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Study on the Method of Preparing Zirconia-Silica Composite Aerogels Based on Zirconium Sol

Di Zhao, Pengyu Zhang, Qian Sun, Yibing Guo, Xiaofeng Shi, Ye Dong, Yifeng Han, Hui Li

Tianjin Institute of Building Materials Science Co., Ltd., Tianjin, China

Abstract: *Zirconia-silica composite aerogels are porous gel materials that possess excellent thermal insulation and flame retardancy, and have been widely applied in the field of chemical production. However, during the preparation process of zirconia-silica composite aerogels, raw materials dominated by zirconia sol may undergo violent temperature-varying reactions, which are likely to adversely affect the structural stability of zirconia-silica composite aerogels under high-temperature conditions. Therefore, how to use zirconia sol materials to prepare qualified industrial raw materials of zirconia-silica composite aerogels should be regarded as the key to the innovation of chemical material technology.*

Keywords: *Zirconia Sol; Zirconia-Silica Composite Aerogel; Preparation Method; Influencing Factors*

1. Introduction

At present, with the accelerated development of the chemical industry, the advantages of zirconia-silica composite aerogels as new chemical materials have been very prominent. The main preparation materials of zirconia-silica composite aerogels include zirconia sol, zirconium dioxide, silicon dioxide, etc. Technicians need to reasonably control the reaction temperature and reaction time to maximize the use value of composite aerogels. Scientific experiments and research have shown that zirconia-silica composite aerogels prepared based on zirconia sol can maintain structural stability in extreme temperature and humidity environments, thus verifying the feasibility of the preparation scheme of zirconia-silica composite aerogels.

2. Properties and Applications of Zirconia-Silica Composite Aerogels

2.1 Material Properties

Zirconia-silica composite aerogels mainly use zirconia sol as the preparation material and belong to one of the new porous nanomaterials. Composite aerogels containing two core components, zirconia and silica, generally have the significant characteristic of high porosity. The maximum porosity of such composite aerogels has exceeded 99.5%, and the specific surface area of the material can reach 1000 ㎡/g or more. During the preparation of composite aerogels, the liquid components in the original chemical materials will be completely converted into gaseous mixtures, which determines that the reaction products of aerogels can generally be controlled within a relatively low material density range. Researchers at home and abroad generally indicate that zirconia-silica composite aerogels should be "the least dense solid composites known so far." Compared with other forms of nanocomposite materials, zirconia-silica composite aerogels exhibit special properties in thermal, optical, mechanical, electrical, acoustic and other aspects, which determines that zirconia-silica composite aerogels are suitable for application in cutting-edge industrial fields such as energy storage and chemical wastewater treatment. Since the end of the last century, zirconia-silica composite aerogels have gradually attracted great attention from researchers, transforming the original nanoporous materials into a new type of "super thermal insulation chemical material[1]."

Specifically, the special properties of zirconia-silica composite aerogels are concentrated in physical properties, chemical properties, structural properties and other aspects. Zirconia-silica composite aerogels have a very special microstructure, which determines that the preparation process requirements of such aerogels are relatively high. In fact, each type of composite aerogel has a special microporous structure, and technicians can analyze its structural characteristics by measuring the nanoscale of such aerogels. At the same time, zirconia-silica composite aerogels themselves exhibit transparent or translucent optical properties, resulting in the structural units of such substances being smaller than the wavelength of most visible waves, and causing the aerogels to show a pale blue or dark blue apparent color under high-intensity light irradiation. Analyzing from the mechanical properties of zirconia-silica composite aerogels, due to their special brittleness and hardness characteristics, the structural strength of the aerogels themselves is relatively low. Most of the currently known zirconia-silica composite aerogels are stacked by sheet-like structural materials, and the overall structural strength of the aerogel composites often depends on the "penetration degree" of the material matrix. Therefore, researchers need to further explore innovative schemes in the preparation process of zirconia-silica composite aerogels[2].

2.2 Application Occasions

Due to the above characteristic advantages, zirconia-silica composite aerogels have shown good technical application prospects in many industrial fields. Because of the relatively high porosity and specific surface area of aerogel composites, such chemical raw materials have been widely used in fields such as adsorbing harmful and toxic chemical wastes, storing hydrogen fuel, and preparing filter membranes. At present, zirconia-silica composite aerogels can also be used to make catalysts and can absorb part of the chemical reaction media, so they are generally regarded by technicians as "excellent catalysts or catalytic carrier materials."

In recent years, zirconia-silica composite aerogels have achieved good technical promotion results in the optical field and the outer space field. For example, a technical experimental team from Europe is currently testing the function of composite aerogels in capturing tiny dust in the universe through satellites or space shuttles. Researchers in the experimental team once used composite aerogels composed of zirconia and silica to capture small dust particles from comets during an experiment. Some researchers in the experimental team also proposed that silica aerogel composites with small self-weight can also capture small-volume dust or other particles in the universe. This indicates that aerogel composites may be used to purify the cosmic space environment in the future, and such substances show good technical promotion value in collecting and capturing space pollutants[3].

