

Drying of an Activated Carbon Column after Steam Regeneration*

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Experiments of the adsorption cycles, which include adsorption, steam desorption, and drying/cooling, are conducted using a well instrumented pilot-scale apparatus with toluene as the sorptive. Special attention can be paid to the temperature profiles and the changes in water and toluene loading during drying since there is the possibility to take samples from inside the column. Water isotherms were gravimetrically determined and correlated with the association theory of Talu and Meunier. The mathematical description of the air drying of the packed bed with activated carbon is based on an equation system with coupled heat and mass transfer between the bed and the fluid. The first calculations show good agreement with experimental results.

1 Introduction

Activated carbon (AC) adsorbers are used to recover dilute solvent vapors in the air from process streams before the air is vented to the atmosphere. Steam is usually used in the regeneration of the AC because of its advantages [1–3]. After steaming (steam regeneration), the carbon becomes wet and hot, which is not suitable for the next adsorption. Thus, the so-called drying (and perhaps cooling) process is very important, in order to retrieve the adsorption ability of the AC [3].

The drying processes are commonly used industrially, yet little fundamental research of either a theoretical or experimental nature has been performed to improve our understanding of the process. Improved understanding can be expected to lead to improved design methods and optimization of existing processes. The prediction of the adsorption capacity also relies on the state of the adsorber after drying [3]. As an *in situ* process, drying can be processed with hot purging gases (e.g., inert media: air, nitrogen, flue gas) or ambient air. An extra air cooling might be needed to cool down the adsorber after a hot purge. Drying with ambient air (without a heat source) is sometimes too weak because the energy supply required for the desorption of water is lacking, especially when the humidity of the air is high.

From an engineering viewpoint, the advantages and disadvantages of water loading on AC are discussed elsewhere [4]. Naujokas [5] regards water on AC as important for preventing bed fires and as a medium for a dissipation of the adsorption heat of a solvent. Parmele et al. [6] and Mastroianni and Rochelle [7] show that cooling and drying of an adsorber after steam regeneration are necessary for good performance (reduction of corrosion and prevention of solvent degradation). Scamehorn [8] studied the drying step with a simple model but concluded that no simple mechanism can describe the drying process. He found also that drying is

slow without heating. Ustinov [9] presented a model to optimize the energy consumption but without experimental confirmation. Schweiger [4] showed in his simulation results with a simple equilibrium drying model that large accumulations of water result when drying is unsatisfactory. Erpelding et al. [9] and Schweiger [4] discussed the effect of residual water loadings on the following adsorption cycles of the solvent. Börger's [10] experiments show that the direction of airflow also plays an important role in hot purging.

In this work, the jointed drying and cooling process (D/C) with ambient air directly after steaming is experimentally tested. This is of great advantage, when combined with an optimal steaming process. A mathematical model, which can simulate the bed temperature profiles and water loading during D/C, is proposed.

2 Experimental

D/C experiments are performed using a well instrumented pilot-scale apparatus with two columns which allow continuous adsorption cycles (Fig. 1). The standard adsorption processes and steam regeneration are included in the experiments, while the process is performed in a cycle, consisting of—adsorption, steaming, and D/C. Special attention can be paid to the temperature profiles and the changes in the water and toluene loading during D/C since there is the possibility to take samples from inside of the apparatus. In the experiments the parameters have been varied as shown in Tab. 1.

The adsorber column diameter is 500 mm, the height is 1500 mm, and it is filled with 96.5 kg activated carbon (Sorbonorit 4). Toluene is added to the air as the model substance. Some important data of Sorbonorit 4 are shown in Tab. 2.

The bed temperatures and gas concentrations in the column are recorded at 5 different axial positions. Carbon samples could be taken at 7 axial positions, in order to obtain the toluene and water loading profiles. The total loading of water and toluene was estimated gravimetrically by desorption of the samples under 150 °C and 0.5 mbar. After extraction of the carbon sample in a solvent mixture (1:1 of methanol:methylene chloride), the water loading was measured with Karl-Fischer titration.

