THE INFLUENCE OF DEPOSITION RATE ON IMPREGNANT UTILISATION

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Introduction

Considerable interest has been paid to the adsorptive properties of triethylenediamine (TEDA) impregnated carbons [1-3], which stemmed from their application in the nuclear industry and has been extended to the field of military adsorbents. Often, for applications where protection against a wide spectrum of vapours is required, several impregnants are applied to the same adsorbent. This can result in overloading of the carbon, causing a reduction in its physical adsorption capacity. In such systems the utilisation of the impregnant is of great importance. Previous studies have demonstrated the importance of correct carbon selection [4] and also that the conditions used for impregnation affect utilisation[5]. The work presented here has focused on varying the rate of deposition of TEDA onto coal based carbon and studying the effect this has on impregnant utilisation, with an appropriate probe gas. During this study the adsorption isotherm of TEDA on several carbons was recorded and is reported.

Experimental

The equipment used to vary the rate of TEDA deposition and measure the adsorption isotherm is shown in figure 1. TEDA vapour addition was controlled by a solenoid valve actuated by changes in the barocel pressure reading. Weight changes were recorded using a modified Ohaus balance. The rate of impregnation was varied by changing the temperature of the system. Samples (20g) of dry BPL were exposed to TEDA vapour until 2% uptake was achieved. At this point the sample was removed. Uptake was confirmed using a standard analytical balance. Utilisation of TEDA was measured using the probe gas 3-chloropentafluoropropene (CPFP), as described previously [6]. Full TEDA adsorption isotherms were measured on carbons derived from coal, nutshell and wood.

Results and Discussion

All isotherms were measured at 80°C to ensure that the saturated vapour pressure was high enough to allow accurate control at low partial pressure. Figure 2 compares the adsorption isotherms for three of the samples studied. All three are type I according to the IUPAC classification, indicating that adsorption of TEDA into the micropores predominates. The shape of the isotherm for the BAX (wood based) sample is indicative of micropore and mesopore filling. Nitrogen isotherm data in table 1 shows that BAX and SSC possesses similar nitrogen total pore volume. However the difference in the corresponding TEDA isotherms indicates that greater TEDA capacity is observed with predominantly microporous carbons.

It is clear from figure 3 that the rate of uptake of TEDA on BPL is highly dependent on temperature, which as a consequence allows easy control of the rate of impregnation. It should be noted that although all the plots shown are linear (range 0-2%) in the cases of samples left to equilibrate for isotherm measurement the curves level out as equilibrium is attained. The gradient of these plots gives the rate of impregnation. Figure 4 shows a plot of the utilisation of TEDA, which was normalised for impregnant loading, against the rate of impregnation. A clear relationship is evident, with the slowest rate resulting in the highest level of utilisation. This observation may be explained if we consider the strong affinity of the carbon for TEDA, which is evident from the isotherm data. At high rates of impregnation, when a large number of TEDA molecules are adsorbing, there may be a tendency for clusters of TEDA to form, leading to a poor distribution and perhaps pore blocking effects. It is also possible that at elevated temperatures the efficiency of diffusion increases to the extent that TEDA is permeating to pores which allow strong adsorption, but offer limited access to subsequent vapours.

Conclusions

The adsorption isotherm of TEDA vapour on activated carbon has been measured. Adsorption predominates in the micropores but also occurs in the mesopore region. The rate of deposition of this impregnant is important in determining impregnant utilisation.

References

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Table 1. Nitrogen porosimetry data

Sample	Precursor	BET SSA (m^2g^{-1})	Total Pore Volume (cm^3g^{-1})	Mesopore Volume (cm^3g^{-1})	Micropore Volume (cm^3g^{-1})
BPL	coal	1154	0.542	0.112	0.430
SSC	nutshell	2098	1.075	0.220	0.855
BAX	wood	1352	1.009	0.579	0.430

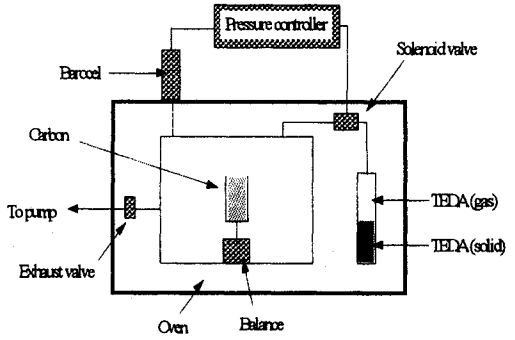

Figure 1. TEDA adsorption apparatus

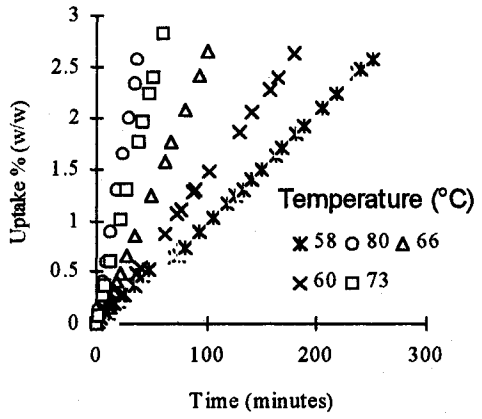

Figure 3. The effect of temperature on rate of impregnation

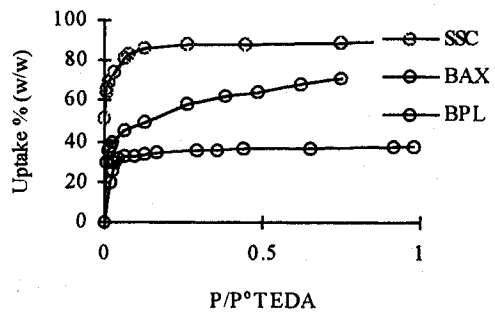

Figure 2. Adsorption isotherms of TEDA on activated carbons at 80°C

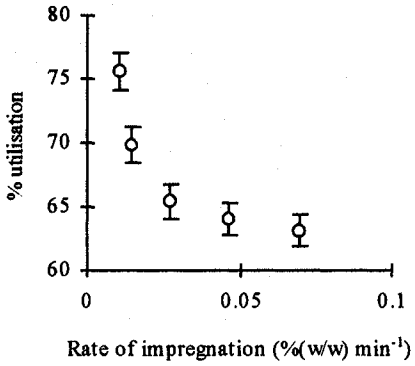

Figure 4. The effect of rate of impregnation on impregnant utilisation