Separation Science and Technology
Publication details, including instructions for authors and subscription information:
http://www.informaworld.com/smpp/title~content=t713708471

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To cite this Article Chang, Ching-Yuan , Tsai, Wen-Tien and Lee, Horng-Chia(1996) 'Desorption Kinetics of A N, N-Dimethylformamide Vapor from Granular Activated Carbon and Hydrophobic Zeolite', Separation Science and Technology, 31: 12, 1675 — 1686

To link to this Article: DOI: 10.1080/01496399608000718

URL: http://dx.doi.org/10.1080/01496399608000718

SEPARATION SCIENCE AND TECHNOLOGY, **31**(12), pp. 1675–1686, 1996

Desorption Kinetics of *N,N*-Dimethylformamide Vapor from Granular Activated Carbon and Hydrophobic Zeolite

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ABSTRACT

Such thermodynamic properties as enthalpy, free energy, and entropy of adsorption have been computed for *N,N*-dimethylformamide (DMF) vapor on two commercial adsorbents: coconut shell Type PCB of activated carbon and Type DAY of hydrophobic zeolite. The computation is based on the Langmuir adsorption isotherms obtained at 293, 303, and 313 K as reported by Tsai et al. The laden adsorbents were regenerated with hot inert nitrogen gas and studied by thermal gravimetric analysis at three different heating rates. The apparent activation energies (E_{des}) of thermal desorption were determined by using the Friedman method. The zeolite DAY has an adsorption potential higher than that of activated carbon PCB as indicated by the more negative value of the adsorption enthalpy of DMF vapor. The average value of E_{des} of zeolite DAY is larger than that of activated carbon PCB.

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INTRODUCTION

Adsorption processes employing granular activated carbon (GAC) have been used in a number of control designs to capture and recover organic compounds from air and water sources (1–4). However, GAC presents several problems (5, 6). Carbon is a flammable material, potentially promoting fire when heated above 393 K (120°C) so that solvents boiling above 423 K (150°C) cannot be efficiently removed. Impurities (e.g., ash) naturally existing in carbon can act as catalysts and promote polymerization or oxidization of some reactive organics during carbon steam regeneration. Hydrophobic zeolite, a new synthetically produced adsorbent, does not have such disadvantages and offers a distinct advancement in solvent separation or recovery. Therefore, GAC and hydrophobic zeolite may be an ideal adsorbent medium in some situations, for the two adsorption technologies are complementary rather than competing approaches (6).

In Taiwan, *N*,*N*-dimethylformamide (DMF) has been recently used as a polar solvent, especially in the production of fibrous polyacrylonitrile and synthetic polyurethane (PU) leather (7). The annual consumption of DMF has exceeded 50,000 metric tons since 1994 (8). This solvent is strongly toxic to the liver after extensive occupational exposure, and it is listed as a hazardous air pollutant or air toxic in Title III of the US Clean Air Act Amendments (CAAA) of 1990 (7). DMF-induced hepatitis has been documented among synthetic PU leather workers as one of the ten most serious occupational diseases in Taiwan during 1983–1991 (9). Because of the toxicity of DMF, the organic vapor should be captured or destroyed by using appropriate control technologies. The adsorption and catalytic incineration of organic vapors containing DMF have been studied previously (7, 10).

Since GAC and hydrophobic zeolite are rather expensive, several thermal methods for regenerating spent adsorbents, including steam regeneration and hot inert gas regeneration, have been developed (1, 11). Direct steam regeneration is the most widely used technique. However, some hydrolyzed hydrocarbons easily react with the carbon itself and/or with steam during regeneration. They are hydrolyzed slightly to form acids and other organics due to the presence of steam and the catalytic action of the impurities (e.g., transition metals in the ash) in the carbon at regeneration temperatures (5, 12). An inert gas (e.g., nitrogen) is usually used instead of steam for the recovery of reactive, water-miscible solvents. This direct recovery technology can avoid the need for add-on units (e.g., decanter/distillation column) to separate solvents from water. DMF, used as a target adsorbate in the present study, has little tendency to undergo hydrolysis to formic acid and dimethylamine (13, 14).

