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M. Ribeiro Carrott[a], (✉), P.J.M. Carrott[a], P.F.M.M. Correia[b]

[a] Centro de Química de Évora, LAQV-REQUIMTE, Instituto de Investigação e Formação Avançada, and Departamento de Química, Escola de Ciências e Tecnologia, Universidade de Évora, Colégio Luís António Verney, 7000-671 Évora, Portugal

[b] SGL Composites, S. A., Apartado 5, 2836-908 Lavradio, Portugal

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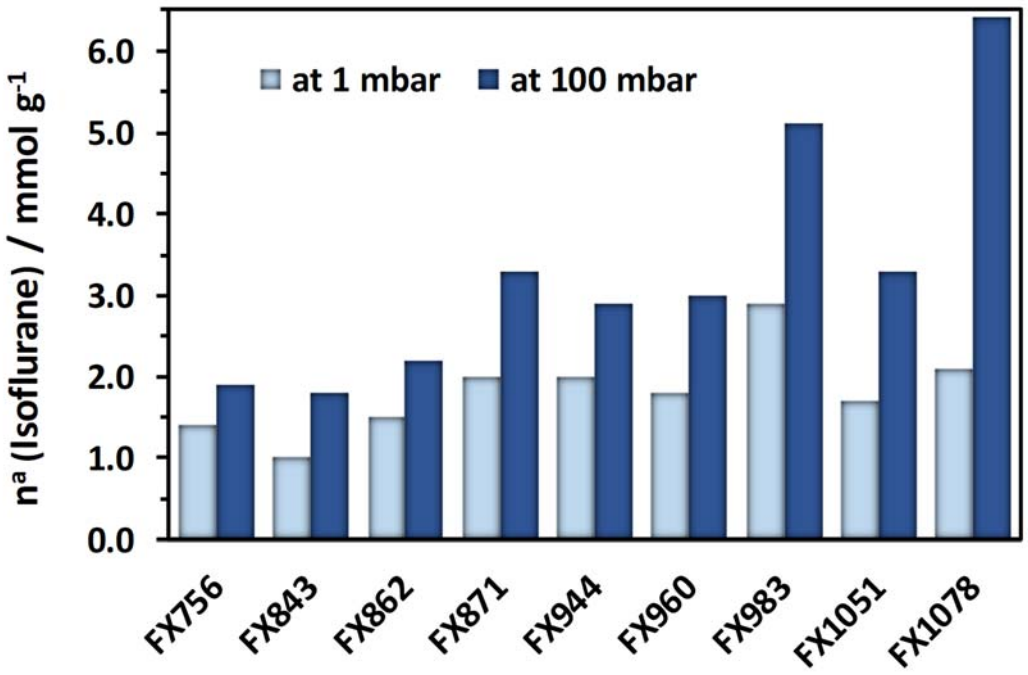
Graphical Abstract



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[b] SGL Composites, S. A., Apartado 5, 2836-908 Lavradio, Portugal

M. Ribeiro Carrott (✉)
E-mail address: manrc@uevora.pt
ORCID: 0000-0003-4856-5150

P.J.M. Carrott
ORCID: 0000-0002-9065-7000

Abstract

Activated carbon fibres with a range of pore sizes, prepared from pre-oxidized acrylic fibre by physical activation in CO_2 at different temperatures and times, were investigated as adsorbents for the inhalation anaesthetic isoflurane (2-chloro-2-(difluoromethoxy)-1,1,1-trifluoroethane). Adsorption-desorption isotherms of isoflurane were determined at 293 K and were analysed by the α_S method using reference adsorption data, determined also at 293 K, on non-porous carbon. One of the samples obtained showed a much higher adsorption uptake at 100 mbar than reported in previous studies on different materials, while another sample not only showed a higher adsorption uptake than reported previously, but also a higher adsorption uptake at a pressure as low as 1 mbar showing potential for the capture of isoflurane. The results indicate that isoflurane does not adsorb in fine ultramicropores, of width less than about 0.5 nm. Comparison of the pore size distributions of these materials indicated that the presence of supermicropores, as well as ultramicropores, is relevant for the adsorption of isoflurane even at trace levels. Reference adsorption data for isoflurane on non-porous carbon is also presented.

