Effect of Slurry Dispersion on Properties of Silica Fiber Based Thermal Insulation Materials

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Abstract. Short silica fibers are prone to conglomerate in aqueous medium, which means that dispersity and suspension stability show key effect on the uniformity of ceramics with silica fibers as raw materials. In this work, various parameters including pH values, dispersants and dispersing approaches, were systematically studied to investigate the effect of slurry dispersion on properties of thermal insulation materials with silica fibers as matrix. Moreover, the novel composite dispersants were creatively introduced in this work and the corresponding synergetic dispersion mechanism was investigated as well. Fiber slurries with optimized dispersing and suspension properties was obtained when pH values were set as 4, the content of R type dispersant was adjusted to 0.12% and the addition of PEO was fixed at 0.04%. After settling for 360s, no obvious agglomeration could be found. Rigid thermal insulation materials with homogeneous structure and high performance were obtained with conditions mentioned above, which can be expected to further expand the application of short silica fibers.

1. Introduction

Rigid thermal insulating materials with short silica fibers as matrix, sintered at high temperature after wet molding by suction filtration, have been widely used in thermal-protective fields[1]. As is well known, short silica fibers are prone to conglomerate in aqueous medium[2], which means that dispersity and suspension stability show key effect on the uniformity of ceramics with silica fiber as raw materials. Dispersants or other dispersing approaches are usually introduced to address this issue[3]. According to the studies of Orlando J, factors such as fiber length, structure and contents of dispersants, pH value and Zeta potential of dispersing medium were found to obviously affect the dispersity of fiber slurries[4].

In this work, short silica fibers in diameters from 1μm to 5μm, lengths from 0.2mm to 0.7mm with SiO2 more than 99.90 w% are selected as main raw materials, to prepare silica slurry. Hydrochloric acid was added to adjust pH values of the medium, and polyethylene oxide (PEO) and R-type dispersants developed in our lab were creatively introduced as novel composite dispersants to improve its dispersity and the corresponding dispersion mechanism was investigated as well. Finally, rigid thermal insulation materials with homogeneous structure and high performance were obtained with conditions mentioned above. Various parameters including pH values, dispersants and dispersing approaches were systematically studied to investigate the effect of slurry dispersion on properties of silica fiber based thermal insulation materials.
2. Materials and Methods

To explore the affection of acidity on slurry dispersity, short silica fibers were added to deionized water in a weight ratio of 1:80 while ultrasonic stirring for 10min, with hydrochloric acid used to adjust the pH values from 2.0 to 7.0. When pH value was set as 4.0, PEO in a weight ratio of 0.004%–0.032% to deionized water was introduced to investigate the effect of PEO content on slurry dispersity. Then, various amount of PEO and R-type dispersants were mixed as composite dispersants to further study the effect of their ratio on the dispersity and suspension properties of silica fibers.

Finally, rigid thermal insulation samples were prepared using the optimized silica slurry with pH value at 4.0, content of PEO and R-type dispersant as 0.04% and 0.12% respectively. Scanning electron microscope (SEM) was employed to characterize the microstructure of the as-prepared materials. Reference samples were prepared as well using silica slurry without dispersants, to investigate the influence of slurry dispersity on the structural materials.

Table 1. Dispersing performance of silica fibers at different pH values.

| pH values | 2.0 | 3.0 | 4.0 | 5.0 | 6.0 | 7.0 |
|-----------|-----|-----|-----|-----|-----|-----|
| Dispersity (Degree of agglomeration) | Slight | No | No | Slight | Slight | Slight |

Figure 1. Sedimentation photos of silica slurry at different pH values with increasing time: (a) 0s, (b) 60s, (c) 120s, (d) 180s, (e) 240s, (f) 300s and (g) 360s.
3. Results and Discussions

3.1. Effect of pH values on slurry dispersity and suspension

The dispersing performance of silica fibers in water medium at different pH values is summarized in Table 1. As pH value increases from 2.0 to 7.0, the dispersity shows a tendency to get better before getting worse and the best dispersity is obtained with pH value at 3.0 and 4.0, without obvious agglomeration in silica slurry.