There are many reports about regeneration tests and desorption kinetics of spent GAC (15–19). Suzuki et al. (15) divided the characteristics of thermal regeneration of spent activated carbon into three types according to the kind of adsorbates, and analyzed the rate of thermal decomposition of adsorbates by a first-order equation at a 6 $K{\cdot}min^{-1}$ heating rate. Jonas and Sansone (16) studied the first-order desorption rate constant of the Wheeler desorption equation for spent GAC adsorbed on carbon tetrachloride at different degrees of saturation. Urano et al. (17) studied the regeneration rates of commercial GAC containing adsorbed organic matter under five thermal decomposition conditions by analyzing the thermogravimetric curves at a constant heating rate (7 or 17 $K{\cdot}min^{-1}$). Krebs and Smith (18) studied the desorption kinetics of activated carbon adsorbed on phenol by measuring the weight and rate of weight change in runs at a constant heating rate (6 $K{\cdot}min^{-1}$) and at a constant temperature where the only desorption product was phenol. Schäfer et al. (19) studied the adsorption behavior of halogenated hydrocarbons on activated carbon and the subsequent desorption kinetics of a first-order reaction in a thermogravimetric analyzer under nonisothermal conditions (5 $K{\cdot}min^{-1}$). However, those studies were partial investigations under specific conditions, and the relationships between adsorption thermodynamics and desorption kinetics of DMF on commercial microporous adsorbents are not reported in those studies, particularly for hydrophobic zeolite.

The objective of this study was to obtain the adsorption thermodynamics properties of commercial GAC (coconut shell, Type PCB) and hydrophobic zeolite (Type DAY) from previous Langmuir isotherm data (10). The loaded adsorbents were regenerated with a hot inert nitrogen gas to avoid the slight hydrolysis reaction of DMF, and the thermal gravimetric analysis (TGA) was studied at three different heating rates. The corresponding apparent activation energies of desorption kinetics were thus determined. Further, the relationships between the thermodynamic parameters (e.g., enthalpy) of adsorption and the parameter (e.g., activation energy) of desorption kinetics are also discussed in the present work.

EXPERIMENTAL SECTION

Materials

The adsorbents used in this study were granular activated carbon (coconut shell, Type PCB) and hydrophobic zeolite (Type DAY) provided by Calgon Carbon Co. and Degussa AG Co., respectively. Their physical characterizations have been measured and reported previously (10). DMF, provided by Merck Co., was of 99.9% purity. Its latent heat of vaporization is 42.3 $kJ{\cdot}mol^{-1}$.

Experimental Apparatus and Procedure

The adsorption isotherm data of DMF have been reported previously (10). Therefore, the thermodynamic parameters of adsorption were calculated from the adsorption equilibrium constant obtained from Langmuir's equation by using van't Hoff plots and thermodynamic relationships in the present work.

The desorption experiments were carried out under nonisothermal conditions by employing a thermogravimetric analyzer (CAHN-1000 Recording Electro-Balance), a temperature programmer (Eurotherm 815 S with RS 232 interface), and a data processing unit. The experimental system has been described elsewhere (20). The reactor was made of a dual tube of quartz. The quartz disk (of 2 cm diameter and 1 mm thickness) was hung on the suspension wire (made of platinum, 0.18 mm diameter) of the TGA microbalance and enclosed in the reactor.

The sample (about 50 mg), saturated with DMF adsorbate, was removed in a closed tube from a refrigerator, immediately placed on the disk, and then heated under nitrogen flow at constant heating rates of 2, 5, and 7 $K{\cdot}min^{-1}$ up to about 673 K. The flow rate of nitrogen was 50 $mL{\cdot}min^{-1}$ (STP), which is large enough so that the gas film resistance caused by the heat and mass transfer can be neglected, and low enough so that the effect of flow rate on the measurement of the mass change was negligible (21). The mass losses of the sample and the temperature were recorded simultaneously. The determination of the kinetic parameters (e.g., activation energy) of desorption was carried out by employing the Friedman method (22), described later.