3. Experimental Design for Preparing Zirconia-Silica Composite Aerogels Based on Zirconia Sol

The main experimental raw materials include silica sol, basic zirconium carbonate, nitric acid, propylene oxide, and ethanol. The specific experimental process is designed as follows: first, take basic zirconium carbonate and dissolve it in water, and place it in a four-necked flask; then add a certain amount of nitric acid for dissolution, and after mixed heating, reflux condensation and stirring, obtain zirconia sol through aging reaction. In the above steps, the concentration of basic zirconium carbonate is about 5%, the concentration of nitric acid is controlled at 6% and 10%, the sol reaction temperature is set at 75℃, and the reaction time is 6h and 12h.

As shown in Table 1 below, it is the ratio design of the zirconium sol preparation experiment:

Table 1. Preparation Ratio Design of Zirconia Sol

Serial Number	Initial Solid Content	w% (H^+)	Reaction Time	Ceramic Content
1	5%	6	12h	3.5%
2	5%	10	12h	4.2%
3	5%	6	6h	3.5%
4	5%	10	6h	3.6%

After the above experimental operations, technicians take zirconia sol and silica sol for mixing, where the molar ratio of zirconium to silicon is (0.25:1), (0.5:1), (0.75:1), and (1:1). On this basis, they are mixed as 1#, 2#, 3#, and 4# respectively, and the normal-temperature stirring time of the mixed sol reaches 1h. Then, the mixture is added dropwise to the propylene oxide mixed sol system until the pH value of the mixture reaches between 6.5 and 6.8. After the dropwise addition is completed, continue to stir the mixture for 1-2h to ensure that the mixed sol and propylene oxide are uniformly mixed and the reaction is sufficient. After standing the mixed sol for 1h-2h, place the wet gel in a water bath at 50℃ for aging; on the basis of aging for 24h, add ethanol for replacement (the number of replacements is three, with an interval of 12h each time). Finally, the wet gel is dried by ethanol supercritical drying

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process to obtain zirconia-silica composite aerogels.

As shown in Figure 1 below is the schematic diagram of the Tyndall effect of zirconia sol:

Figure 1. Schematic Diagram of the Tyndall Effect of Zirconia Sol

4. Analysis of Influencing Factors for Preparing Zirconia-Silica Composite Aerogels Based on Zirconia Sol

4.1 Initial Solid Content and Reaction Time

Initial solid content and reaction time are both key factors affecting the performance of zirconia-silica composite aerogels. Only when technicians strictly control the above two indicators during the experiment can they further improve the chemical material performance of composite aerogels. Through the above experimental operations, technicians concluded that under the condition of initial solid content of 5%, the increase of H^+ concentration will promote the increase of ceramic content. And the longer the reaction time, the more significant the promoting effect of H^+. Based on the ceramic yield data of zirconia sol in Table 1, serial number 2# was finally selected as the zirconium source for this experiment. It was verified that the ceramic content of serial number 2# is basically consistent with that in silica sol, so it is easier to stabilize the gel system of the mixed sol. The following table details the test results of bulk density, thermal conductivity, and specific surface area of zirconia-silica composite aerogels. Technicians can analyze the result data obtained from this experiment and analyze the potential influencing factors of the composite aerogel products during the entire preparation stage through the surface phenomena of the experiment[4].

As shown in Table 2 below are the test results of zirconium-silicon aerogel:

Table 2. Test Results of Thermal Conductivity and Bulk Density of Zirconium-Silicon Aerogel under Normal Temperature Conditions

Serial Number	Bulk Density (kg/m^3)	Thermal Conductivity W/(m·K)	Specific Surface Area (m^2/g)
1	35.4	0.017	501.89
2	38.3	0.018	500.86
3	40.0	0.017	475.24
4	35.6	0.016	475.59

4.2 Raw Material Composition

The composition of reaction raw materials during the preparation of zirconia-silica composite aerogels is directly related to the structural stability of the final product. In this experimental study, technicians summarized based on the test results of XRF and EDS that the finished zirconium-silicon composite aerogels prepared by compounding zirconia sol and silica sol have a more stable elemental structure. The elemental content in the finished product is basically consistent with the feeding amount before and after, thereby effectively avoiding the massive loss of zirconium ions, and the zirconium and silicon elements also form a more stable structure. It can be seen that reasonably controlling the raw material composition in the chemical reaction helps zirconia-silica composite aerogels achieve optimal performance. During the entire chemical reaction of the experiment, technicians should also accurately record the results of various experimental data, and then fully input the data indicators obtained from this experiment into the computer database to provide reliable decision-making guarantee for improving the performance of composite aerogels.