Keywords

Activated carbon fibres; Acrylic fibre; Isoflurane adsorption; Anaesthetic; Carbon black; Reference adsorption data

1 Introduction

The inhalation anaesthetics which are most widely used in both human and veterinary surgery are fluorinated ethers. They offer good performance with minimal direct side effects for the patient when correctly administered. However, in order to protect the surgical staff, they should not be allowed to accumulate in the air of the operating theatre. Furthermore, after surgery the inhaled anaesthetics will be slowly released by the patient's respiratory system and hence appropriate air conditioning systems with filters for capturing the exhaled anaesthetics must be installed in post-operation recovery rooms. There are also other reasons for capturing and recovering used anaesthetics. On the one hand, they are potent greenhouse gases making a significant contribution to global warming (Sherman and McGain 2016) and may also react with stratospheric ozone (Mehrata *et al.* 2016). For both reasons they should not be vented to the atmosphere. On the other hand, they are comparatively expensive, and their recycling would contribute towards a reduction in health care costs.

Several studies have been carried out to evaluate the effectiveness of different types of adsorbent for capturing anaesthetics. In the case of isoflurane, these include carbons (Kim and Sircar 1977; Mehrata *et al.* 2016; Ortmann *et al.* 2016), zeolites (Doyle e*t al.* 2002; Mehrata *et al.* 2016; Ortmann *et al.* 2016), silica gel (Mehrata *et al.* 2016), porous molecular crystals (Chen e*t al.* 2015) and metal-organic frameworks (MOFs) (Abrahams e*t al.* 2017). As the isoflurane molecule is relatively small the adsorbents should, in principle, contain a high volume of micropores. In addition, the rate of adsorption from a gas flow should be rapid, subsequent desorption of the anaesthetic and regeneration of the adsorbent should be possible under relatively mild conditions, and it should be possible to make a reusable low pressure drop filter unit.

In recent work we have demonstrated that an industrially pre-oxidized acrylic fibre can be used as precursor for making activated carbon fibres by physical activation in CO_2 and that the pore size can be tailored in order to obtain different materials ranging from those which contain

only ultramicropores to others with a pore size distribution extending up to 4 nm (Carrott *et al.* 2018). The principal objective of the present work reported here was to evaluate the performance of these materials in adsorbing and capturing isoflurane and to identify the optimal range of pore sizes. For the analysis of the adsorption data it was necessary to establish a reference adsorption isotherm on a non-porous carbon surface, and the corresponding data are also presented here.

2 Experimental

The samples of activated carbon fibres used in this work were some from previous work (Carrott *et al.* 2018). The precursor was a pre-oxidized acrylic fibre specially prepared by SGL Composites, S. A. The samples are designated FXtq, where t is the activation temperature in ^{o}C divided by 100 ^{o}C and q is the % burn-off. Details of the preparation of the activated carbon fibres, by physical activation with CO_2 at temperatures from 700 to 1000 ^{o}C, and results of the characterisation by nitrogen adsorption at 77 K using a Quantachrome Autosorb iQ, were previously presented (Carrott *et al.*, 2018). A sample of non-porous carbon black N375, which has featured in previous works (Carrott *et al.* 2000; Carrott *et al.* 2001a; Carrott *et al.* 2001b), was also used.

The anaesthetic used in this work was isoflurane (2-chloro-2-(difluoromethoxy)-1,1,1-trifluoroethane) of purity > 99.9 % from Abbott Laboratories Ltd. Before determination of the isotherms the isoflurane was thoroughly outgassed by three freeze-thaw cycles. Adsorption-desorption isotherms of isoflurane vapour were determined gravimetrically at 293 K using a CI Precision vacuum microbalance coupled to a Disbal control unit with pressure measurement by means of an Edwards Barocel 600 capacitance manometer. The vapour pressure was taken as 317.31 mbar and the liquid density as 1.50 g cm^{-3}. The minimum diameter of the isoflurane molecule is 0.52 nm (Ortmann *et al.* 2015), while for N_2 is 0.30 nm (Gregg and Sing 1982).