RESULTS AND DISCUSSION

Thermodynamic Parameters of Adsorption

The adsorption isotherms of DMF on activated carbon PCB and zeolite DAY investigated previously in the 293–313 K temperature range may be fitted by Langmuir's equation (10), written as

$$q/q_m = KC/(1 + KC) \tag{1}$$

where q is the adsorption capacity, q_m is the adsorption capacity at monolayer saturation, C is the adsorbate equilibrium concentration, and K is the adsorption equilibrium constant. The constants (q_m and K) involved in Eq. (1) have been obtained and reported previously (10).

The thermodynamic parameters free energy ($\Delta G°$) and enthalpy ($\Delta H°$) of DMF adsorption were calculated from the adsorption equilibrium constant K by using the following relationships (23, 24):

$$\Delta G^\circ = -RT \ln K \tag{2}$$

$$\ln K = -\Delta H^\circ/RT + \text{constant} \tag{3}$$

The values of ΔH° were calculated from the slope of the linear variation of $\ln K$ vs $1/T$. The values of ΔS° were then calculated by using the well-known equation

$$\Delta G^\circ = \Delta H^\circ - T\Delta S^\circ \tag{4}$$

The values of ΔG°, ΔH°, and ΔS° for the DMF adsorption system, computed from Eqs. (2)–(4), are listed in Table 1. It is seen that zeolite DAY has a adsorption potential higher than activated carbon PCB as indicated by the more negative value of ΔH°, i.e., 40.0 vs 37.5 kJ·mol^{-1}. Further, the magnitude of $-\Delta S^\circ$ for zeolite DAY is larger than that for activated carbon PCB. Hence, the adsorption affinity, which is paralleled by an increase in $-\Delta H^\circ$ and $-\Delta S^\circ$ of adsorption, indicates that the adsorbate–adsorbent interactions of DMF–zeolite DAY is slightly stronger than that of DMF–activated carbon PCB.

Analysis of Desorption Kinetics

For the derivation of the relevant kinetic parameters such as activation energy (E_{des}) from thermal desorption in TGA experiments, different mathematical methods have been developed by previous investigators (25, 26). These methods are primarily based on the utilization of classical laws of kinetics. The rate of desorption, dX/dt, can be expressed as a function of the mass loss fraction, X, using the general formulation.

$$dX/dt = k \cdot f(X) \tag{5}$$

TABLE 1
Values of Thermodynamic Parameters for DMF Adsorption on Activated Carbon PCB and Zeolite DAY

Adsorbent	Temperature (K)	ΔG° (kJ·mol^{-1})	ΔH° (kJ·mol^{-1})	ΔS° (J·mol^{-1}·K^{-1})
Activated carbon-PCB	293	−17.23		−69.08
	303	−15.73	−37.47	−71.75
	313	−15.87	(R^2 = 0.873)	−69.10
Zeolite-DAY	293	−16.11		−81.54
	303	−16.44	−40.00	−77.76
	313	−14.45	(R^2 = 0.795)	−81.63

where k is a rate constant and $f(X)$ is some function of the degree of desorption. In thermogravimetry, X can be expressed in terms of initial mass (W_0), instantaneous mass (W), and final residual mass (W_f) of the sample by

$$X = (W_0 - W)/(W_0 - W_f) \qquad (6)$$

In Eq. (5) the rate constant k depends on the temperature (T) according to the Arrhenius relationship

$$k = A \exp[-E_{des}/(RT)] \qquad (7)$$

where A is a preexponential (frequency) factor, R is the gas constant, and E_{des} is the apparent activation energy of the desorption process in the nonreadsorption case.

In the present work the residue (with mass W) after desorption is expressed on a normalized basis, that is, residual mass fraction $M = (W - W_f)/(W_0 - W_f) = (1 - X)$. The variations of M of activated carbon PCB and zeolite DAY adsorbed on DMF with desorption temperature (T) at heating rates (β) of 2, 5, and 7 K·min^{-1} are shown in Figs. 1 and 2, respectively. Obviously, there were gradual mass changes in the temperature range of the experiments for all three heating rates. The mass changes of activated carbon PCB and zeolite DAY proceeded at a very slow rate