As shown in Tables 3 and 4 below are the test results of XRF and SEM/EDS:

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Table 3. Test Results of Zirconium-Silicon Aerogel by XRF

Serial Number	ZrO_2/conc. (%)	SiO_2/conc. (%)
1	2.7	94.6
2	2.8	96.2
3	2.5	95.4
4	2.4	95.2

Table 4. Test Results of Zirconium-Silicon Aerogel by XRF

Serial Number	Zr/Weight%	Si/Weight%
1	6.99	93.01
2	13.02	86.98
3	16.03	83.97
4	12.93	87.70

5. Process Optimization for Preparing Zirconia-Silica Composite Aerogels Based on Zirconia Sol

In recent years, domestic researchers have focused on innovating the preparation scheme of zirconia-silica composite aerogels, aiming to simplify the aerogel preparation process, optimize resource allocation, and promote chemical enterprises to achieve the optimal economic benefit goal. However, in the actual reaction process, the operation process of preparing zirconia-silica composite aerogels is usually affected by factors such as manual operation, external environment, and raw material quality, which determines the complexity of the entire reaction process. Based on the above considerations, technicians in chemical enterprises at present need to improve the traditional preparation process of gel composites in combination with actual conditions, save valuable resource costs for enterprises, and promote the transformation of the domestic chemical industry towards refinement. Specifically, the process optimization for preparing zirconia-silica composite aerogels based on zirconia sol should be reflected in the following measures:

5.1 Strictly Control Reactant Concentration, Temperature and Time Conditions

In most cases, factors such as reactant concentration, solution temperature, and reaction time during the chemical reaction for preparing composite aerogels will directly affect the entire reaction process. Therefore, technicians should strengthen the monitoring and control of indicators such as reactant concentration, reaction temperature, and time length. The optimal experimental conditions for preparing zirconia-silica composite aerogels using zirconia sol as raw material are: basic zirconium carbonate concentration 5%, nitric acid concentration 10%, reaction temperature 75°C, reaction time 12h (corresponding to 2# experimental parameters). The core basis for drawing the above conclusion is that the zirconia sol prepared under this condition has a ceramic content of 4.2%, which is basically consistent with that of silica sol, and can ensure the stability of the gel system of the mixed sol to the greatest extent, thus laying a good foundation for the subsequent composite reaction. At present, technicians can also make full use of Internet of Things tools to realize real-time monitoring and feedback of abnormal conditions during the entire experiment, thereby providing strong guarantee for the optimization of the preparation process of composite aerogels[5].

5.2 Real-Time Determination of Material Specific Surface Area and Thermal Conductivity

Both the specific surface area and thermal conductivity of zirconia-silica composite aerogels are key technical indicators, so technicians need to strengthen the monitoring of the above two indicators during the entire reaction process. The core performance indicators of zirconia-silica composite aerogels prepared based on zirconia sol are: bulk density 35.4-40.0kg/m³, material thermal conductivity as low as 0.016-0.018W/(m·K), and material specific surface area reaching 475.24-501.89m²/g. It can be seen that the important advantages of adopting the above preparation process lie in the extremely low thermal conductivity (far superior to traditional thermal insulation materials), large specific surface area (reflecting the advantages of porous structure), and light weight (small bulk density) of composite aerogels. The zirconium-silicon composite structure has strong stability, and no element loss phenomenon was found. The zirconia-silica composite aerogels prepared by the improved zirconia sol have a longer service life and are suitable for scenarios requiring high thermal insulation performance and stability of composite materials[6].

5.3 Innovate Composite Performance Testing Methods

Through three types of tests: real-time inductively coupled plasma spectroscopy test, XRF test, and SEM/EDS test, technicians summarized the following test results: the inductively coupled plasma spectroscopy test results showed that the content of zirconium element in the waste liquid after secondary replacement was 0, and only a small amount remained after primary replacement (≤0.002%); XRF and SEM/EDS test results showed that the content of zirconium and silicon elements in the prepared composite finished products was basically consistent with the initial feeding amount, with no obvious deviation. This indicates that zirconium and silicon elements form a stable composite structure, which effectively improves the loss phenomenon of zirconium ions. At present, technicians need to further innovate the scheme design of inductively coupled plasma spectroscopy testing to save material costs on the basis of ensuring the accuracy of test results. In addition, it is necessary to reasonably control the time length and temperature and humidity of the entire reaction to ensure the orderly progress of the reaction process and avoid affecting the quality of aerogel reaction products due to external factors.

6. Conclusion

In summary, using basic zirconium carbonate as the zirconium source, and adding nitric acid under heating and stirring conditions to prepare zirconia sol; then compounding the prepared product with silica sol can finally prepare zirconium-silicon composite aerogels. Current scientific experimental data show that the quality of zirconium-silicon aerogels is the best when the zirconium concentration is 5%, the nitric acid concentration is 10%, and the heating and stirring time reaches 12h. The thermal conductivity of zirconium-silicon aerogels meeting the above conditions is 0.018W/(m·K), and the specific surface area is 500.86m²/g. In addition, no zirconium element was found in the waste liquid after primary sol replacement, which indicates that the composite degree between various elements tends to be stable.

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