Challenges in the Up-scaling of Resorcinol-Formaldehyde Aerogel Micro-beads Production by Jet-Cutting Method

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Abstract

Carbon aerogels (CAs) are excellent materials with high surface area and unique microstructures consisting nano-particles and nano-pores. CAs find potential applications in catalysis, chromatography, energy, adsorbents, desalination, etc. In recent times, researchers focused on the development of CAs in the form of micro-spheres and/or micro-beads due to the reduction in process time and equipment costs for various applications. Carbon micro-beads are produced from coal or petroleum pitch by carbonizing at 350-500 °C to achieve mesophase spheres in pitch. The carbonization is quenched before an extensive growth and aggregation of the spheres and separated as solids from soluble pitch by extraction with a solvent. Carbon aerogel micro-beads of 1 μm to 3 mm are also fabricated by using an inverse emulsion gelation of resorcinol-formaldehyde (RF) solution at elevated temperatures followed by freeze drying or super-critical drying and subsequent carbonization. The freeze drying or super-critical drying techniques consume large amount of solvents, energy and time for the large scale production.

Fig. 1. Overview of the RF and Carbon Aerogel Micro-beads Production.

Herein, we report the development of RF aerogel micro-beads through dropping method. The shaping of RF sol solution into a micro-beads is quite challenging because of its low apparent viscosity (~ 0.5 Pa.s) and faster sol-gel transition. Hence, we aimed to improve the viscosity of the RF sol solution using a sustainable polysaccharide based thickener to achieve suitable viscosity (1 - 10 Pa.s) in-order to shape them into a micro-beads. The pre-gelled RF sol solution with various concentrations of thickener is dropped into an acid bath to form micro-beads followed by curing, washing and ambient pressure drying and carbonization. The ambient

pressure drying of the micro-beads is one of the greatest advantages of the process in-terms of low-cost. The overview of the RF and Carbon aerogel micro-beads production at the laboratory and large scale is depicted in Fig. 1. However, large scale production of RF aerogel micro-beads by Jet-Cutting method poses 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.

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

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 micro-beads with low-viscous fluid is no means of complicated. However, the fluid velocity, diameter of the nozzle and number of cutting tool rotation need to be understood. In the next step, large scale production of the RF micro-beads using Jet-Cutting method will be optimized and demonstrated.

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