The highest surface area derived via ambient pressure drying - synthesis and characterization of DMF-modified silica aerogel for thermal insulation

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Abstract

Silica aerogels are a unique class of highly porous materials (80 - 99.8%) with large specific surface area $(500 - 1200 \text{ m}^2/\text{g})$. Due to their properties, silica aerogels are considered as objects of widespread interest through last years, e.g. as excellent insulating materials [1,2]. This super-insulating property is due to the air entrapped inside the pores within silica backbone.

In this work, the hydrophobic silica aerogels were prepared via ambient pressure drying method (APD) by a surface silylation using TMCS/n-hexane mixture [3,4]. The structural and physical properties of synthesized DMF-modified and unmodified aerogels were characterized using BET, TG, SEM, FT-IR and Raman techniques.

The BET specific surface area for pure silica sample (unmodified with DMF) is quite large (estimated at ~828 m²/g). Particularly, the BET surface area of DMF-modified silica sample is much more higher and reaches up ~1231 m²/g. Such high surface area is comparable with the values obtained by other researchers for silica aerogels dried in supercritical conditions and significantly higher from the surface areas obtained via APD [1,3]. As far as we know, such high BET surface area for APD aerogels was reported for the first time.

On the basis of the obtained results have been presented the differences in structure between samples before and after a surface silvlation, which was not previously documented. The structural measurements confirmed the efficient silvlation process (TMCS/n-hexane), as well as the presence of DMF residues of hydrogen-bonded with unreacted Si-OH groups within silica backbone after surface modification. Based on TG analysis, it has been found that DMF addition improves thermal resistance (up to ~320°C) and hydrophobic character (up to ~270°C) due to the presence of Si-(CH₃)₃ modified groups of prepared aerogel. The chemical aspects of modification, influence on the structure and physical behavior have been described in details and compared with unmodified aerogel.

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