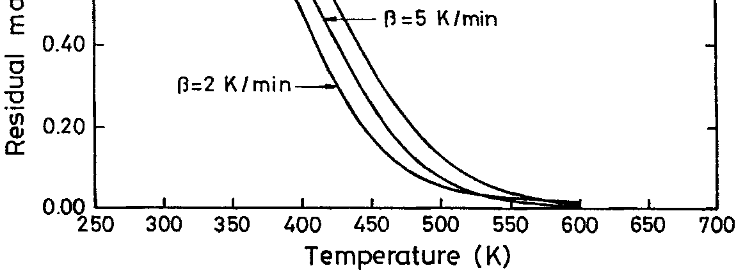

FIG. 1 TGA desorption curves of activated carbon PCB with adsorbed DMF under heating rates (β) of 2, 5, and 7 K·min^{-1}.

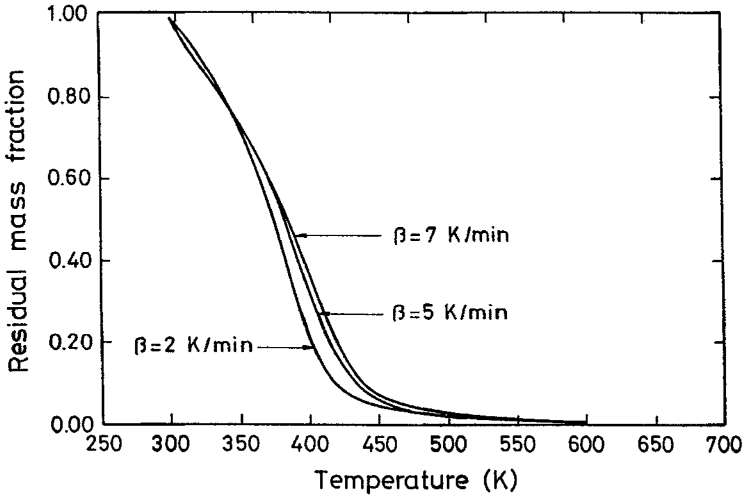

FIG. 2 TGA desorption curves of zeolite DAY with adsorbed DMF under heating rates (β) of 2, 5, and 7 K·min^{-1}.

as *M* decreased to about 0.05, where the corresponding temperatures reached about 525 and 450 K, respectively. The results indicated that the complete desorption of zeolite DAY was easier than that of activated carbon PCB. This seems to be correlated with the micropore volumes obtained from adsorption isotherms such as the Dubinin–Radushkevich equation (10), i.e., 0.214 cm^3·g^{-1} of zeolite DAY vs 0.415 cm^3·g^{-1} of activated carbon PCB. In addition, as a lower heating rate results in a longer retention time of the adsorbent at a given temperature, a higher heating rate shifts the residual mass curve to the right to a higher temperature range as shown in Figs. 1 and 2.

Variations of the instantaneous mass loss rates, dX/dt, of desorption of activated carbon PCB and zeolite DAY adsorbed on DMF with the temperature at heating rates (β) of 2, 5, and 7 K·min^{-1} are shown in Figs. 3 and 4, respectively. The maximum peak rates of activated carbon PCB are lower than those of zeolite DAY and occurred at different heating rates. Further, the distributions of the mass loss rate of activated carbon PCB are wider than those of zeolite DAY, which is consistent with the pore size distributions of amorphous activated carbon and crystalline zeolite which are very wide and homogeneous, respectively.

The Friedman method was employed to calculate the apparent activation energy (E_{des}) of desorption in the present study because this method

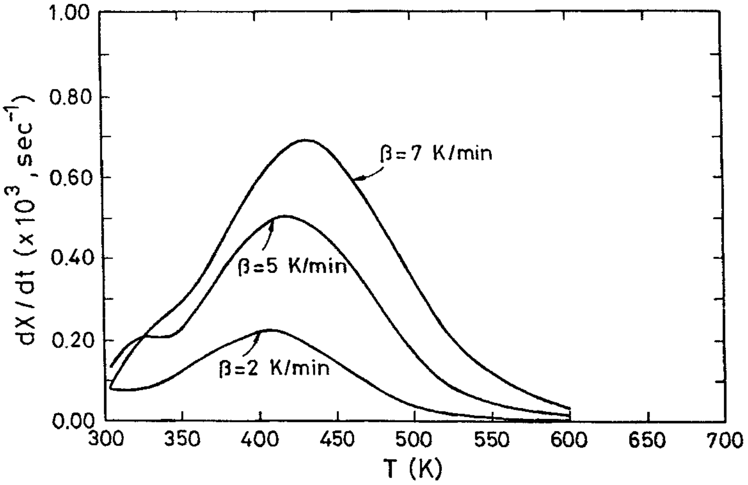

FIG. 3 Desorption rate (*dX/dt*) curves of activated carbon PCB with adsorbed DMF with temperature (*T*) under heating rates (β) of 2, 5, and 7 K·min^{-1}.

