

Carbon Aerogels by Supercritical Carbon Dioxide Drying

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Carbon aerogels are mesoporous materials with unique physical properties that enable them to serve as catalyst supports, electrodes for supercapacitors and rechargeable batteries, thermal insulators, adsorbents and chromatographic packing. Carbon aerogels (CAs) can be produced either as monoliths, powders, thin films or microspheres.

In this work, CAs were synthesized by the pyrolysis (carbonization) of resorcinol-formaldehyde aerogels (RFA) in a quartz process tube at 1000°C under flowing N₂ (with a flow rate of 100 cm³/min). The RFA were obtained via a sol-gel route and supercritical carbon dioxide (scCO₂) drying. The so-called sol-gel route involves the polymerization (gelation) of resorcinol (R) and formaldehyde (F) with a basic catalyst (C) environment with water (W) in room temperature for one day followed by a curing sequence (one day at 50°C and three days 90°C) which provides a cross-linked structure within the polymer to obtain an aquagel. Afterwards, the gel was exposed to solvent-exchange (acetone ↔ water) for at least twelve hours and finally dried using scCO₂ in order to remove the solvent (acetone) and replace the pores with air to produce an aerogel. Then, the gels were dried using scCO₂ at 13.8 MPa and 50°C for two days. The purpose of using scCO₂ arises through its unique properties; carbon dioxide has relatively reachable critical pressure (7.38 MPa) and temperature (31.1°C), and is relatively inert, non-combustible, and environmentally benign. Furthermore, the liquid-vapor boundary vanishes above the critical point which aids the interfacial forces to disappear whose presence may otherwise cause the shrinking of the gel.

The synthesis method used can result in CAs with reproducible physical properties such as high surface area (400 – 2000 m²/g), high porosity, controllable pore size in mesoporous range (2-50 nm), very sharp pore size distribution, low mass density and high electrical conductivity (1-15 S/cm). In the framework of this study, BET analysis confirmed the high surface area and the nano-range of the pore sizes of the synthesized CAs. The possibility of controlling the pore size of the gels by varying R/C, R/F and R/W molar ratios has also been noted. The various compositional ratios used in this study resulted in aerogels with a pore size range of 4 to 22 nm and with surface areas through 600 to 800 m²/g. We have observed that at constant R/W and R/F ratio (0.08 and 0.5, respectively), an increase of R/C ratio from 50 to 100 results in an increase of the pore sizes of CAs from 3.2 to 4.3 nm. On the other hand, at constant R/C and R/F ratio (100 and 0.5 respectively), an increase of R/W ratio from 0.04 to 0.08 results in a decrease of pore sizes of CAs from 7.9 to 4.3 nm. This behavior can be explained primarily by less cross-linking in the gel after the curing process when either R/W mole ratio is decreased or R/C mole ratio is increased causing the formation of larger pores. Furthermore, with lower R/W, more water occupies the interstices of the gels, resulting in a higher pore diameter and volume. It was also observed that after the pyrolysis process, the pore sizes of the gels decreased consistently.

The CAs can further be used in numerous applications. Efforts are under way in our laboratory to incorporate metallic nanoparticles on CAs to use them as catalysts in promising applications such as fuel cells.