PHYSICAL AND CHEMICAL ACTIVATION OF CARBON FIBERS AND THEIR ADSORPTION PROPERTIES UNDER DYNAMIC CONDITIONS

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Introduction

A considerable amount of research has been carried out into the adsorption properties of activated carbons under static conditions[1,2]. Information on the performance of these materials under dynamic adsorption conditions is less readily available. In this study either pitch-based or PAN-based carbon fibres have been activated by either physical or chemical methods. The resulting activated carbon fibres have been characterised by static adsorption methods in terms of their specific surface area, micropore volume, mesopore volume, water uptake, and hexane uptake.

The performance of the activated carbon fibres against a typical non-polar physisorbed challenge, *e.g.* hexane has been investigated using dynamic filter testing methods. The effectiveness of the activated carbon fibres to remove these vapour challenges under either dry or humid conditions is described.

Experimental

Activated carbon fibres were prepared by both physical and chemical methods. Pitch-based carbon fibres were physically activated to various degrees of burn-off using either CO_2 or steam. PAN-based carbon fibres were activated by chemical methods. The fibre was immersed in various concentrations of either $ZnCl_2$ or KOH prior to heat treatment to 700^{o}C in N_2 for 1 hour.

Activated Pitch-based or PAN-based carbon fibres (0.8g) were mixed with water and drawn into a volume analysis (VA) tube (2 cm diameter x 2.5 cm length) using a modified Büchner funnel technique. The samples were then dried in a vacuum oven for 4 hours. A hexane/ air mixture of concentration 4000 mg m^{-3} was prepared using either dry or humid (RH80%) air.

The VA tube containing the fibres was placed in-line and 4000 mg m^{-3} of hexane was pulled through the sample at a flow rate of 1 dm^3 min^{-1}. The effluent was monitored using a quadrupole mass spectrometer. Adsorbent testing was carried out at 22^{o}C for all experiments. For the experiments under dry conditions both the hexane/ air mixture and the activated fibres were dry (<5 % RH). For the experiments under humid conditions, the hexane/ air challenge was prepared using air of RH80%. The fibres were also pre-wetted by placing them in an environmental chamber set at RH80% until they reached equilibrium.

Results and Discussion

The physical characterisation data obtained from static adsorption measurements for the activated carbon fibres are shown in Table 1. Dynamic breakthrough curves for the physically activated carbon fibres are shown in Figure 1.

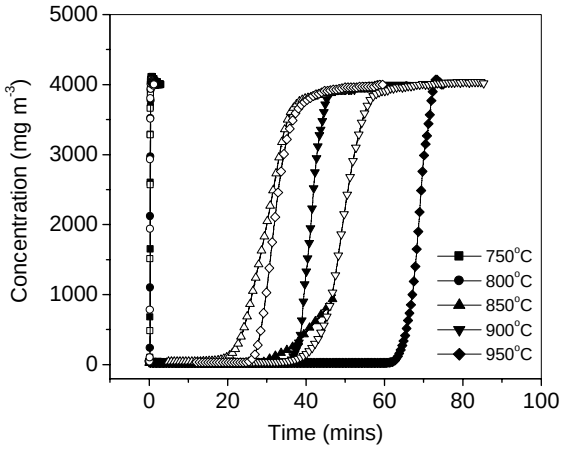

Figure 1 Dynamic hexane breakthrough profiles under both dry (closed symbols) and humid (open symbols) conditions for physically activated carbon fibres

The shape of the breakthrough profiles shows that maximum hexane breakthrough is achieved a relatively short time after the initial breakthrough. Figure 2 shows the time to initial breakthrough as a function of burn-off (%). Under dry conditions (closed symbols) there is an almost linear increase in the time to breakthrough with increasing burn-off (%). However, under humid conditions (open symbols), following an initial increase in time to breakthrough with burn-off, a decrease is observed at high burn-off values (~80%). Looking at the water uptakes for the carbon fibres under static adsorption conditions shows that the fibre at burn-off ~80% has a water uptake of ~90 %. This shows that the

competing water adsorption greatly diminishes the hexane adsorption capacity of the fibre.

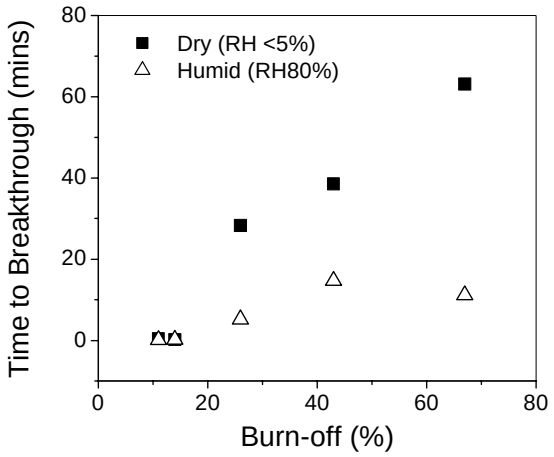

Figure 2 Variation in time to breakthrough with burn-off for the physically activated pitch-based carbon fibres

The hexane breakthrough profiles for the chemically activated carbon fibres are shown in Figure 3.