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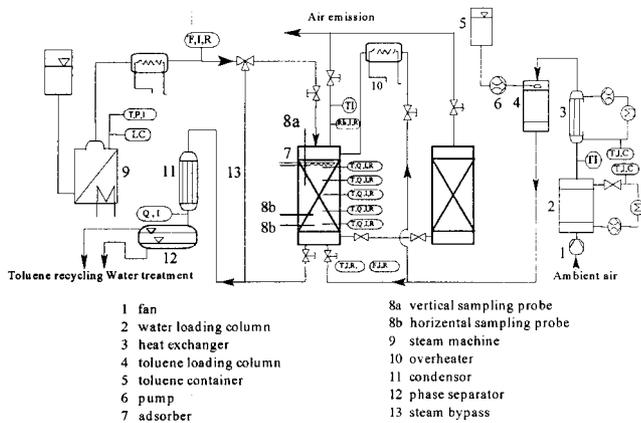


Figure 1. Flow schema of the pilot plant adsorption unit.

Table 1. Operation conditions (1 bar).

Steaming (standard)	
Steam temperature	100, 120, 140 °C
Empty tube velocity of steam	0.2 m/s
Steam used	0.85 kg/(kg activated carbon)
Flow direction	countercurrent to adsorption
D/C	
Air temperature	20 – 30 °C (ambient)
Flowrate	100, 200, 400 m ³ /h
Air humidity	30, 50, 70 %
Adsorption (standard)	
Air temperature	20 – 30 °C
Empty tube velocity of air	0.3 m/s
Air humidity	35 %

3 Mathematical Model

As shown in the experiments, in the D/C process the bed temperature and water loading change very fast and markedly. The description and modeling of the D/C process is rather tedious, since one has to consider coupled mass and energy balances and mass transfer of water-solvent mixtures in porous systems. When the air passes through the AC bed, which is just regenerated with hot steam, the moisture of the AC will be taken off into the air and the bed temperature will sharply decrease because of the desorption enthalpy of water and of convective heat transfer between bed and fluid. An analysis of the basic phenomena [3] leads to the following model assumptions:

- The shrinkage of AC particles during D/C is negligible.
- Both phases – the gas phase and solid phase – can be considered as homogeneous at each section of the bed.
- Heat transfer from the solid to the gas phase is through forced convection.
- Desorption heat of water (and toluene) is taken from the solid phase.
- The transport of the sorptive from AC to bulk flow is through convection.
- There is no radial heat transfer.
- The axial dispersion of heat and mass is so small that it can be neglected.

Table 2. Substance properties (Sorbonorit 4)

Shape	cylindrical granular
Length	5 – 13 mm
Particle diameter	3.8 mm
Porosity between particles	0.4 m ³ /m ³
Packing density	410 kg/m ³
BET - surface	1200 – 1300 m ² /g

- The pressure in the column is uniform because of small flow velocity (0.15–0.45 m/s).
- The volume of the adsorbate is neglected and the heat capacity of steam and carbon is constant.

When considering a bed element of unit cross-sectional area between position z and $z+dz$, the mass balances of the gas phase comprises normally three terms: the changes with time, axial bulk flow, and desorption from AC.

The water balance in the gas phase is therefore given by¹⁾:

$$\varepsilon \frac{\partial C_w}{\partial t} + \frac{\partial(uC_w)}{\partial z} + (1-\varepsilon) \rho_s \frac{\partial X_w}{\partial t} = 0 \quad (1)$$

Similar for the toluene mass balance:

$$\varepsilon \frac{\partial C_t}{\partial t} + \frac{\partial(uC_t)}{\partial z} + (1-\varepsilon) \rho_s \frac{\partial X_t}{\partial t} = 0 \quad (2)$$

Air is considered as inert for adsorption; the mass balance for air can be written:

$$\varepsilon \frac{\partial C_a}{\partial t} + \frac{\partial(uC_a)}{\partial z} = 0 \quad (3)$$

Together with the closing condition, the total mass balance reads:

$$\rho_g = C_t + C_w + C_a \quad (4)$$

Based on assumptions above, the energy balance of wet air in the element is

$$\frac{\partial(\rho_g C_{pg} T_g)}{\partial t} + \frac{\partial(u \rho_g C_{pg} T_g)}{\partial z} = h_s (T_s - T_g) \quad (5)$$

where the first term denotes temperature change after time, the second the energy change because of bulk flow, and the third the heat transfer between the two phases. The components properties are obtained from the linear mixing rule.