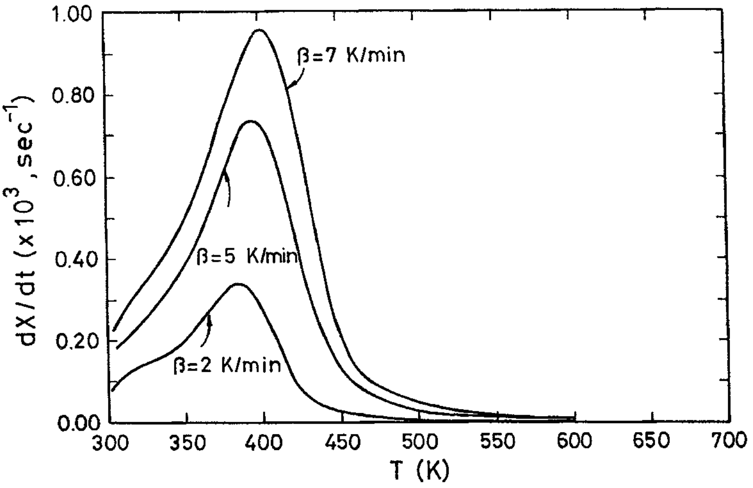

FIG. 4 *dX/dt* curves of zeolite DAY with adsorbed DMF with *T* under heating rates (β) of 2, 5, and 7 K·min^{-1}.

produces less error for the activation energy (25). Equations (5) and (7) were combined and rearranged to give the following logarithmic form:

$$\ln(dX/dt) = \ln[Af(X)] - (E_{des}/R)(1/T) \tag{8}$$

Therefore, a plot of $\ln(dX/dt)$ vs $1/T$ was made under constant X, where the values of T were taken from the curves of Figs. 1 or 2 for three different heating rates. The plot should determine a straight line with a slope of $-E_{des}/R$, using the least-squares method, and E_{des} was thus obtained at each constant value of X.

The apparent activation energies of desorption of activated carbon PCB and zeolite DAY adsorbed on DMF under different conversions (X = 0.3–0.8) at heating rates (β) of 2, 5 and 7 K·min^{-1} are illustrated in Figs. 5 and 6, respectively. The results indicate that E_{des} of activated carbon PCB and zeolite DAY has values in the range of about 60–90 kJ·mol^{-1} and 80–180 kJ·mol^{-1}, respectively. Obviously, the average value of E_{des} of activated carbon PCB is lower than that of zeolite DAY, i.e., 75.1 vs 109.5 kJ·mol^{-1}. This is also consistent with the adsorption enthapies shown in Table 1. Further, it is seen that the heats of adsorption (Q, $Q = -\Delta H°$) are lower than the apparent activation energies of desorption. The difference ($= E_{des} - Q$) is equivalent to the apparent activation energy of the adsorption process in cases of physical adsorption (27, 28).

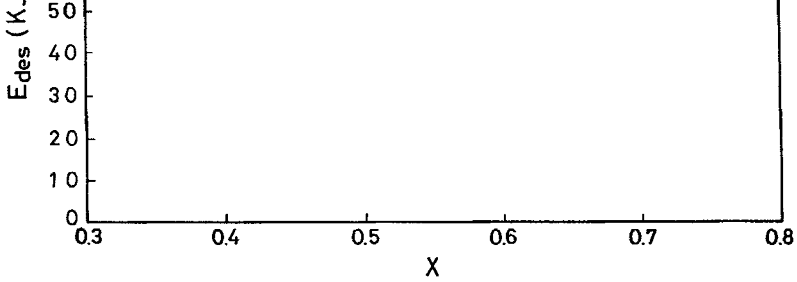

FIG. 5 Apparent activation energies (E_{des}) of thermal desorption at different mass loss fraction (X) for activated carbon PCB with adsorbed DMF.

