

CARBON BONDED CARBON FIBER COMPOSITES FOR REGENERABLE FILTRATION

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Introduction

CBD Porton Down is investigating the use of carbon bonded carbon fibre composites for adsorption applications. Composites have been obtained from the Centre for Applied Energy Research at the University of Kentucky and from Oak Ridge National Laboratory, Tennessee. The materials are prepared from a pitch-based carbon fibre precursor, which is mixed with a binder to form a monolith and then activated to form a highly porous composite (nitrogen BET surface areas of up to *ca.* $1700 \text{ m}^2 \text{ g}^{-1}$ have been measured for the samples used in this work). The continuous carbon skeleton means that the composites conduct electricity, and can thus be directly heated by applying a potential difference across them. For this reason, particular emphasis is being placed on assessing their potential for use in regenerable filtration.

Experimental

Small sections of composite 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 $22 \pm 1^\circ\text{C}$ 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.

For regenerability tests, the composite was “challenged” for a set time period as above. The flow direction was then reversed and a potential difference applied across the composite. This was controlled to initially achieve rapid heating of the composite and then to maintain it at the required temperature. After a set time, the power was isolated and the composite allowed to cool prior to the next adsorption phase. The amount desorbed was analysed using a quadrupole mass spectrometer.

Results and Discussion

Figure 1 illustrates breakthrough profiles for 2 cm lengths of carbon composite challenged with 8000 mg m^{-3} hexane in humid (RH80%) air. In particular, the contrast is shown between sections of low (0.11 g cm^{-3}) and high (0.75 g cm^{-3}) density composites. The high density composite shows a higher adsorption capacity, due to the higher mass of carbon. However the high density is accompanied by a high pressure drop (78 cm water gauge compared to $<1 \text{ cm}$ for the low density composite). This is thought to promote the presence of leak paths through the composite, resulting in an earlier initial breakthrough for this sample.

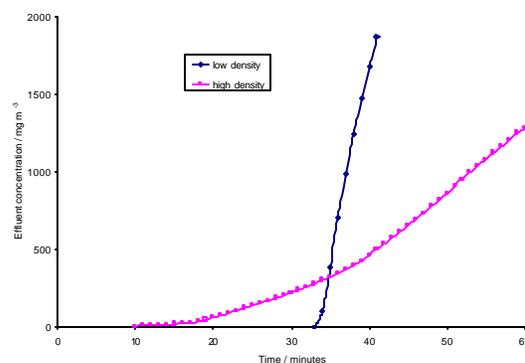


Figure 1. Breakthrough profiles for 2 cm length, 2.5 cm diameter carbon fibre composites challenged with 4000 mg m^{-3} hexane in RH80% air at 22°C at 4.8 cm s^{-1} linear flow velocity

Figure 2 compares breakthrough times (*i.e.* time for the effluent concentration to reach 1% of the influent level) for hexane, cyanogen chloride and hydrogen cyanide, tested in dry (RH $<5\%$) conditions. Data has been measured for composites containing different ratios of carbon fibre to binder, and for composites which have been activated to various burn-off levels. It is clear that different combinations give the longest breakthrough times for the polar and non-polar species. For hexane, the longest breakthrough results from the higher burn-off / low binder combinations. However for the polar adsorptives, longer breakthrough times are observed for the low burn-off / high binder content composites.

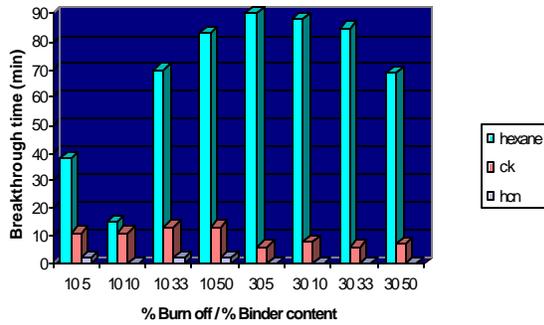


Figure 2. Comparison of breakthrough times for hexane, cyanogen chloride (CK) and hydrogen cyanide through carbon fibre composites with varying burn-off and binder content.

