Characterization of Spherical, 50 Micron Polymer Carbon Sieves and Graphitized Polymer Carbons for Sample Prep

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Abstract

The sample preparation technique of solid phase extraction (SPE) has been utilized for several decades as a concentrating step for biological, environmental and industrial sample preparation applications. Typically, a packed bed cartridge, containing approximately 0.1 to 1.0 grams of packing, is used to extract the analytes of interest from the liquid sample matrix (e.g., water), then the analytes are removed/extracted from the cartridge using a strong solvent which is miscible with the analytes. The use of carbons for concentrating analytes such as oligosaccharides, small polar molecules, pesticides, herbicides and insecticides has been well known for several decades as well. Improvements in the carbon purities, particle size distribution, pore structure and surface chemistries have led to the ability to analyze trace-levels of the respective analytes. The preparations of a spherical, high purity 50 micron carbon molecular sieve (CMS) and the preparations of a 50 micron spherical, high-purity graphitized polymer carbon (GPC) have led to the development of SPE cartridges and micropipette tips containing these carbons. A nitrogen porosimeter and helium pycnometer have also been used for this study.

Introduction

Two new 50 micron carbons have been prepared for sample preparation applications. Improvements in the carbon purities, particle size distribution, pore structure and surface chemistries have led to the ability to analyze trace-levels of analytes such as mono-, di- and oligosaccharides, small polar molecules, acidic and basic pesticides, herbicides and insecticides. Environmental protocols, and the reduction in volume requirements for biological analyses have necessitated these carbon improvements. The preparations of two new carbons are discussed here.

The preparation of a spherical, high purity 50 micron carbon molecular sieve (CMS) with a large microporous regime has led to the preparation of SPE cartridges for use in environmental applications focused on insecticides, herbicides, pesticides. Biological applications of this same CMS focused on the concentrating and subsequent analyses of monosaccharides and disaccharides.

The preparation of a spherical, high-purity graphitized polymer carbon (GPC) has led to development of SPE cartridges and micropipette tips containing the GPC particles adhered to the inside of the working tip area using a proprietary, patented adhesive. These tips are used for environmental applications and small volume (e.g., 1-10 uL) biological applications [1,2].

Methods

The preparation of the first carbon, the microporous CMS, entailed the preparation of a spherical polymer bead using a suspension polymerization process. This polymer was prepared to possess a microporous and mesoporous regime. Following polymerization, an ion-exchange resin (IER) was prepared by addition reaction of the ion-exchange group to the unsaturated ring structure of the aromatic polymer. The IER was subsequently pyrolyzed to obtain the CMS.

The preparation of the second carbon, the GPC, entailed the preparation of a spherical polymer with large macropores tapered to mesopores in a range of 50 to 500 Å. Following polymerization, an ion-exchange resin was prepared, then pyrolyzed and subsequently graphitized.

A nitrogen porosimeter was used to study the surface areas, pore size distributions and total pore volumes of the carbons [3]. A helium pycnometer was used to determine the helium density of the carbons. X-ray diffraction data were generated for the GPC carbon. Adsorbent capacities and reversible adsorption characteristics have been determined using the respective sample preparation processes.

Results and Discussion

The data obtained for the microporous CMS are presented in Table 1 and Figure 1, below. The nitrogen porosimetry and DFT plot data illustrate the highly microporous (and some small mesopores) regime of this amorphous carbon. The helium pycnometry data provide the density of this carbon. The analytical data (i.e., cartridge capacity and carbon capacities) for several key analytes are presented in Table 2, below. These data indicate the effective performance for this carbon in the requisite application.

Table 1. Porosimetry Data for 50 Micron CMS.

