



Development of adiabatic foam using sodium silicate modified by boric acid



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ABSTRACT

Adiabatic foam has been successfully prepared through sintering sodium silicate which is modified by boric acid. The effect of the sintering temperature, boric acid content and modulus of sodium silicate on the thermal conductivity, density, compression strength and microstructure of the adiabatic foam was studied in detail. In the sintering process, B^{3+} ion can take the place of the Si^{4+} ion by incorporating into the Si–O–Si structure via Si–O–B bridges, increasing the chemical stability of the adiabatic foam. High temperature, low boric acid content and low modulus of sodium silicate can reduce the thermal conductivity, density and compression strength. In order to meet the application of exterior wall thermal insulation, 450–500 °C of the sintering temperature, 18–24 g boric acid per 600 g sodium silicate solution, and 2.5–2.8 modulus of sodium silicate are chosen. The samples with thermal conductivity less than 0.044 W/m·K, density less than 100 kg/m³ and compression strength more than 0.7 MPa are obtained.

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1. Introduction

Since there were several serious fire accidents aroused by the organic thermal insulation materials in China, the inorganic thermal insulation materials received widespread attention. For the moment, rock wool [1], thermal insulation mortar [2], expanded perlite board [3] and glass foams [4] are the main inorganic thermal insulation products in the building market. However, some disadvantages limit their application, such as complex process, high sintering temperature and low strength. Therefore, we made an attempt to use sodium silicate to fabricate an inorganic foam material with excellent performance, simple preparation technology and wide application.

In previous limited number of investigations, Kluth et al. [5] fabricated water glass foam based on alkali metal silicates. Though the samples have an outstanding thermal insulation performance, they are not suitable for the heat insulation application due to the low compression strength. Shi et al. [6] improved the strength of this material by using the foam slurry from the reaction of sodium silicate and azodicarbonamide. But the thermal

conductivity and density are higher than those of the products in the market. Therefore, it is necessary to find an additive to modify sodium silicate and then improve the performance of the adiabatic foam material. In fact, sodium silicate solution has many diversified industrial application [7–10], and in order to improve the performance of products, some additives are added into the sodium silicate solution, including mineral acid/salt (hydrochloric acid [11,12], boric acid [13], and sodium phosphate [14,15]), mineral product/industrial wastes (fly ash, blast furnace slag, and metakaolin) [16], and organic matter (urea [17] and ester [18]). In this paper, we chose boric acid as the additive to enhance the strength of the adiabatic foam. As for boric acid, there is non-bridging oxygen in a sodium silicate structure composed of SiO₂ and Na₂O, where BO₄ tetrahedra can be generated in a sodium silicate structure by adding boric acid (H₃BO₃) and adjacent Na⁺ ions make compensation for valence electrons [17]. As a result, by replacing SiO₂ with H₃BO₃, the oxygen is to be bridged again, enabling the structure to be more stable [17].

In this work, we characterized the thermal conductivity, density, compression strength and microstructure of the products fabricated by sodium silicate with boric acid. And the effect of the sintering temperature, boric acid content and modulus of sodium silicate on the above performances was studied in detail.

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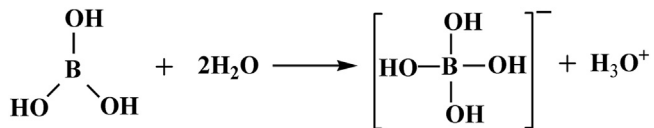
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2. Experiment

2.1. Theory

Sodium silicate solution is a kind of colloid, and thousands of SiO_2 molecular aggregate to form the colloidal nucleus, which is composed of three-dimensional network structure coupled by Si–O–Si bridges [19]. The Si–OH groups form on the interface between the colloidal nucleus and water, which are polar molecules, resulting in the surface of the colloidal nucleus presenting a negative thermodynamic potential [20]. Therefore, hydronium ion, H_3O^+ , and positive ion, Na^+ , are adsorbed on the surface to form the adsorption layer. Moreover, the diffusion layer also forms by the spread of the hydronium ion and positive ion due to the zeta potential of the colloidal particle and thermal motion. All of these result in an electric neutrality of the whole colloidal micelle. The structure of the sodium silicate colloidal micelle is shown in Fig. 1.

When boric acid is added into the sodium silicate solution, hydronium ion, H_3O^+ , and Boron element are introduced.



The hydronium ion, H_3O^+ , can greatly increase the viscosity of the sodium silicate solution because of the micelle cohesion without gel. Moreover, the B^{3+} ion can take the place of the Si^{4+} ion in the SiO_4 tetrahedral network structure, resulting in the structure with a negative charge. Therefore, hydronium ion, H_3O^+ , and positive ion, Na^+ , are tightly adsorbed on the adsorption layer of the colloidal particle, increasing the chemical stability of the colloidal solution [17]. Fig. 2 shows the structure of the sodium silicate sol modified by boric acid.