3 Results and discussion

3.1 Adsorption of isoflurane by non-porous carbon

The adsorption isotherm of isoflurane on the non-porous carbon black N375 was Type II regarding IUPAC classification (Thommes *et al.* 2015). The BET equation was applied using the criteria recommended by Rouquerol *et al*. (1999) and subsequently endorsed by IUPAC (Thommes *et al.* 2015). A value of 49 was obtained for the C parameter, indicating a moderately strong interaction of the isoflurane molecule with the carbon surface, and a monolayer capacity of 0.268 mmol g^{-1}. From the monolayer capacity and the known specific surface area of N375, namely 107 m^2 g^{-1}, a value of 0.66 nm^2 was calculated for the mean cross-sectional area of the isoflurane molecule.

The reference data, which will be used in the following section, was obtained using essentially the same procedure as that used in previous works where reference data for benzene (Carrott *et al.* 2000), methanol (Carrott *et al.* 2001a) and dichloromethane (Carrott *et al.* 2001b), were presented. The data are given in Table 1, where the values of α_s are the ratio of amount adsorbed by the non-porous carbon at relative pressure p/p^o to that at p/p^o=0.4. The factor for calculating specific surface area from the slope of an α_s plot constructed with this data as reference is 280 m^2 $mmol^{-1}$.

3.2 Adsorption of isoflurane by activated carbon fibres

Representative isoflurane adsorption-desorption isotherms are shown in Fig. 1. The isotherms on the other samples are presented in Electronic Supplementary Material. Desorption points were omitted for clarity. For all the activated carbon fibres the isotherms are Type I regarding the IUPAC classification and completely reversible down to pressures of at least 5 mbar. However, when the samples were outgassed at 293 K after determination of the desorption branch of each isotherm it was found that not all isoflurane was removed. The residual adsorbed

amounts are presented in Table 2. If the values are compared with the adsorbed amounts at a pressure of 100 mbar, which in many cases is close to the plateau of the isotherm, it can be seen that the residual adsorbed amount was approximately 30% of the corresponding value at 100 mbar for FX756, the sample activated at the lowest temperature, but less for the other samples activated at higher temperatures. It was found necessary to increase the outgassing temperature up to 413 K in order to completely remove isoflurane from all samples.

Fig. 2 shows the α_s plots corresponding to the isotherms in Fig. 1. It can be seen that all the α_s plots are linear above a value of α_s approximately equal to 1, corresponding to a relative pressure of 0.4. For each sample the specific external surface area and total pore volume were calculated from the slope and intercept of the linear region of the α_s plot and the values obtained are presented in Table 2. It can be seen that the specific external surface area is quite low for all samples except FX1078. This is the sample with also the highest specific pore volume.

It can be seen in Fig. 2 that for all samples the α_S plots show the occurrence of micropore filling over a range of α_S indicating that the secondary cooperative filling of supermicropores (Gregg and Sing 1982, Thommes et al. 2015) occurred in all samples. The α_S plots also indicate clearly that primary filling of ultramicropores (Gregg and Sing 1982, Thommes et al. 2015) also occurred in all samples.

The values of total pore volume obtained in the present study from the α_s plots are compared in Fig. 3 with previously published values of the pore volumes obtained by adsorption of N_2 at 77 K (Carrott *et al.* 2018). There appears to be a linear relationship which passes through the origin of the graph. However, the slope of the plot is only 0.67, that is, much less than unity. Although it is possible that the density of the adsorbed phase is less than the normal liquid density, it is unlikely that there would be such a large difference. Hence, the much lower uptake of isoflurane, in comparison with N_2 at 77 K, also arises due to the impossibility of the larger isoflurane molecule to enter in fine ultramicropores which, based on the minimum diameter of the molecule, are ultramicropores of width less than about 0.5 nm.

The results discussed above indicate that isoflurane does not gain entry to the fine ultramicropores, but that isoflurane is very strongly adsorbed, at very low pressures and 293 K, in other ultramicropores from which it can only be desorbed by outgassing at higher temperature. In supermicropores, and eventually in small mesopores, the isoflurane is reversibly adsorbed.