Figure 1 shows the sedimentation photos of silica slurry with increasing time at different pH values. After settling for about 60s, interface between fibers and water could be clearly observed. The suspension property of silica slurry is influenced largely by pH values, which is the worst at pH value of 3.0.

The sediment volume of silica slurries with increasing time at different pH values is shown in Figure 2. As can be clearly observed, the sediment volume decreases first and then increases with the increasing pH values at fixed settling time. The best and worst suspension property is presented at pH values of 7.0 and 3.0 respectively. Silica fibers subside quickly within settling time from 60s~150s, while the sedimentation velocity becomes slower and slower when the settling time lasts for 150s~360s. Overall considering the dispersing and suspension performance of the silica slurries, pH value is optimized at 4.0 for the following experiments in this work.

As reported in Ref 5, SiO on the surface of silica fibers absorb the H+ in H2O molecule to induce polarization, and the negative ends of H2O toward the exterior of fibers make them intertwine with each other, which is summarized to be static absorption[5]. The H+ introduced by the addition of hydrochloric acid, is supposed to disrupt the static absorption of the system, which accounts for the improvement of silica slurries in this work.

The microstructure of silica fibers is characterized with SEM and shown in Figure 3. The rough surface with many scaly bulges maybe increase the surface friction between fibers to induce agglomeration, which is considered to be negative for the dispersity of silica slurries.

3.2. Effect of PEO on slurry dispersity and suspension

To further improve the dispersing performance of silica fibers in water medium, different amount of PEO (0.004%, 0.008%, 0.016%, 0.024% and 0.032%) is added to the system, and the sediment volume of obtained silica slurries after standing for 120s and 360s is shown in Figure 4. The increasing sediment volume of the fibers with increasing PEO content demonstrates that PEO is beneficial to the suspension property of silica slurries.
According to the dispersion stabilization mechanism of nonionic dispersants reported in Ref 6, PEO macromolecules absorb on the surface of silica fibers to form a layer of hydration film, which effectively prevents the direct contact and inter-attraction of adjacent fibers [6]. That means that the steric hindrance of PEO largely protect the silica slurries from flocculation and agglomeration. Moreover, the increasing viscosity of the water medium with increasing amount of PEO makes silica fibers subside more and more slowly. The two points mentioned above account for the improvement of dispersing and suspension property of silica slurries with PEO in this work.

3.3. Effect of composite dispersants on slurry dispersity and suspension

According to the theory on electrical double layer of electrostatic stability mechanism for water-based dispersants[7], the stability of fibers in water is determined by the balance of electrostatic repulsion potential energy and van der Waals attraction potential energy, which varies with the fiber spacing. Based on this, there are two ways to improve the dispersing stability of fibers, increasing the electrostatic repulsion of the fiber surface or reducing the van der Waals attraction between adjacent fibers. To investigate the synergistic stabilizing effect, R-type dispersants developed in our lab were creatively introduced to traditional PEO as novel composite dispersants to improve its dispersity and the corresponding dispersion mechanism was investigated as well. The dispersing performance of silica fibers in water medium with composite dispersants in different ratio (w %) is summarized in Table 2.
Table 2. Dispersing performance of silica fibers with composite dispersants in different ratio (w %).

| PEO R-type | 0.004% | 0.008% | 0.016% | 0.024% | 0.040% |
|------------|--------|--------|--------|--------|--------|
| 0.05%      | I\textsuperscript{a} | I\textsuperscript{a} | I\textsuperscript{a} | I\textsuperscript{a} | I\textsuperscript{a} |
| 0.12%      | II\textsuperscript{b} | I\textsuperscript{a} | I\textsuperscript{a} | I\textsuperscript{a} | I\textsuperscript{a} |
| 0.25%      | III\textsuperscript{d} | III\textsuperscript{-e} | II\textsuperscript{b} | II\textsuperscript{-c} | I\textsuperscript{a} |
| 0.50%      | IV\textsuperscript{f} | IV\textsuperscript{-g} | III\textsuperscript{d} | III\textsuperscript{-c} | II\textsuperscript{b} |

