Dispersion Properties of Aerogel Slurry and Application of Its Coatings in Thermal Insulation Engineering

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Abstract
Due to low thermal conductivity, SiO₂ aerogel has been widely applied in thermal insulation engineering. SiO₂ aerogel thermal insulation coating is the focus in the field of thermal insulation engineering, and the dispersion stability of aerogel in the coating is mainly related to its development. From the mechanism of dispersion, hydrophobic SiO₂ aerogel was modified to improve their dispersion performance in the aqueous medium. Research the influences of different kinds and dosages of wetting and dispersing agents, different dispersion conditions affect the dispersion performance of SiO₂ aerogel, and through particle size analysis and transmittance to analysis dispersion performance of SiO₂ aerogel slurry. The modified SiO₂ aerogel were characterized by Fourier translation infrared spectrum (FT-IR), scanning electron microscopy (SEM), X-ray diffractometer(XRD) and contact angle measurement. The results showed that organic silicon wetting agent and alkyl amine salt dispersant were added respectively with 10wt% of SiO₂ aerogel, the slurry has the best dispersion effect after dispersing at speed of 1800 r/min for 3 hours, the average particle size was about 80 nm. The surface of SiO₂ aerogel powder adsorbed dispersant after modification, and contact angle was from 145° decrease to 45°, it showed that hydrophilic of the hydrophobic SiO₂ aerogel has enhanced. Surface modification increases the hydrophobic SiO₂ aerogel compatibility in the water medium. At the same time, it reduced the reunion phenomenon in the slurry greatly and laid the foundation for preparation of SiO₂ aerogel thermal insulation coatings. On the basis of this, we studied the thermal insulation properties of SiO₂ aerogel thermal insulation coating, which provided basis for its application in thermal insulation engineering.

Key words: SiO₂ Aerogel, Mechanism, Surface Modification, Dispersion, Particle Size, Insulation Engineering

1. INTRODUCTION
SiO₂ aerogel is a nano lightweight porous material with complex three-dimensional network structure, its main component is air (Hrubesh, 1990; Pajonk, 1998), characterized by low thermal conductivity, low density, high porosity and large specific surface area, it has extensive application prospects (Bond and Saad, 1987; Smirnova and Arlt, 2004; Sun and Zhen; 2013). At present, due to low thermal conductivity and high transmittance, SiO₂ aerogel thermal insulation coatings has attached increasing attention (Budunoglu, 2011; Shi, 2013). SiO₂ aerogel is vital to the preparation of SiO₂ aerogel thermal insulation coating, and the performance of SiO₂ aerogel coating mainly depends on the uniformity and stability of the dispersion of SiO₂ aerogel in aqueous medium.

At present, solving the dispersion problem of nanoparticles is a hot spot and challenge in the preparation of SiO₂ aerogel thermal insulation coating, and poor dispersion performance will affect surface effect, size effect, quantum effect and volume effect of nano materials (Koch, 2007). In order to solve the dispersion problem of nanoparticles, the repulsion between nanoparticles shall be enhanced mainly by increasing solvation repulsion, electrostatic repulsion between nanoparticles and adding dispersants which could absorb and protect the surface of nanoparticles (Tie and Li, 2010; Suttiponparnit, 2010; Yuan and Zhou,2008). Based on the above dispersion mechanisms, main dispersion technologies (Jiang, Günter and Pratim, 2009; Tracton, 2005)can be divided into physical dispersion technologies (mechanical dispersion, ultrasonic dispersion and high energy treatment) and chemical dispersion technologies (dispersant and surface chemical modification).

Hydrophobic SiO₂ aerogel powder has good thermal insulation property, but due to large number of polar groups on the surface, its compatibility with aqueous medium is relatively poor. In addition, it is easy to agglomerate due to large surface energy and high interface thermal resistance, so it cannot disperse in aqueous medium evenly(Tai, 2008). Therefore, the surface of hydrophobic SiO₂ aerogel powder should be modified in the preparation of water-borne coating, to solve the dispersion problem in aqueous medium. This experiment
adopted dispersants to modify SiO₂ aerogel powders, and also used two wetting agents such as EH-9 and Silok 7117W, as well as four dispersants like polyethylene glycol (PEG-400), sodium acrylate (SN-5040), high molecular weight copolymer of alkyl amine (Silok 7195) and silane coupling agent (KH-550). We studied the effects of different wetting agents and dispersants as well as their dosages and dispersion conditions on the dispersion properties of SiO₂ aerogel slurry.