In previously published work different authors have used different experimental techniques and ranges or values of pressure or partial pressure. In order to compare with our results we have included in Table 2 the adsorbed amounts of isoflurane on our activated carbon fibres at pressures of 300 mbar, 100 mbar and 1 mbar. The pressure of 300 mbar is close to the vapour pressure of isoflurane at 293 K. Thus, the adsorbed amount at this pressure will give an estimate of the total adsorption capacity of the adsorbent. The reason for considering a pressure of 1 mbar is the following. The strength of anaesthetic gases is expressed as a MAC (Minimum Alveolar Concentration) value which for isoflurane is approximately 1.17 % for humans, depending on age (Nickalls and Mapleson 2003). A pressure of 1mbar corresponds to a concentration approximately 10 times lower, namely 0.0932 %, 932 ppm (by volume) or 7.57 g m^{-3}, which are in the units used by different authors.

Kim and Sircar (1977) measured adsorption isotherms of several inhalation anaesthetics on a commercial activated carbon and showed that, with the exception of nitrous oxide, the characteristic curve concept applied. The adsorption capacity at pressures close to the saturated vapour pressure of each anaesthetic, including isoflurane, was about 4.1 mmol g^{-1}. Abrahams *et al.* (2017) obtained a similar result, 4.2 mmol g^{-1}, for the adsorption of isoflurane at 100 mbar on a MOF. Chen *et al.* (2015) used a flow of N_2 saturated with isoflurane and found a maximum adsorption by a porous molecular crystal of 3.2 mmol g^{-1}. Doyle *et al.* (2002) also using a flow system, studied the uptake of isoflurane by silicalite, but obtained adsorbed amounts which were much lower. The highest value of adsorption capacity which we have found was reported by Mehrata *et al.* (2016), who measured adsorption isotherms up to 36 mbar on commercial

activated carbons, silica gel and zeolites, and obtained the best result for one of the commercial activated carbons which had a maximum isoflurane adsorption of 5.0 mmol g^{-1}. It can be seen from Table 2 that the activated carbon fibre FX983 gives a slightly higher value, while the activated carbon fibre FX1078 has a much higher adsorption capacity of 6.9 mmol g^{-1}.

At an isoflurane pressure of 1 mbar the highest reported adsorption uptakes were 2.6 mmol g^{-1}, by Mehrata *et al.* (2016) on the same commercial activated carbon previously referred to, and 2.5 mmol g^{-1}, by Ortmann *et al.* (2016) on a highly microporous carbon and using a flow system. Adsorption uptakes of 1.9 mmol g^{-1} were also reported by Ortmann *et al.* (2016) on a zeolite and by Abrahams *et al.* (2017) on a MOF. The latter did not report the adsorption uptake at 1 mbar on their best MOF sample previously referred to above. In the present work the highest adsorption uptakes obtained at a pressure of 1 mbar were 2.1 mmol g^{-1} for sample FX1078 and, what is the highest value so far reported, 2.9 mmol g^{-1}, for sample FX983. It should be noted that, although FX983 has a lower pore volume than FX1078, the adsorption of isoflurane at 1 mbar is greater. This is evident not only from the isotherms in Fig. 1, which cross at a relative pressure of 0.105, corresponding to a pressure of 33.32 mbar, but also from the α_s plots in Fig. 2, and is associated with differences in the pore size distributions of the materials.

Pore size distributions for FX1078 and FX983 calculated by the QSDFT method (Neimark *et al.* 2009) from adsorption isotherms of N_2 at 77 K, are given in Fig. 4. The lower limit of analysis by this method was around 0.5 nm. It is therefore not possible to obtain information about fine ultramicropores. However, as mentioned above, isoflurane does not adsorb in these. It can be seen from Fig. 4 that a high proportion of the porosity of FX1078 consists of mesopores up to almost 4 nm in width, which is the reason for the higher adsorption capacity of this sample in comparison with the other samples. On the other hand, Fig. 4 also shows that FX983 contains a larger volume of both supermicropores (up to about 1.6 nm) and ultramicropores in comparison with FX1078, and this is the reason for its higher adsorption uptake at low pressures.