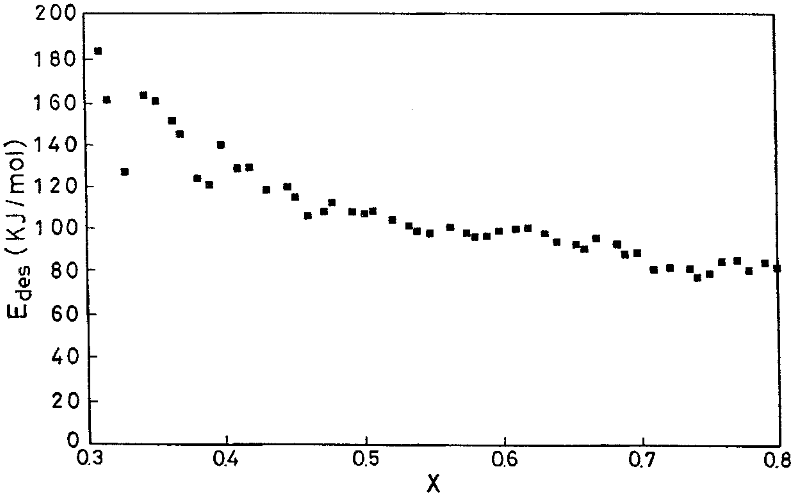

FIG. 6 E_{des} of thermal desorption at different X for zeolite DAY with adsorbed DMF.

Therefore, the apparent activation energy of adsorption of activated carbon PCB is lower than that of zeolite DAY. The micropore size distribution of an adsorbent may be closely correlated with the adsorption enthalpy and apparent activation energies of adsorption and desorption (29).

CONCLUSIONS

The equilibrium adsorption and thermal desorption of DMF on granular activated carbon PCB and hydrophobic zeolite DAY were investigated. Such thermodynamic quantities as $\Delta G°$, $\Delta H°$, and $\Delta S°$ have been computed from the adsorption equilibrium constant of the Langmuir isotherm. It was found that zeolite DAY has an adsorption potential with DMF higher than activated carbon PCB, which is consistent with the apparent activation energy of desorption obtained from TGA desorption results. A simple desorption kinetics seems to be applicable for the desorption of DMF from activated carbon PCB as well as zeolite DAY with different microporous structures.

ACKNOWLEDGMENT

This work was supported by the National Science Council, Taiwan, Republic of China, under Contracts NSC 83-0410-E-002-028 and NSC 84-2211-E-002-058.

NOMENCLATURE

A	preexponential (frequency) factor (s^{-1})
C	adsorbate (i.e., DMF) equilibrium concentration ($mol \cdot m^{-3}$)
E_{des}	apparent activation energy of thermal desorption ($kJ \cdot mol^{-1}$)
ΔG°	free energy of adsorption ($kJ \cdot mol^{-1}$)
ΔH°	enthalpy of adsorption ($kJ \cdot mol^{-1}$)
K	adsorption equilibrium constant of Langmuir isotherm ($m^3 \cdot mol^{-1}$)
M	residual mass fraction, $(W - W_f)/(W_0 - W_f)$ (—)
Q	adsorption heat ($kJ \cdot mol^{-1}$)
q	adsorption capacity ($mol \cdot kg^{-1}$)
q_m	adsorption capacity at monolayer saturation ($mol \cdot kg^{-1}$)
R	gas constant ($= 8.314\ J \cdot mol^{-1} \cdot K^{-1}$)
ΔS°	entropy of adsorption ($J \cdot mol^{-1} \cdot K^{-1}$)
T	temperature (K)
t	time (seconds)
W	total mass of residual sample at time t (mg)
W_0	total mass of initial sample (mg)
W_f	total mass of final residual sample (mg)
X	mass loss fraction, $1 - M$ (—)
β	heating rate ($K \cdot min^{-1}$)

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Received by editor September 18, 1995