2. EXPERIMENT

2.1. Materials and Equipment

Hydrophobic SiO₂ aerogel manufactured by Alison Guangdong High Tech Co. Ltd.; wetting agent EH-9 by Dow Chemical; wetting agent Silok 7117 and dispersant Silok 7195 by Guangzhou Silok Chemical Co. Ltd.; dispersant PEG-400 and SN-5040 by Japan Science Nobel; dispersant KH-550 by Ji'nan's Guobang Chemical Co. Ltd.; deionized water made by the laboratory, waterborne acrylic resin and film-forming agent by Zuhai Jelee Chemical Co. Ltd.; defoamee by Rhodia, and Aluminum Alloy board bought on the market.

The equipment used in the experiment: SEM scanning electron microscope, Fourier transform infrared spectroscopy (FT-IR), X ray diffraction instrument, contact angle meter, UV-Vis spectrophotometer, sand milling and mixing multipurpose machine, ultrasonic cleaning machine, analytical balance and centrifuge.

2.2. Performance Testing

After SiO₂ aerogel slurry was cultured for 10 minutes, the supernatant was taken out and diluted to a certain concentration and then put into sample cell, and Zeta potential meter was used to measure particle size. 10ml SiO₂ aerogel slurry was put into centrifuge to centrifugalize for 5 minutes, and the rotation speed of centrifuge was set at 1500r/min, spectrophotometer was employed to measure the transmittance rate of slurry at 450 nm. Nicolet 6700 Fourier transform infrared spectroscopy (FT-IR) was used to detect the functional groups on the surface of SiO₂ aerogel powder, and samples were pressed into pellets with KBr. X ray diffraction instrument was used for phase analysis of SiO₂ aerogel powder, and the samples were pressed into 2cm * 2cm slices and then measured by Cu Kα ray. The modified SiO₂ aerogel powder was pressed into pellets, and contact angle meter was adopted to measure the contact angles between SiO₂ gas gel pellets and water droplets. Thermal insulation performance testing devices were made according to HC/T 4341-2012 Metal Surface Thermal Insulating Coatings, infrared lamp with power of 275 W was used to simulate sunlight, and the temperatures of bottom surfaces of samples were measured by thermocouple every 20 seconds until the temperatures remained stable.

3. RESULTS AND DISCUSSIONS

3.1. Modification Mechanism of SiO₂ Aerogel

The modification mechanism of SiO₂ aerogel is as shown in Figure 1. Low molecular silicone wetting agent was added into SiO₂ aerogel powder to reduce its surface tension, and thereby improving its wettability, so as to enhance its compatibility with aqueous medium; the SiO₂ aerogel was added with high molecular weight alkylamine salt copolymer dispersant, which prevented flocculation through steric hindrance effect, so as to disperse SiO₂ aerogel evenly. It can be learnt from the figure that wetting agent covers the surface of SiO₂ aerogel powder, to make SiO₂ aerogel powder enter into aqueous phase quickly; anchoring group on one of dispersant binds to the surface of SiO₂ aerogel tightly, and solvated chain on the other end promotes the dispersion of (Haubennestel and Peter, 1987) powder through steric hindrance effect. As super dispersant binds to the surface of powder particles through the anchoring of chemical bonds, modified SiO₂ aerogel powder disperses in aqueous slurry evenly with high stability.

![Figure 1. The sketch map of dispersion mechanism of SiO₂ aerogel](image-url)
3.2. Dispersion characteristics of SiO$_2$ aerogel slurry

SiO$_2$ aerogel slurries were added with 10% wetting agent and dispersants, respectively, and dispersed for 3 hours at shear rate of 1800 r/min after modification, the particle size distribution of SiO$_2$ aerogel slurry is as shown in Figure 2. From the figure, the dispersed particle size of SiO$_2$ aerogel slurry modified by wetting agent Silok 7117 was significantly smaller than that modified by wetting agent EH-9; as for SiO$_2$ aerogel wetted by the same wetting agent Silok 7117, the dispersion effect of super dispersant Silok 7195 was obviously superior than that of other three dispersants.

