

Cost effective synthesis of mechanically strong silica aerogels *via* ambient pressure drying

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Abstract

The unique physical properties of silica aerogels make them attractive for use in several applications. However, silica aerogels have found severe restrictions to be used in several specialized environments, such as in Cerenkov radiation detector in certain nuclear reactors, collector of hypervelocity particles in space, and thermal insulators in space vehicles [1]. The expanded industrial and commercial use of silica aerogels has been difficult to implement because of their poor mechanical properties and fragility. Moreover, the need for drying wet gels with supercritical fluids during manufacturing poses issues of safety and cost of the preparation process. The ambient pressure process is safer and less expensive than supercritical drying process, and has been more actively investigated in recent years. However, it has the drawback of modifying the inner structure of the gel due to the evaporation of the liquid inside the pores that causes serious shrinkage and cracking due to the high capillary pressure at the menisci of the solid-liquid-vapor interfaces inside the drying gel structure.

Several worth mentioning processes avoid this phenomenon caused by the capillary tension inside the gel and crack free ambient pressure dried aerogel monoliths can be obtained [2]. One of these approaches proceeds via the surface modification of the silica to produce a hydrophobic surface. This is done by reacting the silica surface hydroxyl groups with hydrophobic reagents, such as $[(\text{CH}_3)_3\text{Si-OR}]$, or hexamethyldisilazane. Alternatively, the gel inner structure can be strengthened to stand the capillary stresses. This can be achieved by replacing some of the siloxane (Si-O-Si) bonds with flexible and non-hydrolysable organic bonds (Si-R), through the use of organosilanes as co-precursors to produce the aerogel network. Another approach is the crosslinking of silica aerogels with an appropriate polymer to prepare organic-inorganic hybrid material [3, 4]. Here in, the organic group allows the aerogel to spring back to its original wet gel size avoiding in this way, cracks within the gel. Finally, another method to overcome the induced capillary pressures involve the use of low surface tension hydrocarbon solvents [3, 4]. Evaporation of a low surface tension solvent from the silica wet gel network reduces the capillary pressure when compared to the evaporation of an alcohol, since there is a direct relation between the surface tension and capillary pressure [5].

Recently, we have studied the effect of different underlying silica on the physicochemical properties of tri-methacrylate cross-linked silica aerogels [6]. In this context, we have studied the effect of the incorporation of two bis-silane types silica co-precursors of alkyl-linked 1,6-bis(trimethoxysilyl)hexane (BTMSH) and aryl-linked 1,4-bis(triethoxysilyl)-benzene (BTESB) as well as effect of different crucial factors on the final aerogels' properties. We were able to show that thermal conductivity depends on the silica nanostructure namely the mesoporosity that can be controlled, to some extent, by proper design of the synthesis route of the resulting aerogels.

Regardless of the type of underlying silica, we prepared reinforced silica aerogels with density ranging from 0.22 to 0.39 g/cm³, compression strength from 10 to 400 kPa and thermal conductivity from 0.039 to 0.093 W/mK. The basic idea of this study was to produce aerogels made by bis-silane having a rigid aryl spacer. In this way, less structural collapsing upon drying occurs and therefore, a drastic improvement in terms of porosity and thermal conductivity was achieved.

In this context, we have explored the preparation of ambient dried reinforced silica aerogel with optimized properties by submerging the wet cross linked gel inside a low surface tension solvent in our case, hexane. Subsequently the samples were oven dried at ambient pressure. In this way we prepared aerogel-like material with comparable physical and mechanical properties with their aerogel counterparts (Figure 1). In this methodology we obtained aerogel-like material with approximately more than 3 order of magnitude improvement in compression strength over their non-reinforced counterparts with densities ranges between 0.27 and 0.55 g/cm³ and thermal conductivity ranges of 0.06- 0.09 W/mK. By avoiding supercritical drying an important move was made for large scale and cost effective production of aerogel- like materials.

References

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Figures

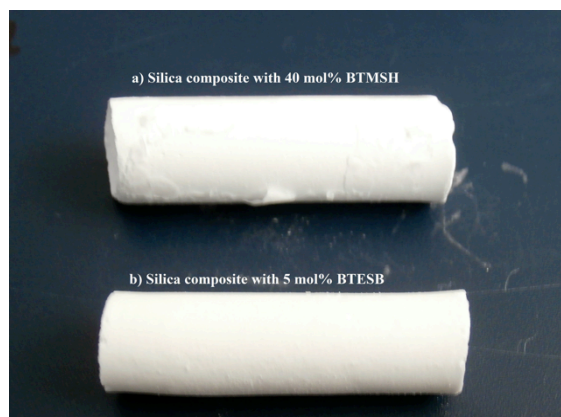


Figure 1. Two examples of prepared polymer-silica aerogel composite a) by replacing 40 mol% of total silicon by BTMSH and b) 5 mol% of total silicon by BTESB.