POROSITY AND DENSITY CHARACTERISTICS OF CARBON MOLECULAR SIEVES PREPARED FROM POLYMER PRECURSORS

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Introduction

A series of spherical, carbon molecular sieves particles have been synthesized from polymer precursors. These carbons have been prepared using spherical polymers (both porous and non-porous) and have been pyrolyzed and/or graphitized to produce carbon molecular sieves and graphitized polymer carbons. Syntheses of the polymers included the use of suspension polymerizations, emulsion polymerizations and a modified microemulsion process to produce spherical carbon spheres with a variety of porosities and densities.

A nitrogen porosimeter has been used to study the surface areas, pore size distributions and total pore volumes of the carbons. Carbon pore diameter plots have been generated using DFT software. A helium pycnometer has been used to determine the helium density of the carbons. The variety of carbons prepared allows for adsorption applications in several fields of interest.

Experimental

The experimental approach to preparing the polymer carbons entailed the processing of porous polymers in the 1-10 micron particle size range using a modified suspension polymerization process. The production of pores in the spherical polymer beads entailed varying the phase extender type and concentration, as well as varying the copolymer ratios to achieve the desired polymer pore structure.

The experimental approach to carbonizing the polymer entailed the preparation of an appropriate ion-exchange resin (IER) with subsequent carbonization of the IER.

The experimental approach for the pycnometer analyses entailed an initial thermal/vacuum cleaning of the carbon at 225ºC prior to sample introduction into the pycnometer system. The maximum number of system purges [1] were

incorporated for the samples and 10 analytical runs were performed and averaged to obtain the density values for each sample. Activated coconut charcoal was added as a sample for reference purposes.

The experimental approach for the porosimetry analyses entailed an initial vacuum degas of the samples at 300ºC, with subsequent analyses using the incremental dose method at 4.0 cc/g [1]. The data reported were: BET surface area, total pore volume, t-Plot micropore volume and area, Density Functional Theory (DFT) plots, and Dubinin-Radushkevich plots.

For the pycnometer effort, five probe gases were chosen for the analyses of the three carbons. These five probes were: helium, nitrogen, argon, carbon dioxide and neon.

For the porosimeter effort, four probe gases were chosen for the analyses of three carbons (two synthetic, polymer carbons and activated coconut charcoal). The four probes were: nitrogen, argon, carbon dioxide and neon.

Results and Discussion

The data obtained from the pycnometer effort indicated that only helium and neon provided effective density data at 19.5 psig.

The data obtained from the porosimetry effort indicated that one of the synthetic polymer carbons and the activated coconut charcoal possessed only micropores, and the second synthetic, polymer possessed micropores, mesopores and macropores. The difference between the two polymer carbons is a result of the copolymers chosen as well as the phase extenders used to create the pores during the polymerization process.

Conclusion

Two synthetic, polymer carbons have been synthesized in the 1-10 micron particles size range. These carbons have been analyzed using different probe gases in both a pycnometer and a porosimeter, and the results compared to activated coconut charcoal analyzed using the same analytical parameters. The use of different probe gases, to study the carbon pore regions, provided insight into the interaction of the probe gases and the carbon pores.

References

1. Webb, P.A. and Orr, C., Analytical Methods in Fine Particle Technology. Micromeritics, Norcross, GA, 1997.