![Figure 2](image-url)

**Figure 2.** The influence of (a) wetting agent EH-9 and (b) wetting agent Silok 7117 on the dispersion

Table 1 shows the transmittances of SiO$_2$ aerogel slurries at 450nm. Transmittances can better reflect the dispersion property of SiO$_2$ aerogel, the higher the transmittance is, the more serious the agglomeration of SiO$_2$ aerogel will be. On the contrary, the lower the transmittance is, the higher the dispersion stability will be. From the table, when added with EH-9 wetting agent and four different types of dispersants, the transmittances of SiO$_2$ aerogel slurries were high, indicating the dispersion effect was poor. When added with wetting agent Silok 7117 and four different types of dispersants, the transmittance of SiO$_2$ aerogel slurries were low, suggesting that wetting agent Silok 7117 had better wetting effect to powder. Slurry added with Silok 7195 dispersant had the smallest average particle size, most uniform distribution and the lowest transmittance, indicating that SiO$_2$ aerogel was dispersed evenly, consistent with the results of slurry particle size analysis.

**Table 1.** The influence of different type of wetting agents and dispersants on the transmittance

<table>
<thead>
<tr>
<th>Wetting agents</th>
<th>Dispersants</th>
<th>Transmittance (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>EH-9</td>
<td>SN-5040</td>
<td>66</td>
</tr>
<tr>
<td>EH-9</td>
<td>PEG-400</td>
<td>50</td>
</tr>
<tr>
<td>EH-9</td>
<td>KH-550</td>
<td>47</td>
</tr>
<tr>
<td>EH-9</td>
<td>Silok 7195</td>
<td>42</td>
</tr>
<tr>
<td>Silok 7117</td>
<td>SN-5040</td>
<td>57</td>
</tr>
<tr>
<td>Silok 7117</td>
<td>PEG-400</td>
<td>40</td>
</tr>
<tr>
<td>Silok 7117</td>
<td>KH-550</td>
<td>37</td>
</tr>
<tr>
<td>Silok 7117</td>
<td>Silok 7195</td>
<td>26</td>
</tr>
</tbody>
</table>

Figure 3 shows the particle size distributions of SiO$_2$ aerogel slurries which were added with different dosages (referring to the percentages in SiO$_2$ aerogel) of wetting agent Silok 7117 and dispersant Silok 7195, and dispersed for 3 hours at shear rate of 1800 r/min. Table 2 shows the average particle size values. The
figure and table show that when added with same wetting agents, the average particle size of slurry decreases at first and then increases with the increase of dispersant. When added with dispersant weighting at 10% of SiO₂ aerogel powder, the average particle size of slurry was the minimum. In SiO₂ aerogel slurries added with small amount of dispersants, fewer anchoring groups of dispersant bind to the surface of SiO₂ aerogel, with smaller repulsive force generated by steric hindrance effect; but when added with excessive dispersant, Polymer solvated chains of dispersants entangle and flocculate, lowering dispersion effect (Cui and Xi, 1996). When added with same amount of dispersants, the average particle size of slurry decreases at first and then increases with the increase of wetting agents. When the wetting agent was less, it was not enough to cover all SiO₂ aerogel powder, weakening its affinity with water and thereby indirectly affecting the dispersion effect. The study also found that when added with wetting agent weighting at 10% of SiO₂ aerogel powder, the average particle size of slurry was the smallest-80.7nm.

