ≤ 0.5 ppm, and a dispersion uniformity in EVA of ≤ 5 μm (agglomerate particle size).

- 5 10. The method for silane coupling agent compounding modification and dispersing optimization for photovoltaic-grade submicron silica micro-powder according to any one of claims 1-8, which is used in preparation of highly-filled composite materials for photovoltaic encapsulants, thermal interface materials or battery backsheets, wherein when the highly-filled composite materials are used for photovoltaic encapsulants, the yellowing index of the encapsulated parts is ≤ 1.0 (after 1000h aging), and when the highly-filled composite materials are used for the thermal interface material, the thermal conductivity is ≥ 1.8 W/(m·K).
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