As stated earlier, it is assumed that water and toluene desorb from the carbon when receiving heat from the bed. At the same time, convective heat and mass transfer occurs between the two phases at the interface. For the condensed phase, in which carbon, water, and toluene are involved, the energy balance is thus given by:

$$(1-\varepsilon) \rho_s (C_{ps} + X_w C_{ps,w} + X_t C_{ps,t}) \frac{\partial T_s}{\partial t} = h_s (T_g - T_s) + (1-\varepsilon) \rho_s (H_{ad,t} \frac{\partial X_t}{\partial t} + H_{ad,w} \frac{\partial X_w}{\partial t}) \quad (6)$$

1) List of symbols at the end of the paper.

where the first term expresses the energy change due to temperature change, the third the enthalpy of desorption, and the middle term the heat transfer between phases which is the same as in Eq. (5).

3.1 Kinetics

In general, the intraparticle mass transfer includes three steps: convective transfer between the bed and the fluid, pore diffusion, and surface diffusion. During D/C the intraparticle conditions change dramatically and the transfer mechanisms are very tedious to evaluate, as they can be influenced by loadings, temperature, pore size distribution, and adsorptive properties. It is convenient to represent the mass transfer rate in terms of an effective overall transfer coefficient k as a linear driving force equation:

$$\frac{\partial X_i}{\partial t} = k_i(C_i - C_{eq,i}) \quad (7)$$

where i represents water or toluene.

3.2 Isotherms

It is well known that the water-carbon isotherm has a characteristic S-shape and includes a hysteresis effect, in which capillary condensation plays a significant role [11]. The co-adsorption mechanism of solvent and water on AC is more complicated than water or solvent alone due to the interaction between the two immiscible substances and the occupancy distribution into the AC heterogeneous pores [12,13]. Considering the fact that there is little change of toluene loading during D/C, the independent isotherms of water and toluene are adapted in this work in order to make the simulation feasible. A gravimetric analysis gave experimental data of isotherms for water adsorption and desorption. Recently, Talu and Meunier [14] proposed a molecule association theory to describe the water isotherms on AC:

$$P_w = H_w[\varphi_w/(1+K_{0w}\varphi_w)]\exp(\varphi_w/N_m) \quad (8)$$

where P_w is the partial pressure of water vapor and the transitional parameters are as follows:

$$\varphi_w = \left(-1 + \sqrt{1 + 4K_{0w}\xi_w}\right) / 2K_{0w} \quad (9)$$

$$\xi_w = N_m N_w / (N_m - N_w) \quad (10)$$

where N_w is the amount adsorbed while N_m is the maximal ability of water adsorption on AC. The constants in Eq. (8) can be given in the following form:

$$K_{0w} = \exp[K_0 - (K_1/T_s)] \quad (11)$$

$$H_w = \exp[H_0 - (H_1/T_s)] \quad (12)$$

The loading of water on AC and the gas phase concentration can be changed into the following forms:

$$X_w = 18 \times 10^{-3} N_w \quad (13)$$

$$C_{eq,w} = 2.18 \times P_w/T_g \quad (14)$$

The constants in Eqs. (8–12) for water on AC (Sorbonorit 4) are estimated to be $N_m = 20.95$, $K_0 = 4.472$, $K_1 = 990$, $H_0 = 21.85$, and $H_1 = 5990$. The data will be shown in the next part of this paper.

