

# Carbon Aerogel Beads: Preparation, Characterization and Upscaling

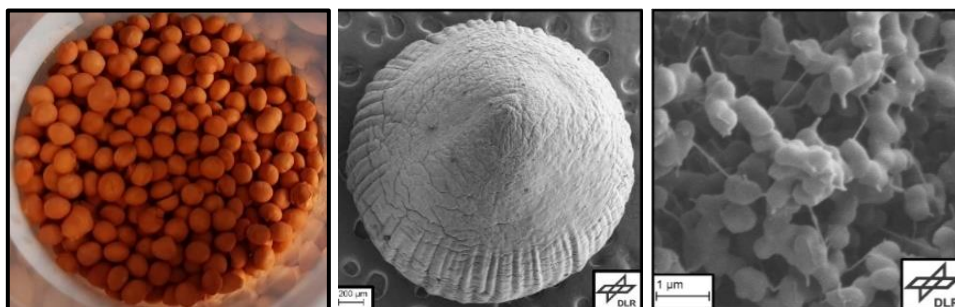
Seeni Meera Kamal Mohamed

Department of Aerogels and Aerogel Composites,  
Institute of Materials Research,  
German Aerospace Center (DLR), Cologne, Germany.  
E-mail: [seenimeera.kamalmohamed@dlr.de](mailto:seenimeera.kamalmohamed@dlr.de)

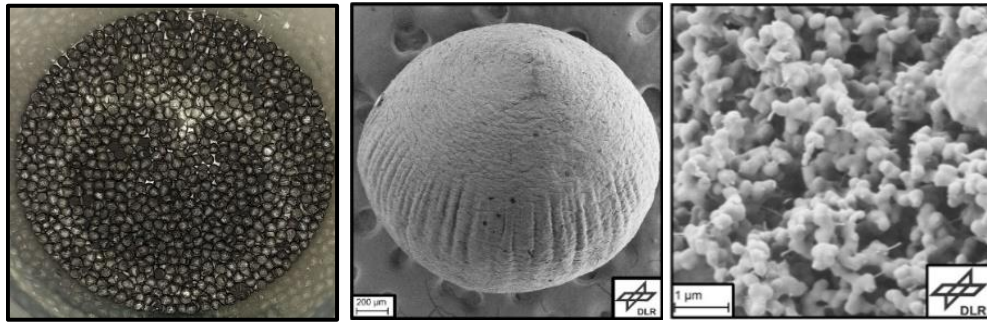
In the last several years, there has been an increased interest among researchers in the development of carbon based materials such as activated carbon, carbon beads (micro- and milli-meter sized) and microspheres, carbon nanotubes, graphene, fullerene, etc. Carbon based materials exhibit unique structures, morphologies, properties and so on. Among these materials, carbon aerogels (CAs) are extraordinary materials with high surface area, high porosity with unique micro-structures consisting of micro-pores, etc. CAs find applications in catalysis, chromatography, energy, adsorbents, desalination, etc. Currently, researchers focused on the development of CAs in the form of spheres and beads instead of monoliths due to the reduction in processing time and equipment costs.

Carbon beads (CBs) are produced conventionally from coal or petroleum pitch by carbonizing them at 350-500 °C to achieve mesophase spheres in pitch. The carbonization process is quenched before an extensive growth and aggregation of the spheres and separated as solids from soluble pitch by extraction with a solvent. Carbon microspheres of 1 μm to 3 mm are also fabricated by using an inverse-emulsion gelation of resorcinol-formaldehyde (RF) solution at high temperatures followed by super-critical drying and subsequent carbonization. The removal of oil and subsequent drying by super-critical drying consume large amount of solvents, energy and time. Henceforth, there is a huge increasing demand for an easy process to form CBs.

