EFFECTS OF CARBON BLENDING AND GRANULE SIZE LAYERING ON BUTANE BREAKTHROUGH CURVES

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ABSTRACT

Butane breakthroughs of packed granulated carbon beds were studied to provide information for the design of longer lasting gas mask canisters. Breakthrough curve data for 1000 ppm butane were obtained and analyzed for adsorption capacities, rates and asymmetry parameters. Two commercial carbons with granules in the 12-30 mesh size range were used: 1) a chromium-free, impregnated carbon, ASZM-T, and 2) an unimpregnated activated carbon, BPL. These carbons were blended and layered in a 3:1 ratio, respectively, by volume to determined effects on adsorption capacities and rates. Six replicate layered beds (with the BPL layer downstream) and seven replicate blended beds of the carbons showed no statistical differences in rates or capacities. The ASZM-T carbon was separated into fractions in the 12-16, 16-20, and 20-30 mesh size ranges and used to prepare layered beds for testing. Keeping the total bed volume the same, backup layers of the smaller sized carbons increased adsorption rates up to 50%. However, because of the dominance of the capacity in determining breakthrough time, corresponding 1% butane breakthrough times increased only up to 6%.

INTRODUCTION

The chromium-free carbon, ASZM-T, now in production for military filtration systems has resulted in a 25% increase in impregnant loading on the carbon substrate, BPL carbon, compared with ASC carbon. These impregnants significantly decrease both the capacities and the adsorption rates of physically adsorbed vapors and gases, as demonstrated with butane. One possibility for recovering some of the adsorption rate is the reduction of carbon granule sizes; however, this does not improve the capacity and has the cost of increased pressure drop. In this paper we present a study of three other options: 1) layering of ASZM-T and BPL carbons, 2) blending of ASZM-T and BPL carbons, and 3) layering of granule size fractions of ASZM-T carbon.

The loss of packed bed contaminant removal efficiency with time of flow of air is described by a breakthrough curve, i.e., contaminant concentration in effluent air vs. time. A higher adsorption rate, such as may produced by the presence of an unimpregnated carbon or smaller carbon granules, should result in a delayed initial breakthrough and a steeper breakthrough curve. A layered bed in which the back layer is made up of smaller granules than the front may delay the initial breakthrough by "sharpening up" the concentration wavefront passing through the bed.

We have developed an equation to which breakthrough curve data can be fit for extracting capacity and rate parameters.² This equation handles ideal, symmetrical breakthrough curves; but also skewed, unsymmetrical ones produced by heterogeneous adsorption sites and heterogeneous beds. It was used for these analyses of butane breakthrough curves.

EXPERIMENTAL

A mixture of 1000 parts-per-million by volume of butane in dry (13-23 % relative humidity) air was prepared by metering 11.7 mL/min of butane into a flow of 11.7 L/min dry air. This mixture passed through a glass manifold and through a glass cylinder (6.9 cm inner diameter) packed to a depth of 2.0 cm with dry (as received) activated carbon. This flow rate corresponds to a residence time of 0.38 s, a linear velocity of 5.2 cm/s, and a flow of 32 L/min through a C-2 canister.

The air effluent from the center of the packed bed was sampled through a fluorocarbon tube. The butane concentration was measured at one-minute intervals with a photoacoustic infrared spectrometer (Bruel & Kjaer Type 1302). These measurements were made from the time of first introduction of butane to where the butane effluent concentration leveled off, indicating bed saturation and no further adsorption. Raw data was stored on a desktop computer for subsequent analysis.

Beds were packed using a vacuum flow through the sample cylinder and slow, random, dropping of the carbon granules through a 1-m tube (the "snowflake" technique). Beds were packed by weight to 2.0 cm depth using bed depth vs. weight calibrations prepared for each granule size range and carbon type.

Pressure drop across the packed bed was measured several times during each test using a water manometer (Dwyer Model XX). Measurements were averaged.

Original carbon samples were taken from larger batches prepared by Calgon Carbon Corporation. The BPL activated carbon was Lot 1330, nominally 12-30 mesh size range. The ASZM-T carbon, Lot CO1142, also nominally 12-30 mesh, was a BPL carbon impregnated with 2% molybdenum, 5% copper, 5% zinc, 0.05% silver, and 3% triethylenediamine by weight. Granule size fractions were prepared by sieving the original carbons through USA Standard Testing Sieves (W.S. Tyler, Inc.) Numbers 12, 16, 20, 30 and 40. A 12-20 mesh fraction was obtained by recombining the 12-16 and 16-20 mesh granules in the original weight ratio. The carbon fractions were used without further pretreatment. Layers were separated by a thin fluorocarbon screen.

RESULTS

Blended Beds

Table I shows adsorption capacities and rate coefficients obtained from breakthrough curves for the original ASZM-T and ASZM-T/BPL blended carbon beds.

Table I. Comparison of Original, Blended, and Layered Carbons.

Carbon(s)	Carbon Weight	Number of	Capacity (mg/g)		Stoichiometric Rate Coefficient (1/min)		1% Breakthrough Time (min)	
	(g)	Runs	Average	SD**	Average	SD	Average	SD
ASZM-T	44	5	39	2.1	3090	140	53	9.4
3:1 Blended*	41	6	40	1.6	3000	230	48	3.3
3:1 Layered*	41	7	39	2.7	3180	330	50	5.0

^{*} BPL (8g) was the downstream layer or the carbon blended with ASZM-T (33 g).