Bart et al. [15] and Ning [16] presented the experimental results about toluene adsorption isotherms on the same AC as this work, which can be given in a Langmuir form:

$$X_t = \frac{0.36 K_{0t} C_{eq,t}}{1 + K_{0t} C_{eq,t}} \quad (15)$$

where the temperature coefficient is

$$K_{0t} = 97.6 \exp(1195.6/T_s) \quad (16)$$

The equation system, given above with coupled heat and mass transfer between bed and fluid, is discretized and then solved with a commercial dynamic solver (SpeedUp[®]) with a central difference approximation. The initial conditions are taken as a physical equilibrium of the loaded AC phase with the steam surrounding.

4 Results and Discussion

4.1 Isotherms

Equilibrium data of water adsorption on AC (Sorbonorit 4) were obtained by a gravimetric approach at three temperatures: 30, 40, and 50 °C. The water isotherms on AC show always S-shaped lines classified as type 5 (BET classification) with an obvious hysteresis (as shown in Fig. 2).

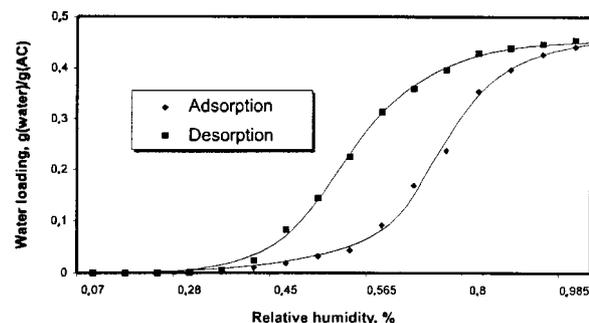


Figure 2. Water isotherm on Sorbonorit 4 at 30 °C.

The water desorption isotherms are of great concern in this work, because water will desorb during D/C. Fig. 3 displays the experimental data and the regression results with Talu and Meunier's [14] associating theory, which consider that water molecules 'like' each other more than they 'like' the carbon surface. However, a high interaction between the solid and guest molecules can result in a type 1 isotherm, which happens for solvent adsorption. The curves in Fig. 3 are related to Eqs. (8–14) described earlier.

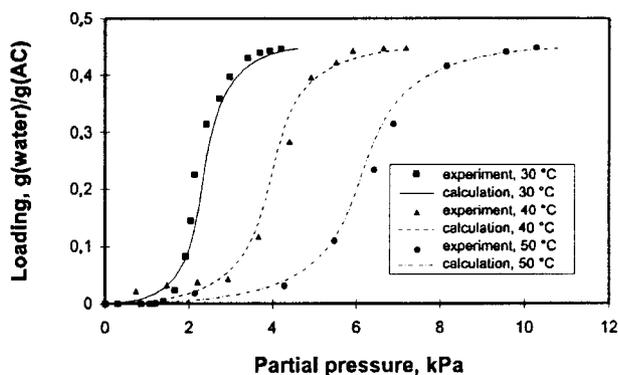


Figure 3. Desorption isotherms of water on Sorbonorit 4.

4.2 Combined Drying and Cooling Process [D/C]

During drying, the AC bed is purged with ambient air. Drying is fast at the beginning, when the bed is still warm, and it is slow after the bed has been cooled down. During the fast drying, heat stored in the bed provides energy to desorb water. The stored energy is limited and is quickly depleted. During the slow drying, the AC bed is at about ambient temperature, and the energy is only provided by the convective air flow. Fig. 4 displays the bed temperature profiles during D/C in a typical experiment, and Fig. 5 provides the changes of water loading on bed. It can also be seen that higher air humidity results in higher residual water loadings. In a very moist environment, for example 50% relative humidity [4,9], D/C will not work, as the water loading cannot be effectively removed and will also be further accumulated even during the adsorption process.