Carbon Description	Absolute Helium Density (g/cc)	Surface Area (m2/g)	Total Pore Volume (cc/g)	Average Pore Diameter (angstroms)
Carboxen-1026	2.2730	1149	0.782	27.2

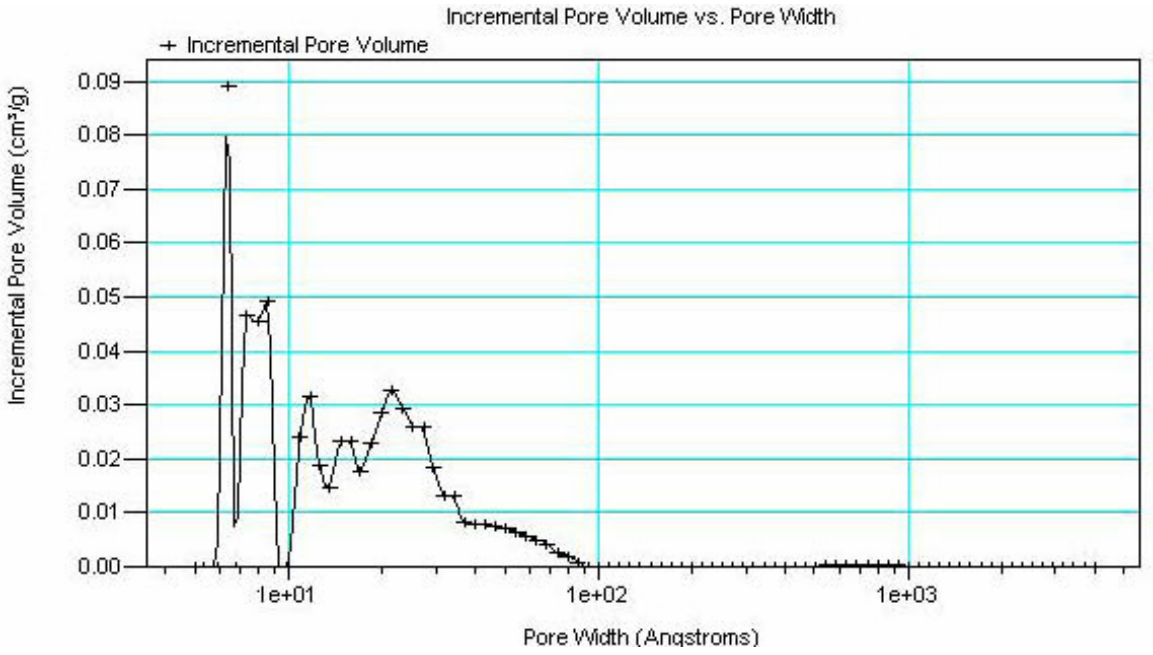

Figure 1. DFT Plot of 20-50 Micron CMS

Table 2. Recovery and Capacity Data for Acephate Using a 0.5 mL SPE Cartridge

Analyte	**Capacity/Breakthrough Volume (liters)**	**Recovery (%)**
Acephate	> 1.0	94.8

The porosimetry and pycnometry data obtained for the GPC carbon are presented in Table 3 and the DFT plot is presented in Figure 2, below. The nitrogen porosimetry and DFT plot illustrate the mesoporous and macroporous regime of this graphitized carbon sample. The helium pycnometry data provide the density of this carbon. The XRD data presented in Table 4 indicate the percentage of graphite in this carbon with 2 distinct phases of carbon (i.e., graphite and amorphous) present in these particles [4].

The analytical data (i.e., tip capacity and carbon capacity) for a key analyte are presented in Table 5, below. These data indicate the effective performance for this carbon in the requisite application. A light microscope photograph of the working end of a micropipette tips is presented in Figure 3.

Table 3. Porosimetry Data for 50 Micron GPC.

Carbon Description	Absolute Helium Density (g/cc)	Surface Area (m2/g)	Total Pore Volume (cc/g)	Average Pore Diameter (angstroms)
Carboxen-1027	1.5032	126	0.542	173

