THE ADSORPTION PROPERTIES OF PHENOLIC RESIN CARBON MONOLITHS

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Introduction

CBD Porton Down is investigating the use of phenolic resin based carbon monoliths for filtration applications. The materials under evaluation are obtained through collaboration with MAST Carbon Ltd. The monoliths are prepared from a phenolic resin precursor [1] and after carbonisation possess a nitrogen BET surface area of approximately 600 m^2g^{-1}. Selected monolith samples have been further activated, increasing the surface area to *ca*. 1000 m^2g^{-1}. The samples are referred to as "carbonised" or "activated" to distinguish their use in this work.

For the monoliths described in this abstract, the structure consists of a 3 cm diameter cylinder containing continual longitudinal square channels approximately 1 mm across, separated by carbon walls of approximately 0.8 mm thickness. The monoliths introduce only a minimal resistance into the air flow, offering the potential to provide significant levels of protection with a very low associated pressure drop.

This abstract describes research to evaluate the adsorption properties of these carbon monoliths against a range of hydrocarbons.

Experimental

Small sections (2 – 10 cm length) of monolith were sealed using "heat-shrink" rubber tubing and mounted in a small scale filter testing apparatus. This enabled the samples to be exposed to controlled concentrations of various test species at constant temperature and relative humidity. All testing reported here was carried out at 22±1°C and at a volumetric flow rate of 2 l min^{-1}. The effluent was monitored using either packed column gas chromatography or quadrupole mass spectrometry, internally calibrated in both cases against the influent concentration level.

In order to assess the leakage of volatile species through monolith sections, short lengths were tested with butane at a range of flow rates and concentrations. This was repeated with monoliths split into several smaller sections to assess the effect on the leak concentration.

Results and Discussion

Short sections (2 cm) of carbonised monolith were tested against 8000 mg m^3 hexane in either dry (<5% RH) or humid (80% RH) air. The breakthrough times (time taken for the effluent concentration to reach 1% of the influent) were 100 minutes and 40 minutes respectively. The breakthrough curves are shown in Figure 1.
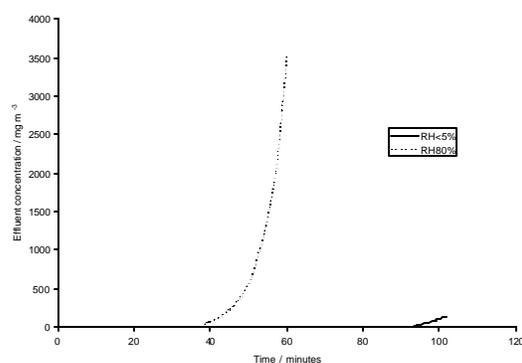

Figure 1. Breakthrough curves for hexane through 2 cm lengths of "carbonised" monolith (3 cm diameter, 2 l min^{-1} flow, 8000 mg m^3 influent concentration)

These results showed a remarkable level of protection for such a short length of monolith; the pressure drop was below measurable levels using a digital manometer (Digitron P200UL).

Figure 2 illustrates the breakthrough curves resulting from exposure of a 7 cm section of carbonised monolith to 8000 mg m^3 butane in dry (<5% RH) air. It can be seen that a small, relatively constant concentration of butane (*ca*. 5% of the influent level) is present in the effluent almost from the start of the test.
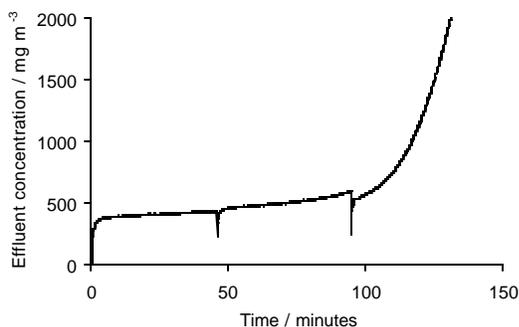
Figure 2. Breakthrough curve for butane through 7 cm length of "carbonised" monolith (3 cm diameter, 2 l min^{-1} flow, 8000 mg m^{-3} influent concentration, RH<5%)