![Figure 3](image-url)

**Figure 3.** Dispersion characteristics of different dispersants when added with wetting agent weighting at (a)5%, (b)10%, (c)15%, (d)20% of SiO₂ aerogel powder

**Table 2.** The influence of different dosages of wetting agent and dispersant on the average particle size

<table>
<thead>
<tr>
<th>Wetting agent (ωt %)</th>
<th>Dispersant (ωt %)</th>
<th>average particle size (nm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>5</td>
<td>5</td>
<td>184</td>
</tr>
<tr>
<td>5</td>
<td>10</td>
<td>151.7</td>
</tr>
<tr>
<td>5</td>
<td>15</td>
<td>208.2</td>
</tr>
<tr>
<td>5</td>
<td>20</td>
<td>207.6</td>
</tr>
<tr>
<td>10</td>
<td>5</td>
<td>106.96</td>
</tr>
<tr>
<td>10</td>
<td>10</td>
<td>80.7</td>
</tr>
<tr>
<td>10</td>
<td>15</td>
<td>158.3</td>
</tr>
<tr>
<td>10</td>
<td>20</td>
<td>182.3</td>
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<td>15</td>
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<td>220.7</td>
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<tr>
<td>20</td>
<td>20</td>
<td>220.7</td>
</tr>
</tbody>
</table>
Figure 4 shows the particle size distributions of SiO₂ aerogel slurries which were added with wetting agent Silok 7117 and dispersant Silok 7195 weighting at 10% of SiO₂ aerogel slurries, and dispersed at different shear rates and lasted for different shear time. From the figure, when the shear rate is 1000r/min, shear time has little effect on the average particle size of slurry, with smaller change in particle size distribution curve, which may be due to insufficient shear force; when the shear rate reaches to 1400r/min, the average particle size decreases with the increase of shear time. But when the shear time exceeds 3h, the average particle size of slurry keeps almost unchanged; the average particle size is the smallest when shear rate reaches 1800r/min and the shear time lasts for 3h, and only one peak shows, with narrowly distributed curve and uniform distribution of particle size. When the shear rate rises to 2200r/min, the average particle size of slurry doesn’t decrease, indicating shear rate has little effect on the dispersion of SiO₂ aerogel slurry when shear force reaches to certain level.

3.3. Change in the Characteristics of SiO₂ Aerogel Powder

Figure 5 shows FT-IR spectra of SiO₂ before and after modification. The FT-IR spectra of modified SiO₂ aerogel has large changes, showing secondary amide-NH stretching vibration peak and C=O stretching vibration peak at 3300 and 1636cm⁻¹, respectively, which indicates that alkylamine salt dispersant is attached to the surface of SiO₂ aerogel. In addition, vibration absorption peaks are enhanced and widened at 1095 and 847 cm⁻¹, which may be due to organic silicon wetting agent showing 2 strong absorption peaks at the two wavelengths.

Figure 4. Dispersion characteristics of different time when shearing rates at (a) 1000r/min, (b) 1400r/min, (c) 1800r/min, (d) 2200r/min

Figure 5. FT-IR spectra of SiO₂ aerogel before and after modification
Figure 6 shows SEM images of SiO$_2$ aerogel before and after modification. It can be found from Figure 6a that unmodified SiO$_2$ aerogel powder aggregates together. This is because SiO$_2$ aerogel powder has small particle size, large specific surface area and high surface energy, so particles can adhere to each other and thereby leading to aggregation (Xu, Ding and Chang 2007). As shown in Figure 6b, SiO$_2$ aerogel powder doesn’t agglomerate and disperse evenly, as wetting agent reduces surface energy of SiO$_2$ aerogel powder, and dispersant effectively prevents the agglomeration of particles through steric hindrance effect (Nel, 2009).

![Figure 6. SEM images of (a) unmodified and (b) modified SiO$_2$ aerogel](image)

XRD was adopted to analyze the phase of SiO$_2$ powder before and after modification, and the results are as shown in Figure 7. It can be learnt from the figure that no obvious crystal characteristic peak shows before and after modification of SiO$_2$ aerogel, but wide diffraction peaks show both before and after modification. It can be seen from the spectrum that a wide amorphous diffraction dispersion peak shows in the vicinity of 20=23$^\circ$, which is also an amorphous characteristic peak of Si-O-Si bond. It can be concluded through comparison that characteristic peak widens after modification, as modified SiO$_2$ aerogel has elevated dispersion, which reduces agglomeration and particle size, so as to widen characteristic peak (Ishikawa, Kazutoshi and Nagaya, 1988). But in general, the position of characteristic diffraction peak of SiO$_2$ does not change after the modification, suggesting its nature doesn’t change after modification.