For all the samples in Table 2, prepared by activation at 700, 800 or 900 °C, the volume of ultramicropores is almost constant at 0.21±0.02 $cm^3 g^{-1}$ (Carrott *et al.* 2018) corresponding to an isoflurane adsorption uptake of approximately 1.2 mmol g^{-1}. For all but one of the samples this is less than the measured isoflurane adsorption even at 1 mbar, which indicates that the presence of supermicropores is also relevant for the adsorption of isoflurane even at trace levels.

4 Conclusions

Activated carbon fibres with a range of pore sizes, prepared from pre-oxidized acrylic fibres by physical activation in CO_2 at different temperatures and times, were investigated as adsorbents for the anaesthetic gas isoflurane. One of the samples, FX1078, prepared by activation at 1000 °C, had a much higher total adsorption capacity than reported in previous studies using activated carbons, zeolites, MOFs and porous molecular crystals. Another sample, FX983, prepared by activation at 900 °C, not only had a slightly higher total adsorption capacity than reported previously, but also a higher adsorption uptake at a low pressure, 1 mbar, corresponding to trace concentration levels, thereby showing potential for the capture of isoflurane. The results also showed that isoflurane does not adsorb in fine ultramicropores, of width less than about 0.5 nm, and that the presence of supermicropores, as well as ultramicropores, is relevant. The higher adsorption uptake of FX983 in relation to other materials is due to the greater contribution of supermicropores.

Acknowledgements

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Table 1 Reference data for isoflurane adsorption, at 293 K, on non-porous carbon (Specific surface area factor = 280 m^2 $mmol^{-1}$)

p/p^o	α_s	p/p^o	α_s	p/p^o	α_s	p/p^o	α_s
0.005	0.146	0.25	0.844	0.50	1.104	0.75	1.436
0.01	0.230	0.26	0.854	0.51	1.115	0.76	1.456
0.02	0.340	0.27	0.864	0.52	1.125	0.77	1.478
0.03	0.415	0.28	0.875	0.53	1.136	0.78	1.500
0.04	0.471	0.29	0.885	0.54	1.146	0.79	1.524
0.05	0.517	0.30	0.896	0.55	1.156	0.80	1.550
0.06	0.554	0.31	0.906	0.56	1.167	0.81	1.576
0.07	0.586	0.32	0.917	0.57	1.177	0.82	1.605
0.08	0.614	0.33	0.927	0.58	1.188	0.83	1.636
0.09	0.638	0.34	0.937	0.59	1.198	0.84	1.669
0.10	0.659	0.35	0.948	0.60	1.209	0.85	1.704
0.11	0.678	0.36	0.958	0.61	1.221	0.86	1.743
0.12	0.695	0.37	0.969	0.62	1.233	0.87	1.785
0.13	0.711	0.38	0.979	0.63	1.246	0.88	1.832
0.14	0.725	0.39	0.990	0.64	1.259	0.89	1.883
0.15	0.738	0.40	1.000	0.65	1.272	0.90	1.941
0.16	0.749	0.41	1.010	0.66	1.286	0.91	2.007
0.17	0.760	0.42	1.021	0.67	1.300	0.92	2.082
0.18	0.770	0.43	1.031	0.68	1.315	0.93	2.171
0.19	0.781	0.44	1.042	0.69	1.330	0.94	2.277
0.20	0.791	0.45	1.052	0.70	1.346	0.95	2.409
0.21	0.802	0.46	1.063	0.71	1.363	0.96	2.580
0.22	0.812	0.47	1.073	0.72	1.380	0.97	2.817
0.23	0.823	0.48	1.083	0.73	1.398	0.98	3.186
0.24	0.833	0.49	1.094	0.74	1.416	0.99	3.928

Table 2 Analysis of adsorption isotherms of isoflurane, at 293 K, on activated carbon fibres[a]