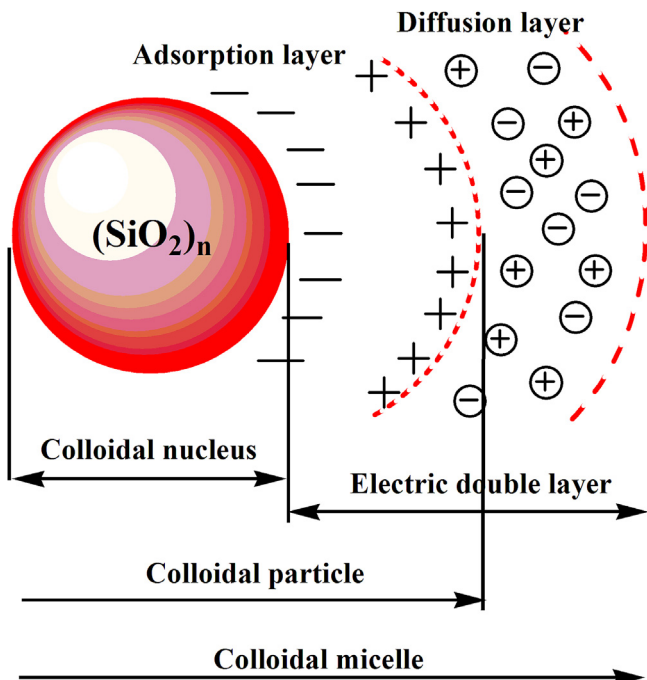


Fig. 1. The structure of the sodium silicate colloidal micelle.

2.2. Experiment process

Sodium silicate (industrial-grade, Shanghai, China) was used as the raw material to fabricate the adiabatic foam. The main parameters are listed in Table 1 and the modulus in the table indicates the molar ratio of SiO_2 and Na_2O in sodium silicate. Sodium hydroxide (Cp, Shanghai, China) was added to adjust the modulus of sodium silicate from 3.0 to 2.0. 6, 12, 18 and 24 g of boric acid were added into the sodium silicate solution (600 g) in order to study the effect of boric acid on the performance of the sintered samples. The aqueous mixture was stirred using a direct driven motor at a speed of 500 rpm for 3 h to produce stable solution. Subsequently, the solution was poured into a mould and sintered in the muffle furnace at 300–500 °C for 30 min with 5 °C/min of heating rate.

2.3. Characterization

The information about various chemical groups was studied using Fourier Transform Infrared Spectroscopy (FT-IR) (Nicolet 8700, Thermo Fisher Scientific, USA). For this purpose, the samples were ground into refined powders, mixed with KBr and pressed to form a sample pellet for the FTIR measurements. The density was determined simply by quality divided volume ($\rho = m/v$). The sample was cut into the regular cuboid and then measure its length, width and height by vernier caliper to calculate its volume. The thermal conductivity of the samples was measured by DRE-2C thermal conductivity tester (China). The microstructure of the sintered samples was observed on fresh fracture surfaces by SEM (JSM-6490LV, Japan). The pore size distribution was obtained by analyzing SEM micrographs using an image analyzer (Nano measurer, China) with a total of at least 150 pores being counted on each image. Compressive strength was measured using a universal testing machine (E3000K8953, Instron Co., USA) with a crosshead speed of 0.05 mm/min. The dimension of the test piece was 20 × 20 × 20 mm. The cross-sectional area of the sample and the maximum failure load were used to calculate the fracture stress.

3. Results and discussion

3.1. FTIR analysis

The IR spectra of the two samples sintered at 450 °C is shown in Fig. 3, and the one is fabricated by sodium silicate without boric acid while the other one is sintered by sodium silicate with 12 g boric acid. It is obvious that the major groups in the IR spectra are the ones associated with the stretching vibration and bending vibration of the Si–O groups [21,22], which correspond to the absorption peaks at 447 cm^{-1} , 771 cm^{-1} and 1017 cm^{-1} . The significant absorption peaks corresponding to the stretching vibration and bending vibration of the –OH groups [23] can be found at around 3419 cm^{-1} and 1653 cm^{-1} , respectively. The two peaks may be attributed to the free Si–OH groups or the constitution water left after the sintering process. The peak at 1459 cm^{-1} exists in the sintered samples without boric acid, which is attributed to CO_3^{2-} . This is considered that CO_2 in air enters into the sodium silicate solution when sintering [24]. Besides, there are slight peak intensities at 2850 cm^{-1} and 2926 cm^{-1} , but it cannot be recognized what groups they represent and thus a further research is needed. However, as the boric acid is added into the sodium silicate solution, some subtle changes of the IR spectra can still be found. First, Fig. 3 ① and ② show the variation of the IR spectra from 1350 cm^{-1} –1450 cm^{-1} for the two samples with and without boric acid. It is observed that there is a sharp absorption peak at 1384 cm^{-1} , which is attributed to the B–O groups [25,26], for the sample with 12 g boric acid though the peak is un conspicuous

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