Since the "leak concentration" remains relatively constant from the start of the test until full breakthrough occurs, the relationships between this concentration and the flow rate and influent concentration could be investigated. Figure 3 illustrates selected data from this study.
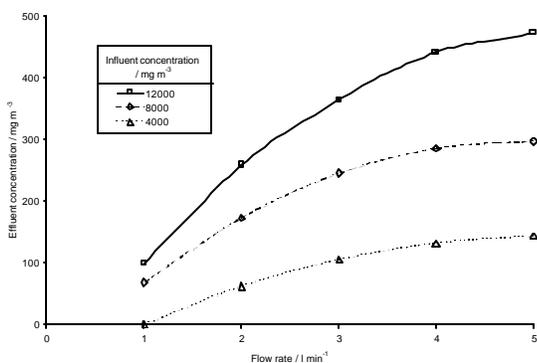

Figure 3. Relationship between "leak concentration" and flow rate for "carbonised" monolith sections exposed to butane (10 cm length, 2 l min^{-1} flow rate, RH<5%)

Two possible reasons were proposed for this "leak effect": firstly that a small proportion of the influent butane molecules were travelling down the centre of the monolith channels, or secondly that although collisions were occurring with the walls, the interaction was not sufficiently favourable for the butane molecules to be retained on the carbon.

Methods were devised to test both scenarios. It was postulated that the flow regime changed from predominantly turbulent towards laminar as the challenge vapour passed through the monolith channels (the monolith effectively acts as a laminar flow element). Collisions between the butane molecules and the walls are likely to be more frequent in the turbulent zone.

Monolith sections were therefore cut into two or more sections and arranged in various configurations to assess the impact of increasing the proportion of the turbulent flow zone.

Figure 4 illustrates the effect on the leak concentration of splitting the monolith into smaller sections. 4 cm and 6 cm sections have been cut into 2 cm sections and mounted either with or without intermediate 1 cm gaps. The results are consistent with the suggestion that increasing the amount of turbulence in the flow would reduce the leak effect. Computational Fluid Dynamics (CFD) studies are currently being carried out to further investigate the flow patterns within monolith channels.
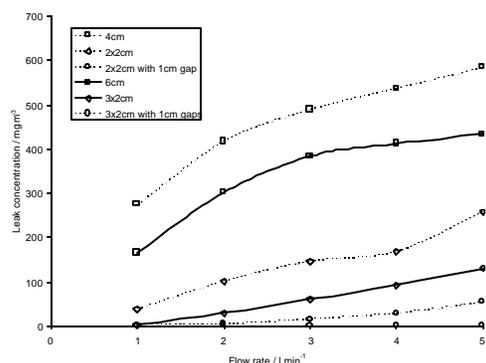

Figure 4. Effect of sectioning monolith lengths on butane "leak concentration" (2 l min^{-1} flow rate, RH<5%)

In a further attempt to minimise leak effects, monoliths were activated to increase their nitrogen BET surface area. Figure 5 compares breakthrough curves for "carbonised" and "activated" sections of monolith chellenged with 8000 mg m^{-3} butane. The activated section shows no significant leakage of butane during the test, as well as demonstrating a higher overall adsorption capacity. This suggests that the collisions between the adsorptive molecules and the carbon are more likely to result in adsorption in the activated section compared to the carbonised monolith.
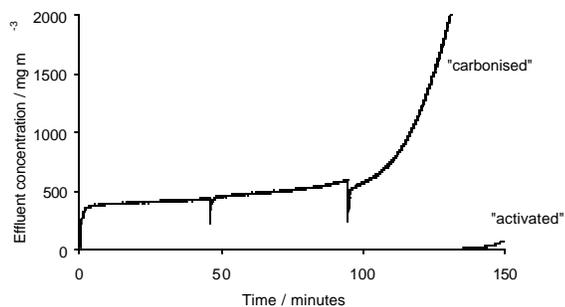
Figure 5. Breakthrough curves for butane through 7 cm length of monolith (3 cm diameter, 2 l min^{-1} flow, 8000 mg m^{-3} influent concentration, RH<5%)

The data clearly show that activation of the monolith removes the leak concentration, leaving a breakthrough time in excess of 130 minutes, which again represents significant adsorption of this relatively volatile adsorptive.

Conclusions

Phenolic resin based carbon monoliths have been shown to possess remarkable adsorption properties, even when tested at very short lengths. This coupled with the very low associated pressure drop clearly demonstrates their potential for use in a range of adsorption applications.

For volatile adsorptives such as butane, activation of the monoliths has been shown to be beneficial in obtaining maximum protection times.

The carbon monoliths are also good electrical conductors, due to their continuous carbon structure, and can be rapidly heated by applying a potential difference across them. This offers potential for use in regenerable filtration, an aspect which is currently being investigated at CBD Porton Down.

References

[1] Tennison S.R. Applied Catalysis A:General 1998:173;289-311

Acknowledgements