Sample	n^a (residual) mmol g^{-1}	A_S $m^2 g^{-1}$	v_S $cm^3 g^{-1}$	n^a (300 mbar) mmol g^{-1}	n^a (100 mbar) mmol g^{-1}	n^a (1 mbar) mmol g^{-1}
FX756	0.58	15.0	0.23	2.0	1.9	1.4
FX843	0.49	18.3	0.22	1.9	1.8	1.0
FX862	0.42	10.2	0.28	2.3	2.2	1.5
FX871	0.18	11.7	0.41	3.4	3.3	2.0
FX944	0.21	16.1	0.35	3.0	2.9	2.0
FX960	0.20	15.6	0.37	3.1	3.0	1.8
FX983	0.20	19.3	0.63	5.3	5.1	2.9
FX1051	0.28	17.3	0.41	3.4	3.3	1.7
FX1078	0.16	36.7	0.81	6.9	6.4	2.1

[a] n^a (residual) – amount retained upon outgassing at 293 K after measuring the desorption branch of the isotherm; A_S and v_S – specific external surface area and specific pore volume obtained from the α_s method; n^a (300 mbar), n^a (100 mbar) and n^a (1 mbar) – amounts adsorbed at 300 mbar, 100 mbar and 1 mbar.

Figure captions

Fig. 1 Representative adsorption-desorption isotherms of isoflurane, at 293 K, on activated carbon fibres (open points – adsorption; closed points – desorption)

Fig. 2 α_S plots, constructed with reference data on non-porous carbon presented in Table 1, corresponding to isotherms given in Fig. 1

Fig. 3 Comparison of total pore volume determined from the α_S plots corresponding to isoflurane adsorption isotherms at 293 K with total pore volume from nitrogen adsorption isotherms at 77 K

Fig. 4 QSDFT pore size distributions of selected samples calculated from nitrogen adsorption isotherms at 77 K

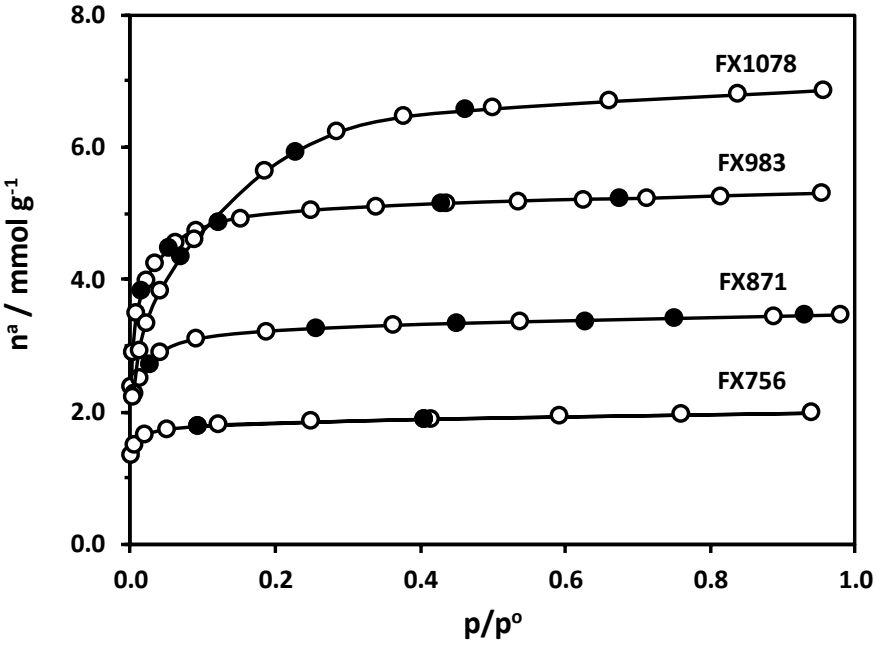
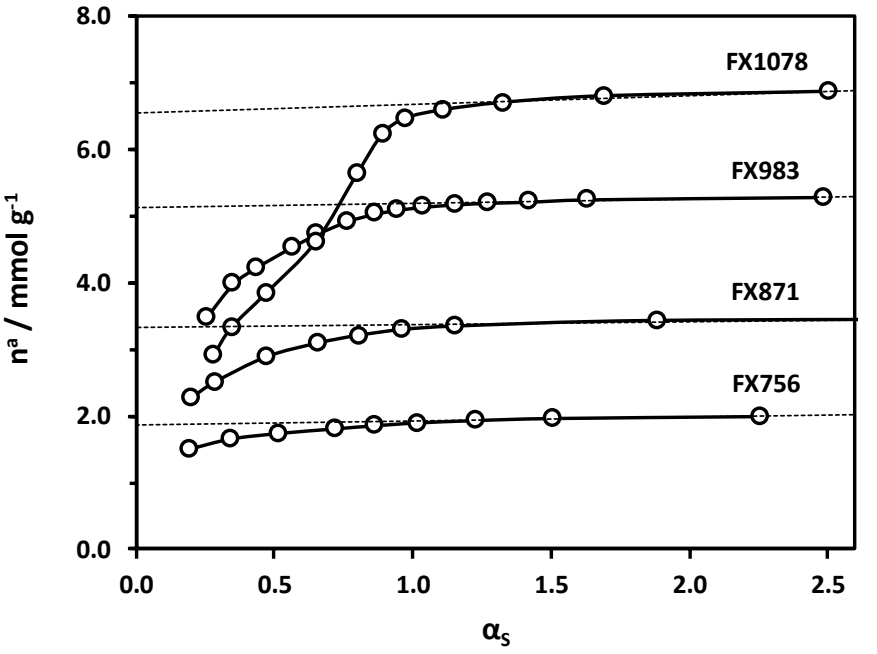

