USE OF GRAPHITIZED CARBON BLACKS FOR THE CHROMATOGRAPHIC SEPARATION OF LIGHT HYDROCARBONS AND OTHER VOLATILE GASES

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Introduction

Graphitized carbon blacks (GCBs) have been prepared for use in gas-solid chromatographic separations of light hydrocarbons and other volatile gases. Preparation of the GCBs involved high temperature furnace processes performed in inert atmospheres to yield a family of carbons. This carbon family possesses surface areas ranging from 5 to 250 meters^2/gram. The GCBs possessing surface areas of 100 meters^2/gram or less have no pores associated with the particles, whereas the GCB possessing a surface area of 250 meters^2/gram possesses mesopores with a mean pore diameter of 100Å. The wide range of physical characteristics allows for tailored chromatographic performance characteristics.

Experimental

The experimental approach consisted of preparing five GCBs with surface areas ranging from 5 to 250 meters^2/gram. The gas chromatographic packed columns were prepared using 2.0 meter x 2.0 mm ID glass columns inserted in a Hewlitt Packard 5890 GC. Starting materials and furnace temperatures were chosen to optimize the surface areas and chromatographic performances of the GCBs. A gas blend of C1 to C6 hydrocarbons was utilized to characterize the GCBs using inverse gas chromatography (IGC). Both isothermal and temperature programmed analyses were used for the IGC characterizations.

The five GCBs were also evaluted for percentage of graphite crystallinity. The carbon samples were prepared for d-spacing measurements by grinding a small amount of sample to less than 200 mesh (i.e., 75 uM), and mixing with a small amount of silicon powder (SRM 640) which is a standard reference material from the former NBS[1]. The samples were subsequently cast on a glass slide for insertion into a Philips Norelco diffractometer.

Results and Discussion

The specific goal of this study was to evaluate the hydrocarbon series with respect to the surface areas of the GCBs. Plots of the hydrocarbon number relative retention time versus carbon surface areas provide insight into the adsorption characteristics of the carbon surfaces. For example, Table 1 lists the absolute retention times for n-butane and the carbons.
Heats of adsorption and spreading pressures can be obtained from these chromatographic retention volumes as well.

Table 1. n-butane retention time data

Carbon	Surface area	n-butane Retention time
Carbopack X	250	15.5
Carbopack B	100	7.8
Carbopack Y	25	4.6
Carbopack C	10	2.4
Carbopack F	5	1.3

The results of the retention studies indicate that a correlation exists between the retention time for the adsorbate molecules and the GCB surface area.

The results of the crystallinity data generation are presented in Table 2.

Table 2. degree of graphitization for the GCBs

Carbon	% graphite crystallinity
Carbopack X	2.0
Carbopack B	20
Carbopack Y	75
Carbopack C	90
Carbopack F	95

Th results of these data indicate that a correlation exists between the percentage of graphite crystallinity and the carbon surface area.

Conclusion

A series of five graphitized carbon blacks has been generated, and chromatographically evaluated using a C1 to C6 hydrocarbons series. Crystallinity data have also been generated. The data indicate that a correlation exist between the carbon surface areas and the chromatographic retention data, and that a correlation exists between the carbon surface areas and the carbon degree of graphitization.

References

1. Aune, F., Brockner, W., and Oye, H.A., *Carbon.* 1992, **30**, 1001.