CHARACTERIZATION OF POLYMER CARBON SIEVES AND GRAPHITIZED POLYMER CARBONS COATINGS FOR SAMPLE PREPARATION APPLICATIONS

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Abstract

The use of carbons in sample preparation techniques such as solid phase extraction (SPE) and air sampling has been ongoing for several decades. Recent advancements in sample preparation techniques have led to the use of carbons in micro-SPE techniques, solid phase microextraction (SPME) techniques and carbon coatings for both gas and liquid sample preparation techniques. Improvements in 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 spherical, high purity 2 micron carbon molecular sieves (CMS) with a large microporous regime allowed for the preparation of coated surfaces for environmental applications. These applications focused on a range of analytes from light gases to the semi-volatiles. These CMS carbons have been bonded to glass, metal and plastic substrates using patented, proprietary adhesives.

The preparations of spherical, high-purity 2 micron graphitized polymer carbons (GPC) have led to development of coated surfaces for liquid phase applications focused on semi-volatile and nonvolatile compounds. These GPC carbons have been bonded to glass, metal and plastic substrates using patented, proprietary adhesives [1].

The preparation of 175nm graphitized carbon blacks (GCB) has also led to the development of coated substrates for sample preparation of semi-volatile compounds.

Nitrogen porosimetry, helium pycnometry, titration and inverse gas chromatography (IGC) techniques were used to study the carbons. Additional adsorbent capacities and reversible adsorption characteristics have been determined using the respective sample preparation processes.

Introduction

A 2 micron CMS carbon has been prepared for coating surfaces intended for gas-phase sample preparation applications. The analyses of trace levels of environmental, industrial, warfare chemicals and explosives contaminants are now realized due to improvements in the carbon purities, particle size distribution, pore structures and surface chemistries. Preparation of a microporous-only CMS with a surface of approximately 700 m^2/gram [2] has been effective for the concentration and desorption of airborne, volatile compounds such as Methyl Chloride. The presence of 5Å pores augments the condensation of the Methyl Chloride molecules in the deep micropores of the carbon. The purity of the carbons and carbon surfaces also allows for the effective adsorption and subsequent desorption of polar compounds such as Ethyl Acetate, Nitromethane, and Propionaldehyde. The technique of Inverse Gas Chromatography (IGC) was utilized for the initial capacity testing of the carbons [3].

The preparation of a 2 micron spherical, high-purity graphitized polymer carbon (GPC) has led to development of coated substrates for environmental applications (both gas-phase and aqueous samples).

The preparation of a 175nm GCB with microporosity and mesoporosity has led to coatings of substrates with these carbons for the adsorption and subsequent desorption of semi-volatile compounds.

Experimental

The preparation of the microporous CMS entailed the processing of a spherical polymer bead using a suspension polymerization process. This polymer was prepared to possess a microporous-only 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 GPC carbon 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.

The preparation of the 175nm GCB entailed graphitization of a carbon black precursor at >2500°C.

A nitrogen porosimeter was used to study the surface areas, pore size distributions and total pore volumes of the carbons. A helium pycnometer was used to determine the helium density of the carbons. A modified HP-6890 gas chromatograph was used to determine the capacities/breakthrough volumes of specific analytes with the respective carbons. Film thicknesses of the coatings were measured using a light microscope with a graduated ocular.

Results and Discussion

The textural data for 3 of the carbons are presented in Table 1. The DFT plot for 3 CMS carbons are presented in Figure 1. The light microscope photographs of example, coated substrates are presented in Figures 2 and 3.

Table 1. Textural Data for a CMS, GPC and GCB Carbon.

	BET	BET	BET	
	surface area	total pore volume	average pore diameter	particle size
carbon description	(m2/g)	(cc/g)	(angstroms)	(um)
CMS	418	0.221	11	2.0
GPC	126	0.542	173	2.0
GCB	77	0.244	127	0.2

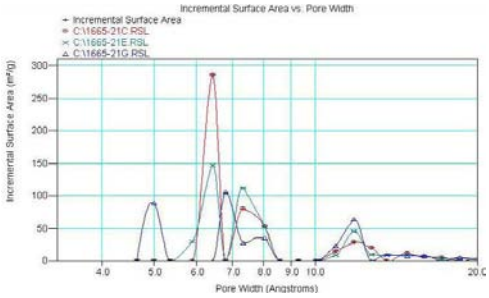

Figure 1. DFT Plot of 2.0 Micron CMS Carbons

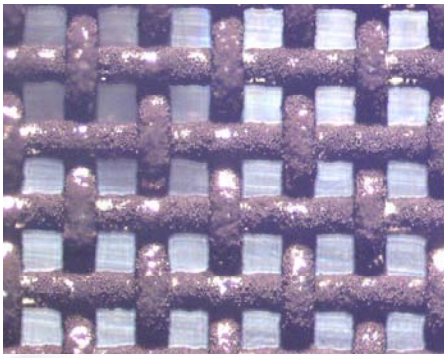

Figure 2. Light Microscope Photograph of CMS bonded to a mesh screen (40X)

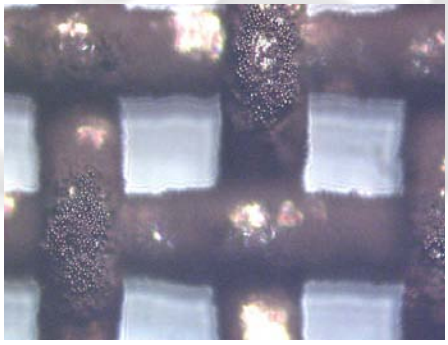

Figure 3. Light Microscope Photograph of CMS bonded to a mesh screen (100X)

Conclusion

The textural and performance characteristics of several new spherical 2 micron carbons and a grapgitized nanocarbon black have been determined. 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

1. Betz, W. and J. Desorcie, Nucleophilic Bodies Bonded to Siloxane and Use Thereof for Separations from Sample Matrices, Japan Patents JP3913760, JP4016051, US Patents US05599445, US05607580, US05609756, US05620603, US05630937.

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

3. Kiselev, A.V. and Yashin, Y.A. (1969), Gas Adsorption Chromatography, Plenum Press, New York.