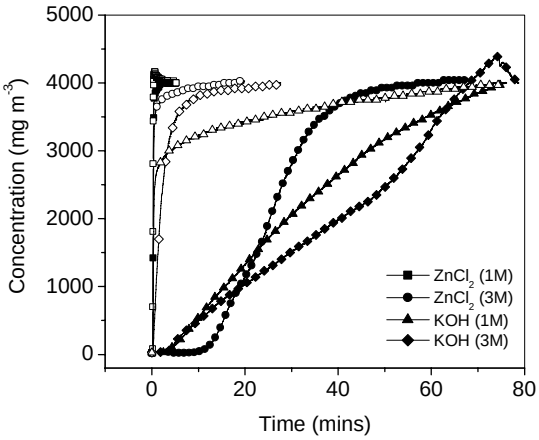

Figure 3 Dynamic hexane breakthrough profiles under both dry (closed symbols) and humid (open symbols) conditions for chemically activated PAN-based carbon fibres

With the exception of the zinc chloride (3M) sample breakthrough is almost instantaneous in all cases whether dry or humid. However, in contrast to the physically activated carbon fibres the time to reach maximum breakthrough concentration is much longer. The chemically activated carbon fibres did not maintain the fibrous nature of the original carbon fibre and channelling may occur in the bed due to poor packing. In all cases the performance of the fibres under humid conditions was poor.

A look at the time to breakthrough as a function of activating agent and activating agent concentration (Figure 4) shows that the times to breakthrough for the fibres are poor in relation to the physically activated fibres. However, as expected the 3M activated fibres perform better than those activated using 1M solutions due to their higher surface areas.

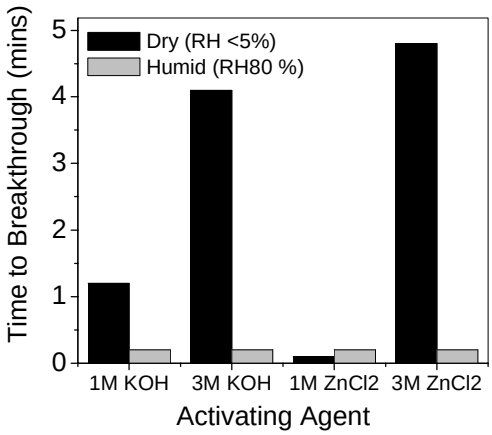

Figure 4 Variation in time to breakthrough with burn-off for the chemically activated PAN-based carbon fibres

Conclusions

In conclusion it is apparent that the physically activated pitch-based carbon fibres exhibit good performance against hexane. However, at high degrees of burn-off, greater amounts of water are adsorbed. This leads to poorer performance against hexane under humid conditions. The fibre activated at 900^{o}C shows the best performance against hexane in both dry and humid environments.

For the chemically activated PAN-based carbon fibres breakthrough was almost instantaneous, although under dry conditions, the time to reach maximum breakthrough concentration was relatively slow.

References

1. Bansal, R.C., Donnet, J.B., and Stoeckli, H.F., *Active Carbon*, Marsel Dekker, 1998

2. Marsh, H., Rodrigues-Reinoso, F., and Heintz, E., *Introduction to Carbon Technologies*, Universidad de Alicante, 1998

Acknowledgement

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Table 1 Physical characterisation data for the physically activated pitch-based carbon fibres and chemically activated PAN-based carbon fibres

Sample	Burn-off (%)	Water Uptake RH95% (%)	Hexane Uptake at P/P_0 =0.9 (%)	BET Surface Area (m^2g^{-1})	Total Pore Volume (cm^3g^{-1})	Micropore Volume (cm^3g^{-1})	Mesopore Volume (cm^3g^{-1})
Physically Activated Pitch-based Carbon Fibres							
Pitch-750	11	11.8	0.5	50	0.04	-	-
Pitch-800	14	14.2	0.9	353	0.155	0.131	0.024
Pitch-850	26	25.3	21.3	798	0.319	0.313	0.006
Pitch-900	43	44.1	31.8	1531	0.624	0.600	0.024
Pitch-950	67	86.2	50.5	2110	1.007	0.914	0.093
Chemically Activated PAN-based Carbon Fibres							
PAN-1M KOH	40	29.0	12.8	285	0.156	0.094	0.062
PAN-3M KOH	42	33.4	30.8	1200	0.505	0.463	0.04
PAN-1M $ZnCl_2$	25	11.4	0	33	-	-	-
PAN-3M $ZnCl_2$	32	30.1	21.9	948	0.350	0.325	0.025