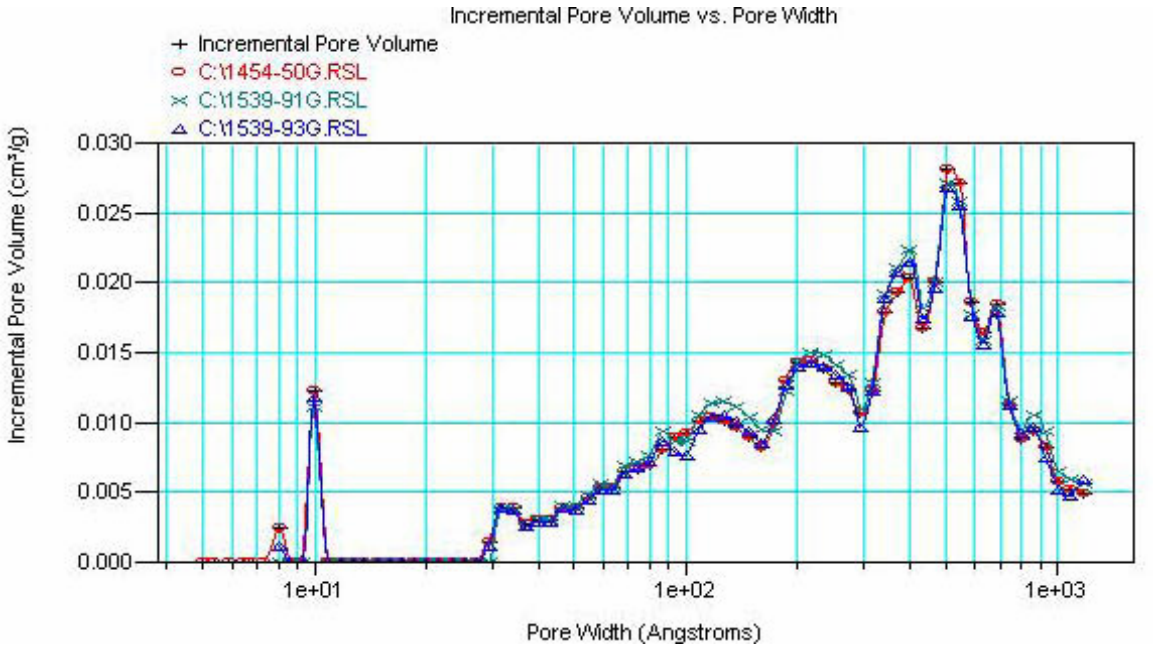

Figure 2. DFT Plot of 50 Micron GPC (3 batches)

Table 4. XRD Data for 50 Micron GPC Carbon

Carbon Description	2θ	d_{0002}(Å)	Lc (Å)	R factor
Carboxen-1027	26.41˚	3.372	30	7.56%

Table 5. Recovery and Capacity Data for Maltohexaose Using a Carbon Pipette Tip

Analyte	**Binding Capacity (µg/tip)**
Maltohexaose	10.2

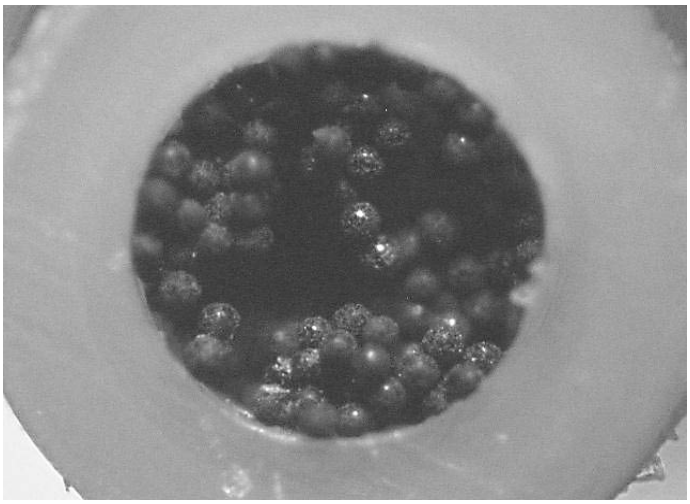

Figure 3. Light Microscope Photograph of Carbon Pipette Tip

Conclusion

The characteristics of two new spherical, 50 micron carbons have been obtained, and tested against the sample preparation performance of these two carbons in the chosen applications. The optimization of the carbons for the specified applications was based on the changes observed with the physical characterization methods employed. The data obtained indicate that the carbons performed effectively for the respective applications.

References

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