The data illustrate the need to balance the various effects in order to achieve the optimum adsorption performance against a wide range of contaminants. Furthermore, the parameters which have been varied here also affect the physical strength of the composites, another important consideration for most practical applications.

The continuous carbon skeleton present in the carbon fibre composites means that they are good electrical conductors, and can rapidly be heated by applying a potential difference across them. This, in conjunction with their adsorption properties, offers potential for use in regenerable filtration applications. Extensive reports in the literature describe their use in this way for separation of volatile species [e.g. 1-3].

CBD are investigating the “electrical swing adsorption” filtration method for providing protection against a wide spectrum of contaminant species, many of which are significantly less volatile than those which have been studied previously. This imposes additional constraints on the adsorbents, since high boiling species may be so strongly bound to the carbon that they can not be easily desorbed, thereby reducing the available capacity for further adsorption cycles.

Figure 3 shows data from two adsorption / regeneration cycles during test of a 7cm length of composite with hexane under dry (RH<5%) test conditions. No breakthrough was observed during the adsorption phase. Approximately 14 Watts of electrical power have been applied during regeneration, heating the composite to approximately 120°C. Under these conditions a significant amount, but not all, of the hexane is desorbed. During the second regeneration, a greater quantity of hexane is desorbed. This trend continues, and is further illustrated in Figure 4, which contrasts residual loadings

during four cycles for low and high burn off composite samples.

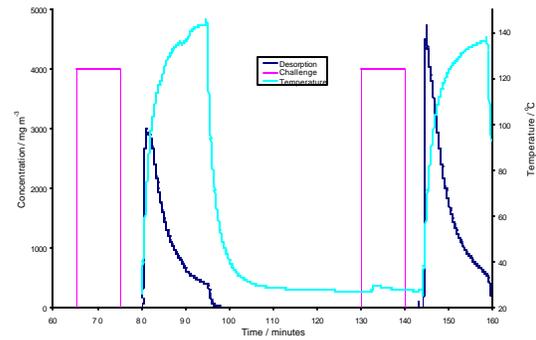


Figure 3. Illustrative adsorption / regeneration cycles for 7cm composite challenged with 4000 mg m⁻³ hexane under dry (RH<5%) conditions.

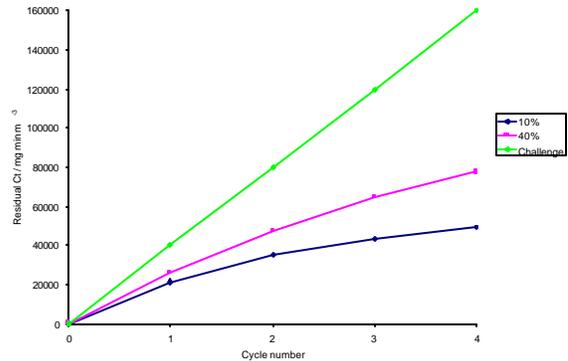


Figure 4. Illustrative residual loadings after each of four adsorption / regeneration cycles for 7cm composite (10% or 40% burn-off) challenged with 4000 mg m⁻³ hexane under dry (RH<5%) conditions.

The data in Figures 2 and 4 show that although the higher burn-off levels result in higher adsorption capacities for hexane, the residual loadings are also higher after regeneration. The trends in both sets of data are for the residual levels to reach a constant value, which is assumed to represent hexane strongly bound within the micropores. No breakthrough was observed during any of the adsorption cycles.

It should be noted that the conditions for these initial tests have been arbitrarily chosen, but the results indicate the potential of these materials as regenerable adsorbents.

Detailed studies using a wide range of adsorptives are therefore being carried out, and further data from the research will be presented.

Conclusions

Carbon bonded carbon fibre composites have been shown to have significant adsorption capacities for a wide range of adsorptives. The adsorption performance is dependent on factors such as the composite density, level of activation and binder content.

The composite materials can be rapidly heated by direct electrical heating, offering scope for use in regenerable filtration applications. In order to achieve the most efficient adsorbent for removing a wide range of contaminants, consideration will need to be given to the optimum properties for achieving efficient adsorption without leaving high residual levels on the carbon after regeneration.

References

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