![Figure 7. XRD spectra of unmodified and modified SiO$_2$ aerogel](image)
Figure 8 shows contact angles of SiO₂ aerogel before and after modification. As shown in the figure, unmodified SiO₂ has high hydrophobic property, and its contact angle is about 145°. After surface modification, the surface of SiO₂ aerogel is wetted by water, and the surface hydrophobic SiO₂ aerogel absorbs amine and other hydrophilic groups, so the contact angle reduces to 45°, which improves its compatibility with water significantly, thus further improving the dispersion of SiO₂ aerogel powder in aqueous medium.

![Image of contact angles](image.png)

**Figure 8.** The contact angle of (a) unmodified and (b) modified SiO₂ aerogel

### 3.4. Thermal Insulation Performance of SiO₂ Aerogel Coating

SiO₂ aerogel slurry with high dispersion was added into coatings, and IR lamp was adopted to simulate sunlight to test the thermal insulation performances of coatings added with different dosages of SiO₂ aerogel. Figure 9 shows temperature change curve of the bottom surfaces of different coatings, it can be seen from the figure that the equilibrium temperature of the bottom surface which is not coated with SiO₂ aerogel thermal insulation coating is 59.2 °C, while the equilibrium temperature of the bottom surface coated with SiO₂ aerogel thermal insulation coating is much lower. With the increase of the dosage of SiO₂ aerogels, the equilibrium temperature of the bottom surface of decreases at first and then increases. When the dosage of SiO₂ aerogel is 5%, the coating has the best thermal insulation performance, and the equilibrium temperature at the bottom is 47.2 °C. Figure 10 shows the curves of thermal conductivity change of different coatings, it can be seen from the figure that if the dosage of SiO₂ aerogel is less than 5%, the thermal conductivity of coatings decreases with the increase of SiO₂ aerogel. When the dosage of SiO₂ aerogel is 5%, the thermal conductivity of coating is the smallest -0.08W·m⁻¹k⁻¹. When the dosage of SiO₂ aerogel is more than 5%, the thermal conductivity of coating increases due to the decrease of dispersion. It is found that when the dosage of SiO₂ aerogel exceeds 8%, the surface of coating will crack, which is probably because SiO₂ aerogel has low density and large specific surface area, the volume and concentration of SiO₂ aerogel added in coating are greater than limits. SiO₂ aerogel coating has good thermal insulation effect, so adding appropriate SiO₂ aerogel can greatly reduce the thermal conductivity of coating, and improve thermal insulation effect.

![Image of temperature change curve](image.png)

**Figure 9.** The change curve of temperature under the model in different mass fraction of SiO₂ aerogel coatings
4. CONCLUSIONS

In order to improve the dispersion of hydrophobic in aqueous medium, and prepare SiO₂ aerogel slurry with high dispersion stability, this paper elaborated the modification principle of SiO₂ aerogel based on dispersion mechanism, and investigated the effects of types and dosages of dispersant and different as well as dispersing processes on the dispersion property of SiO₂, and put forward the optimum dispersion conditions. Finally, this paper introduced the thermal insulation properties of SiO₂ aerogel thermal insulation coating.

(1) SiO₂ aerogel was modified by adding wetting agents at first and then dispersants, to improve its dispersion stability by steric hindrance effect.

(2) The best dispersion condition of SiO₂ aerogel in aqueous medium: added with wetting agent Silok 7117 and dispersant Silok 7195 weighting at 10% of SiO₂ aerogel at the shear rate of 1800r/min, and dispersed for 3h. The modified slurry had stable dispersion, with transmittance rate of 26% at 450nm and average particle size of 80nm.

(3) The nature of SiO₂ aerogel did not change after modification. As the surface was attached with dispersant, the agglomeration of powder reduced significantly, which greatly improved surface hydrophilic properties.

(4) SiO₂ aerogels can significantly reduce the thermal conductivity of coating, and thereby greatly improving its heat insulation performance. SiO₂ aerogel coating could lower the temperature of bottom surface as high as 12°C, so it has good thermal insulation effect in insulation engineering.

5. ACKNOWLEDGEMENTS

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