These results show **no** significant improvement in capacity, stoichiometric (at the breakthrough curve center) rate coefficient, or 1% breakthrough time for the 3:1 ASZM-T:BPL blend over the same volume of ASZM-T carbon.

Layered Beds

One attempt to improve packed bed performance by carbon layering involved layering of the two kinds of carbons, ASZM-T and BPL, in a volumetric ratio of 3:1. Seven replicate runs were made for good statistical comparisons with beds prepared with blends of the same amounts and types of carbons. Table I shows that the layering produced **no** significant differences in capacity, stoichiometric rate coefficient, or 1% breakthrough time.

We also explored the effects of back layers (11 g) of different carbon granule sizes with a front layer (33 g) of the same original 12-30 mesh ASZM-T carbon. Figure 1 shows higher adsorption rate coefficients calculated at the breakthrough curve centers (stoichiometric times) and at 1% breakthrough times increased when the back quarter of a 12-30 mesh carbon bed was replaced with smaller carbon granules. (Unfortunately, there was only enough 30-40 mesh carbon for one experiment.) The differences between adsorption rate coefficients calculated at these two times are due to breakthrough curve asymmetries. Figure 1 also shows that these asymmetries increased with decreasing granule sizes in the back layer of the bed. Capacities and pressure drops were unchanged. Average pressure drops increased from 35 Pa for all 12-30 mesh carbon to only 37 and 47 Pa for back layers of 20-30 and 30-40 mesh carbons, respectively.

^{**} SD = Sample Standard Deviation

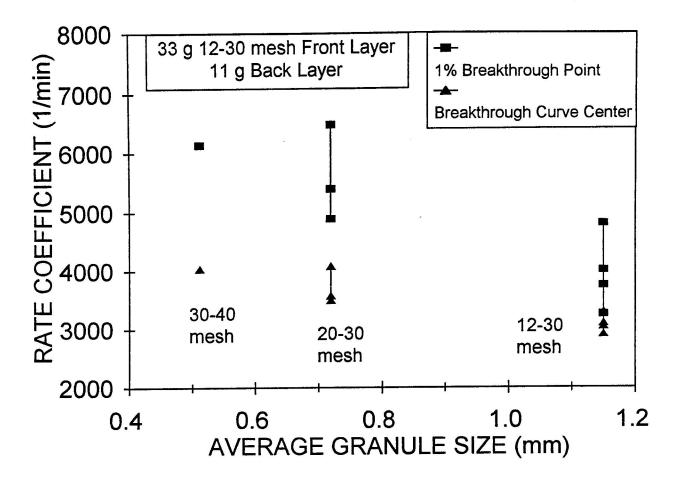


Figure 1. Back layer granule size effects on butane adsorption rate coefficients.

In the third series of experiments we looked at the effect of splitting the original 12-30 ASZM-T carbon into two fractions and then layering them. Upon sieving, the weight ratio of the 12-20 and 20-30 mesh fractions was 7:2. For consistency with previous experiments, tests were done with layered beds consisting of 33 g of 12-20 mesh carbon followed by 11 g of 20-30 mesh. Figure 2 shows that the adsorption rate coefficients calculated at the breakthrough curve centers (stoichiometric times) and at 1% breakthrough times and the breakthrough curve asymmetries increased by this process. Capacities and pressure drops were unchanged.

DISCUSSION

Even though rate coefficient increases up to almost 50% were accomplished by layering, corresponding breakthrough time increases were much smaller, due to the dominance of the capacity in determining breakthrough time. For example, the test with a 30-40 mesh carbon back layer gave a 1% rate coefficient of 6140 min⁻¹ compared with 4160 min⁻¹ for only 12-30 mesh

carbon. At the same equilibrium capacity and test conditions, these correspond to 1% butane breakthrough times of 64 min vs. 60 min, respectively. Enhancement of bed life by layering may be greater for other adsorbates, carbons, and use conditions.

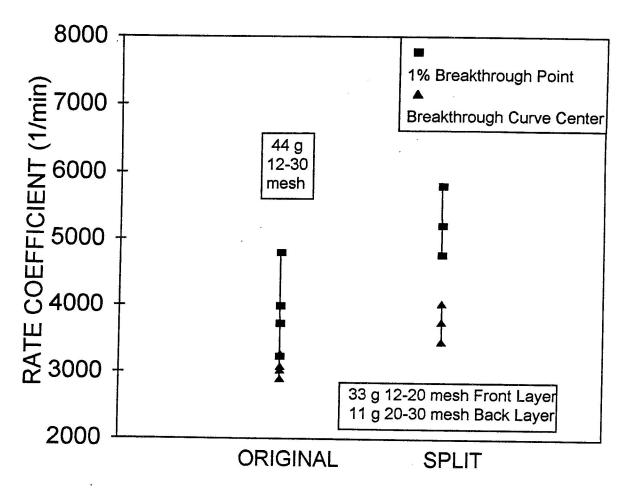


Figure 2. Effect of splitting 12-30 mesh carbon on butane adsorption rate coefficients.

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MAY 1996

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