PREPARATION OF POROUS GRAPHITIZED CARBON BLACKS AND POROUS CARBON MOLECULAR SIEVES FOR AIR MONITORING PROCESSES

W.R. Betz and M.J. Keeler
Supelco, Inc., Supelco Park
Bellefonte, PA 16823 USA

Introduction

A new family of air sampling tubes has been developed which contain several adsorbent beds in one tube. The tubes have been designed to adsorb and subsequently thermally desorb volatile analytes such as methane, acetylene, ethane and light sulfurs as well semi-volatile analytes. The tubes contain one or more graphitized carbon blacks (GCBs) and one or more carbon molecular sieves (CMSs). The GCBs function as the first packed bed(s) to adsorb analytes which are classified as semi-volatile, and the subsequent CMS packed bed(s) are tailored to adsorb the volatile fraction.

The GCBs have been thermally treated at a temperature above 2500ºC, and possess no pores, or mesopores. The porous GCB has a mean pore diameter of 100Å and a surface area of 250 meters^2 per gram. The non-porous GCBs have no porosity, and have surface areas ranging from 5 meters^2 per gram to 100 meters^2 per gram.

The CMSs possess multiporous and monoporous, microporous characteristics, and have been prepared from synthetic, spherical porous polymers, which are then converted to ion-exchange resins, and subsequently pryolyzed to form amorphous carbon sieves.

Experimental

The experimental approach to prepare the GCB carbons involved the choice of starting material and furnace temperature, above 2500ºC, to produce 5 GCBs with surface areas ranging from 5 to 240 meters^2 per gram.

The experimental approach to prepare the multiporous CMSs involved the synthesis of porous polymers possessing the multiporous characteristics, which upon carbonization, yelded a multiporous carbon.

The experimental approach to prepare the monoporous, microporous carbons parallelled the miltiporous CMS approach, with monoporous polymers prepared to produce monoporous, microporous carbons.bon molecular sieve. The porosity characterisitics of the carbon were determined using a Micromeritics ASAP2010 porosimeter[1].The particle size distributions were controlled at the polymer stage.

The second polymer was prepared to possess a monoporous, microporous structure. The particle size distribution range was also optimized in the 2-3 micron particle size range. The polymer was subsequently converted to an ion-exchange resin, and pyrolyzed to produce the carbon molecular sieve. Porosity and size measurements were performed as mentioned above.

Characterization of all the carbons has been accomplished using a Micromeritics ASAP 2010 porosimeter, and sampling characteristics have been determined using inverse gas chromatography (IGC).

Results and Discussion

The data obtained from the porosimetry analyses, for all the carbons prepared, are presented in Table 1. The data obtained from the GCB porosity data indicate that the nonporous carbons possesse Type II isotherms, and the porous GCB (surface area = 240 meters^2 per gram) possessed a Type IV isotherm. The data obtained for the multiporous carbons indicate that these carbons possessed Type IV isotherms, with noticeable to well developed hysteresis loops. The data obtained for the monoporous, microporous carbons indicate that these carbon possess Type I isotherms.

Conclusion

Two families of carbons, graphitized carbon blacks and carbon molecular sieves, have been prepared for use in air monitoring processes. The data generated

for these carbons have been utilized to construct packed tubes for both general and specific applications.

References

1. Webb, P.A. and Orr, C., *Analytical Methods in Fine Particle Technology*. Micromeritics, Norcross, GA, 1997.
2. Betz, W.R. and Lambiase, S.J., *J. Chromatogr.*, 556 (1991)433-440.

Table 1. Porosity data for the graphitized carbon blacks

Carbon description	BET surface area (m2/g)	porosity (cc/g) micro	meso	macro
Carbopack X (GCB)	240	0.00	0.62	0.00
Carbopack B (GCB)	100	0.00	0.00	0.00
Carbopack Y (GCB)	24	0.00	0.00	0.00
Carbopack C (GCB)	10	0.00	0.00	0.00
Carbopack F (GCB)	5	0.00	0.00	0.00
Carboxen-1000 (CMS)	1200	0.44	0.16	0.25
Carboxen-1001 (CMS)	500	0.22	0.13	0.11
Carboxen-1003 (CMS)	1000	0.38	0.26	0.28
Carboxen-563 (CMS)	510	0.24	0.15	0.24
Carboxen-564 (CMS)	400	0.24	0.13	0.14
Carboxen-569 (CMS)	485	0.20	0.14	0.10
Carbosieve S-III (CMS)	820	0.35	0.04	0.00