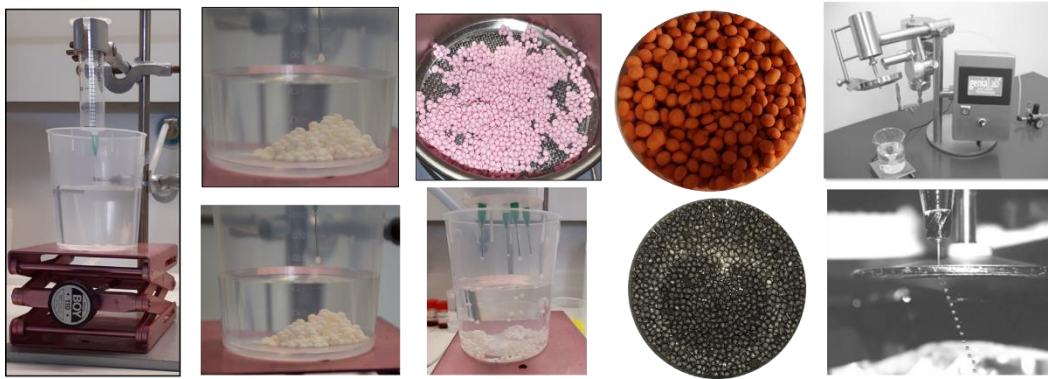
In the current work, we utilized sol-gel chemistry to form RF aerogel beads (Fig. 1) after ambient pressure drying and converted them using a thermal treatment process into carbon beads. The existing challenge is the shaping of RF sol into bead form by dropping method which has never been reported in the literature because of the low sol viscosity (~ 0.5 Pa.s). Hence, we adjusted the viscosity of the RF sol by adding a polysaccharide-based thickener to achieve RF bead formation (1-10 Pa.s), followed by sub-critical drying and carbonization. The average sizes of the carbon beads (Fig. 2) are in the range of 1.75-2.02 mm with skeletal densities of 2.0-2.4 g/cm<sup>3</sup>. The micropore surface area and pore volume are ranging from 735-901 m<sup>2</sup>/g and 0.28-0.35 cm<sup>3</sup>/g, respectively. SEM images reveal that the beads are spherical and its inner-morphology composed of inter-connected particles grown on the top of the thickening agents' fibers.



**Fig. 1:** Photographic and SEM images of RF beads.



**Fig. 2:** Photographic and SEM images of carbon beads.



**Fig. 3:** Overview of the RF and Carbon aerogel beads production.

The production of RF and Carbon aerogel beads at the laboratory and large scale is presented in Fig. 3. The large scale production of RF aerogel beads by Jet-Cutting method has several challenges *viz.*, handling of enormous amounts of RF sol and acid bath, process parameter optimization, waste disposal, etc. Hence, we adopted low to high viscous alginate solution (0.5 - 3wt.%) with a viscosity ranging from 0.08 to 10 Pa.s, as a model system to optimize the Jet-Cutting parameters. The diameter of the beads ( $d_{bead}$ ) can be described in eq. 1.

$$d_{bead} = \sqrt[3]{\frac{3}{2} D^2 \left( \frac{u_{fluid}}{n \cdot Z} - d_{wire} \right)} \quad \text{--- (1)}$$

Where,  $D$  - diameter of the nozzle,  $u_{fluid}$  - fluid velocity,  $n$  - number of cutting wires,  $Z$  - number of cutting tool rotation,  $d_{wire}$  - diameter of the wire.

The operation of Jet-Cutter® for the production of beads with low-viscous fluid is no means of complicated. Hence, the fluid velocity, diameter of the nozzle and number of cutting tool rotation were understood. The large scale production of the RF beads using Jet-Cutting method was achieved. The produced RF beads were pyrolyzed and characterized.

## References

- [1] N. Liu, S. Zhang, R. Fu, M. S. Dresselhaus, G. Dresselhaus, *Carbon*, **2006**, 44, 2430-2436.
- [2] S. Chandrasekaran, P. G. Campbell, T. F. Baumann, M. A. Worsley, *J. Mater. Res.*, **2017**, 32(22), 4166-4185.
- [3] B. Milow, S.M. Kamal Mohamed, German Patent Application, **2021**, No.: 10 2021 100 898.0.
- [4] T. Anklam, Synthesis and Characterization of Resorcinol-Formaldehyde (RF) and Carbon Aerogel Microbeads by Dropping Method, Master's Thesis, 2021, University of Cologne, Cologne.
- [5] M. Chen, M. Pan, Y. Chong, J. Wang, D. Long, *Carbon*, **2018**, 130, 782-791.
- [6] M. Kamal Mohamed, C. Heinrich, B. Milow, *Polymers*, **2013**, 13, 2409.
- [7] S. Mulik, C. Sotiriou-Leventis, N. Leventis, *Chem. Mater.*, **2007**, 19, 6138-6144.
- [8] U. Prübe, F. Bruske, J. Breford, K-D. Vorlop, *Chem. Eng. Technol.*, **1998**, 21(2), 153-157.
- [9] U. Prübe, B. Fox, M. Kirchhoff, F. Bruske, J. Breford, K-D. Vorlop, *Chem. Eng. Technol.*, **1998**, 21(1), 29-33.