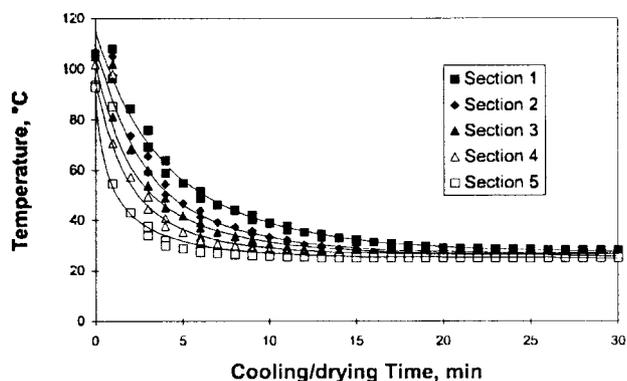


Figure 4. Bed temperature profiles during D/C (air flowrate: 200 m³/h; temperature: 24 °C; relative humidity: 30%)

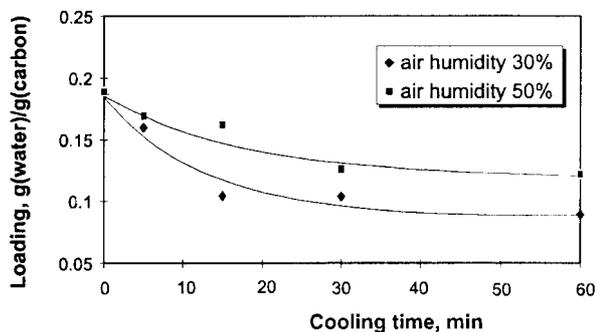


Figure 5. Mean water loading during D/C (air flowrate: 200 m³/h; temperature: 30 °C).

The experiments show that toluene loading changes very little during D/C, because the solvent is more tightly adsorbed on AC than is water (Fig. 6). When the D/C time is very long, which is not usual in practice, the toluene loading will also be slightly shifted along the air flow direction.

Water loading will change dramatically and relies on the flow direction of the ambient air (Fig. 7). Upward air flow, which is in the same direction as adsorption but converse to the steaming, is recommended [17]. A very low water loading is achieved at the bottom of the column, which favors the next adsorption, since the moisture in the upper part of the column will be purged during the adsorption without extra cost. However, drying with hot purging gas in the same direction as steam flow may increase the adsorption performance of regenerated adsorbers [10].

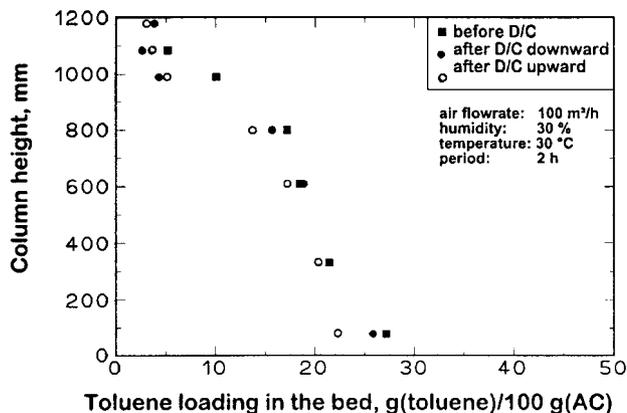


Figure 6. Effect of D/C on toluene loading [2].

At the beginning of D/C, the bed is very hot, so that a high emission concentration of solvent will occur, which depends upon the rest loading of solvent, which is also the desorption depth. To reach a high removal efficiency, it is necessary to control these emissions, for example by recycling the exhaust to the bed currently in the adsorption stage with additional costs [6]. As a result of this work, a suitable arrangement of the air flow direction also resulted in a satisfactory outlet concentration of the solvent even at the beginning of purging. In Fig. 8, this is achieved with an upward air flow, while the rest loading of solvent is much lower at the top of column (Fig. 6).