8.0
6.0
4.0
2.0
0.0
0.0
0.2
0.4
0.6
0.8
1.0
FX1078
FX983
FX871
FX756
n^a / mmol g^{-1}
p/p°

Fig. 1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6.0
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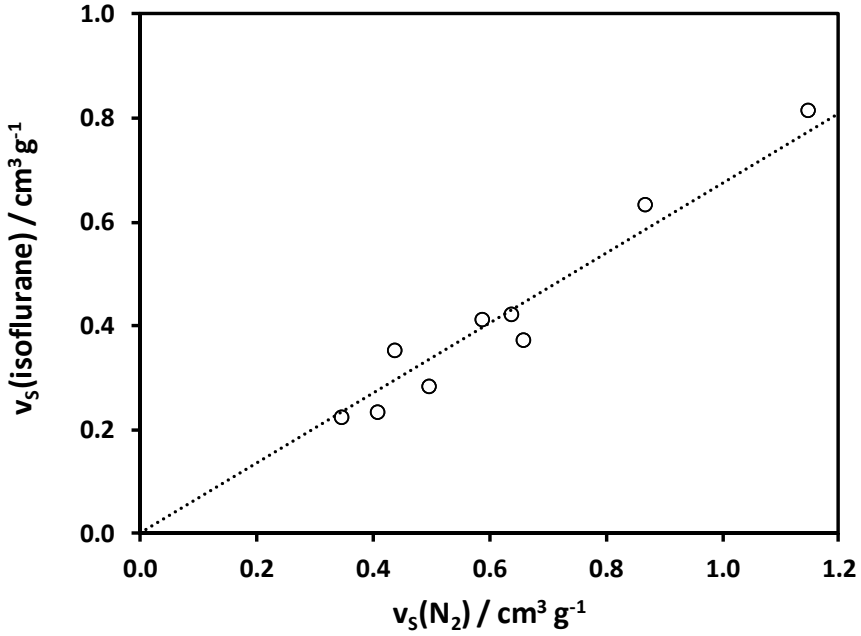
2.0
0.0
0.0
0.5
1.0
1.5
2.0
2.5
FX1078
FX983
FX871
FX756
n^a / mmol g^{-1}
α_S

Fig. 2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0.8
0.6
0.4
0.2
0.0
0.0
0.2
0.4
0.6
0.8
1.0
1.2
v_S(isoflurane) / cm^3 g^{-1}
v_S(N$_2$) / cm^3 g^{-1}