\textsuperscript{a} No flocculation
\textsuperscript{b} Slight flocculation
\textsuperscript{c} Better than slight flocculation
\textsuperscript{d} Moderate flocculation
\textsuperscript{e} Better than moderate flocculation
\textsuperscript{f} Severe flocculation
\textsuperscript{g} Better than severe flocculation

The sediment volume of silica slurries settling for 360s with composite dispersants in different ratio (w %) is shown in Figure 5. As can be seen from the figure, the sediment volume of the fibers increases first and then decreases with the increase of R-type dispersant, while continues to increase with the increasing PEO. When the content of R-type dispersant and PEO is adjusted to 0.12% and 0.04% respectively, silica slurries with optimized suspension property can be obtained.

![Figure 5. Sediment volume of silica slurries with composite dispersants in different ratio (w %).](image)

Comprehensive considering the stability of silica slurries as shown in Table 2 and Figure 5, the synergetic dispersion mechanism of the composite dispersants is illustrated as follows. With the increase of R-type dispersant adsorbed on the fiber surface, the electrostatic repulsion between fibers increases, and the suspension property correspondingly increases to give an increasing settlement volume in Figure 5. When the amount of R-type dispersant increased to 0.12%, the adsorption of R-type dispersant on the fiber surface was basically saturated and the sediment volume was optimized to the largest. If more R-type dispersants was added, the increasing free R-type dispersants in the solution resulted in bridging effect, which could lead to fiber flocculation and poor dispersion stability of the silica slurries. At this time, the thickening protective isolation layers formed by increasing PEO addition were supposed to gradually weaken the bridging effect mentioned above and reduce the fiber
flocculation finally. Therefore, composite dispersants developed in this work, can effectively improve the dispersing and suspension property of silica slurries.

In conclusion, silica slurries without obvious agglomeration of fibers were obtained with pH value of 4, R-type dispersant content of 0.12% and PEO content of 0.04%. The sediment volume was 350mL after settling for 360s, and settlement volume rate was about 87%.

3.4. Effect of dispersants on microstructure of the rigid thermal insulation matrix

Microstructure of rigid thermal insulation matrix obtained from silica slurries with and without dispersants is given in Figure 6. It can be seen that introduce of dispersants has a great influence on the uniformity of the matrix. Figure 7a showed that the green body fibers prepared without dispersants were not evenly distributed with many loose and agglomerated defects. By contrast, the green body fibers with dispersants shows a good three-dimensional network framework microstructure, which is uniformly distributed in all directions without obvious defects as shown in Figure 7b. Therefore, it can be convincingly concluded that the uniformity in microstructure of the rigid thermal insulation matrix can be significantly improved by the rational use of dispersants.

![Figure 6](image_url)

**Figure 6.** Microstructure of matrix obtained from silica slurries without (a) and with (b) dispersants.

4. Conclusions

The effect of acidity and dispersants on silica slurry dispersion is systematically studied in this work. Specifically, the sediment volume decreases first and then increases with the increasing pH values at fixed settling time. The dispersity and suspension properties of silica fibers are effectively improved with the addition of traditional PEO, while the novel composite dispersants composed of PEO and R-type dispersants in rational ratio are found to significantly improve the stability of silica slurries. Silica slurries without obvious agglomeration of fibers were obtained with pH value of 4, R-type dispersant content of 0.12% and PEO content of 0.04%. The sediment volume was 350mL after settling for 360s, and settlement volume rate was about 87%. Rigid thermal insulation samples prepared using the optimized silica slurry further confirms the improvement of uniformity in microstructure of the silica fiber based matrix, by the introduction of composite dispersants, which can be expected to further expand the application of short silica fibers.

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