

Syntesis of silica aerogel by surface modification

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Syntesis of silica xerogel by surface modification: *Using tetraethoxysilane (TEOS) and trimethylchlorosilane (TMCS) as co-precursors, we explore the synthesis of hydrophobic silica aerogel by in situ surface modification at ambient pressure. Compared with hydrophilic feature of the products prepared by TEOS single precursor (without TMCS surface modification procedure), hydrophobic behavior of the aerogel obtained from TEOS and TMCS co-precursors was observed. Fourier Transform Infrared spectra (FTIR) and Thermogravimetric analysis (TGA) were conducted to confirm the successful introduction of the hydrophobic group on the silica surface.*

Key words: *aerogels, xerogels, surface modification*

INTRODUCTION

Aerogels were first prepared by Kistler in 1931 [1] and they are highly porous materials with a high specific surface area, alows density, dielectric constant and thermal conductivity. Silica aerogel is the most common type of aerogel and the interconnected nano particle building blocks for manopen and highly porous three-dimensional network. They have shown promising application sinthermal insulation, acoustic insulation, catalysis, etc. [2–5]. In the production of silica aerogel, the most important step is to removet hesol vent from the wet gel and maintain the continuous solid network of the same size and shape. If the wet gel is simply dried on its own, it normally shrinks. Such shrinkage is often accompanied by severe cracking of the gel. Supercritical fluid drying has been performed to produce high-quality silica aerogel by venting the ethanol above its critical point or by prior solvent exchange with CO₂ followed by supercritical venting [6]. In order to get commercialize lowercost aerogel, Brinker and his colleagues successfully replaced the supercritical drying step by chemically modifying the surface of the gel. The hydrophobic functional groups were introduced and attached to the wet gel backbone by reacting with the hydroxyl groups, which avoids the shrinkage during the subsequent drying process at ambient pressure [7]. Multiple chemical modification is an effective approach to prepare the silica aerogel at ambient pressure and trimethylchlorosilane (TMCS) is the most used surface modification reagent [8–10]. Compared with the traditional supercritical drying process, such multiple treatments were found to need long time and several washing and solvent exchange steps to get the hydrophobic silica aerogel in terms of low cost and safety. In this report, we explore the one-step synthesis of hydrophobic silica aerogel by in situ surface modification with using tetraethoxysilane (TEOS) and trimethylchlorosilane (TMCS) as co-precursors to reduce the processing time of multiple chemical modification approach.

EXPERIMENTAL

In order to determine the difference between the products produced from single precursor and co-precursors, we prepared the silica aerogel based on the multiple chemical modification approach and the detailed experimental information can be found in the reference [9]. The experimental procedure is much simplified for the preparation of hydrophobic silica aerogel by the co-precursor method. It means that the formation of silica wet gel and surface modification occurs at that moment. TEOS and TMCS were used as co-precursors and mixed with ethanol, water and HCl solution (Molar ratio in the solution ;TEOS: TMCS: Ethanol: H₂O: HCl = 1: 0.4:3:1.4:7x10⁻⁴). Then the solution was refluxed for 1h. The distilled solution was redissolved in ethanol and reacted with additional water/ ethanol solution under NH₄OH catalyzed conditions (Ethanol : H₂O: NH₄OH = 1: 7: 5x10⁻⁴). Finally the sol was poured into petri dish to gel and age at room temperature for 24h. Both the wet gels produced from single precursor and co-precursors were washed by n-hexane and left to dry at 50°C for 4h. Fourier transform infrared spectroscopy (FTIR) was

employed to investigate the chemical bonding state of surface modifying agent with aerogels. The thermal behaviour of the dried gel was examined using DSC/TG analysis.

RESULTS AND DISCUSSION

After washing and drying at ambient pressure, the samples produced from the single precursor and co-precursors were measured to determine the wettability. Fig. 1(a) shows the FTIR spectra of the as-synthesized products by co-precursor and single precursor methods. It is seen that the bands at 1080 and 450 cm^{-1} for Si–O–Si bonds are present in the spectra of both products. The spectra of single-precursor samples display abroad band located at 3450 cm^{-1} attributable to O–H stretching vibrations of Si–OH and H–OH bonding. A weaker O–H bending vibration band is observed at 1600 cm^{-1} . The bands located at 2980, 1255, 1400 and 850 cm^{-1} are attributed to the $-\text{CH}_3$ of the grafting of $-\text{Si}-(\text{CH}_3)_3$ groups attaching on the silica backbone, which indicates the successful introduction of the hydrophobic group on the silica backbone. Through eliminating the attractive forces between water and the silica surface, the hydrophobic behavior protects the aerogel from damage by liquid water. The FTIR spectra of the silica aerogel prepared from TEOS single precursor with a single TMCS surface modification the hydrophobic group on the silica backbone [9].

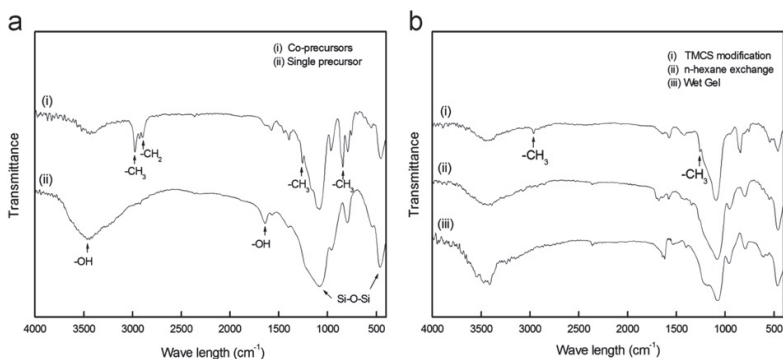


Fig. 1. FTIR spectra of the synthesized products

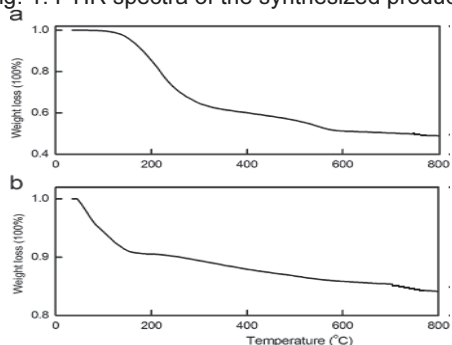


Fig. 2. TGA curves of the products prepared by co-precursor (a) and single precursor (b) methods

Thermo-gravimetric analysis (TGA) was carried out from 30 to 800°C for the products prepared by co-precursor and single precursor methods. The results are shown in Fig. 2. For the co-precursor aerogel, no weight loss is observed up to 140°C.

It reveals that water or ethanol does not exist in the aerogel for its hydrophobic feature. Then about 50 % dramatic weight loss is followed, which corresponds to the decomposition of chlorine and the oxidation of the $-CH_3$ groups attaching on the silica backbone. The TGA curve shows a 10% weight loss ending up at $160^{\circ}C$ for the single-precursor aerogel, which is assigned to the adsorbed solvents. And the 5% weight loss from 250 to $800^{\circ}C$ is attributed to the pyrolysis of residual organics.

CONCLUSIONS

One-step synthesis of hydrophobic silica aerogel by in situ surface modification at ambient pressure was explored with tetraethoxysilane (TEOS) and trimethylchlorosilane (TMCS) co-precursors method. It was found that the procedure for synthesis was simplified and long processing time was avoided in comparison with the multiple chemical modification approach.

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За контакти:

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This paper has been reviewed