Fig. 3

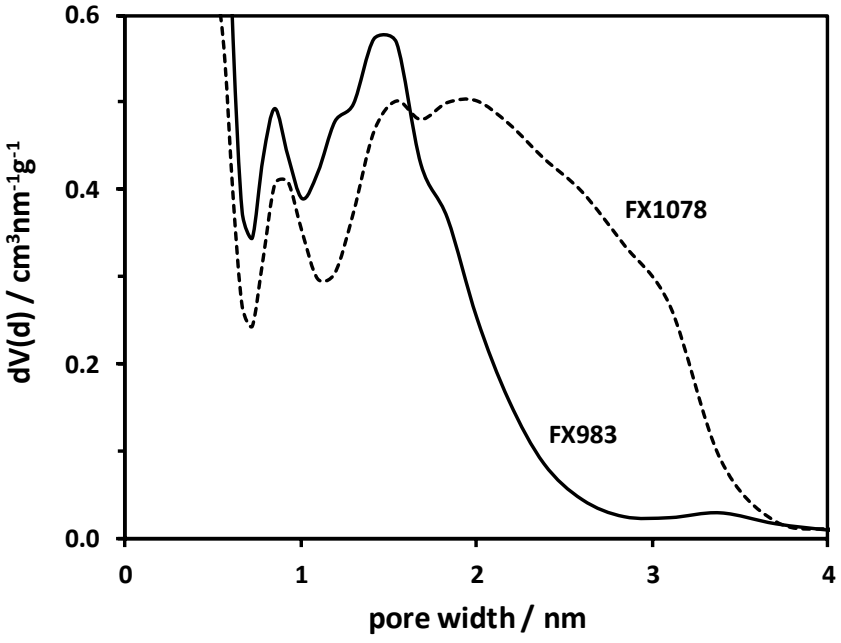
0.6
0.4
0.2
0.0
0
1
2
3
4
dV(d) / cm^{3}nm^{-1}g^{-1}
pore width / nm
FX1078
FX983

Fig. 4

Adsorption of the inhalation anaesthetic isoflurane by activated carbon fibres with reference data on non-porous carbon

Supplementary material

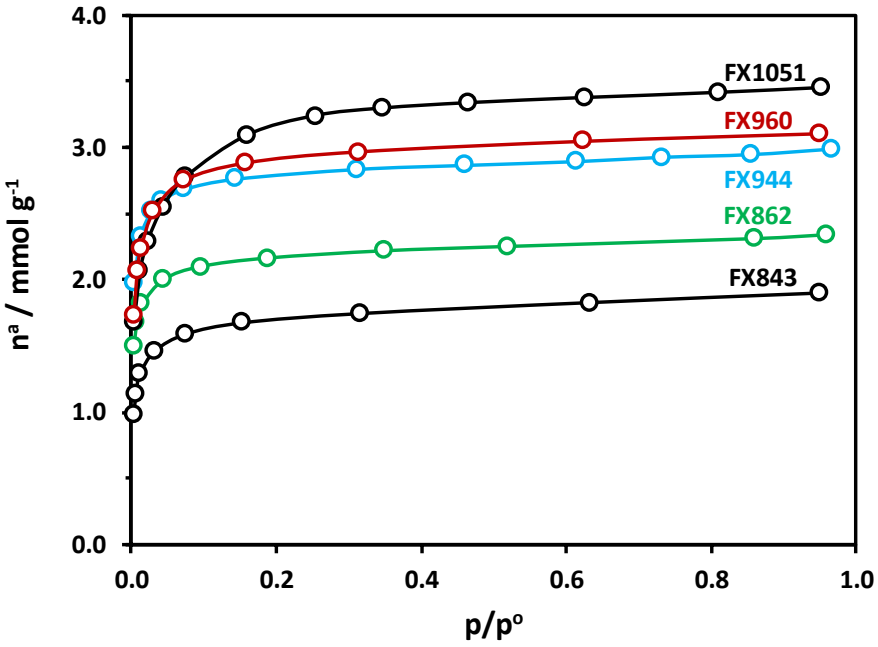

Fig. S1 Adsorption isotherms of isoflurane, at 293 K, on activated carbon fibres FX843, FX862, FX944, FX960 and FX1051