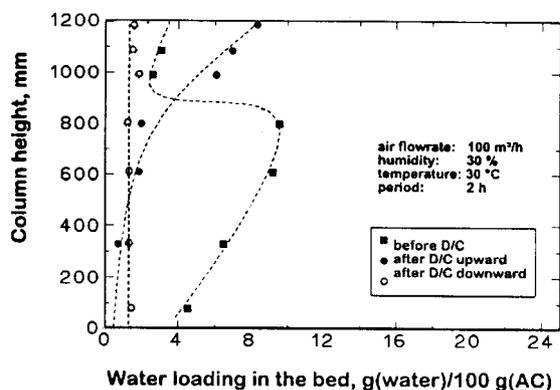


Figure 7. Water loading change in the bed [2].

The temperature profiles in the bed at different levels are shown in Fig. 9. The cooling is very fast at the beginning when the bed is still hot. It is shown that the model proposed in this paper can predict well the bed temperature profiles during D/C. In the simulation, the mass and heat transfer coefficients are correlated with the experimental data in a small scale adsorber [18], which have been checked with the data in literature [2,19]. In the case shown in Fig. 9, the heat transfer coefficient is $h = 95 \text{ W}/(\text{m}^2\cdot\text{K})$ and the mass transfer coefficients for both substances are $k_{\text{water}} = 0.1 \text{ m}^3/(\text{kg}\cdot\text{s})$; $k_{\text{toluene}} = 0.048 \text{ m}^3/(\text{kg}\cdot\text{s})$, respectively.

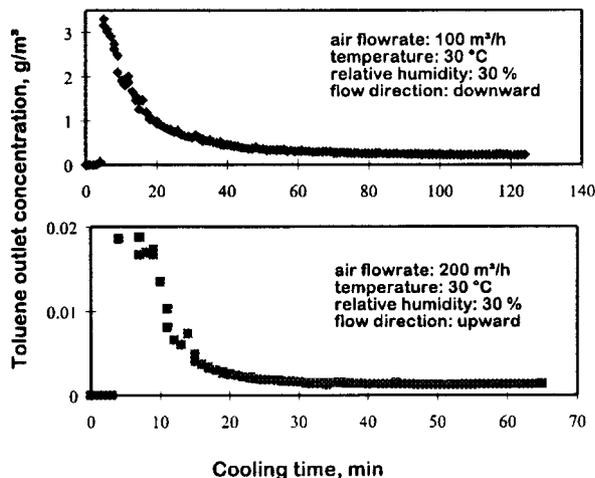


Figure 8. Influence of air flow direction on emission concentrations.

5 Conclusions

The adsorption cycles with toluene as the sorptive in an air stream have been studied in a pilot-scale activated carbon (AC) column. Special attention has been paid to the possibility of combining the drying and cooling process (D/C) with ambient air directly after steam regeneration. The results show that ambient air is a suitable medium to dry the column and to cool it down, when air is not too humid (relative humidity $< 50\%$), otherwise a hot purging gas is necessary. At the beginning of D/C, the drying is fast, while the adsorber is

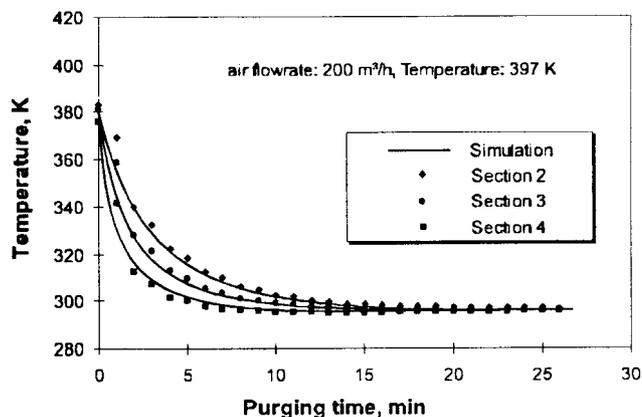


Figure 9. Comparisons of calculated and experimental temperature profiles during cooling at different column height.

still warm. The temperature of the adsorber decreases very fast because of energy consumed during the desorption of water (and toluene). The ambient air should be introduced into the column in the same flow direction as adsorption flow, but reverse to the regeneration steam, in order to maintain the emission limit during D/C. The water isotherms on AC, which show a significant hysteresis, are measured gravimetrically and correlated with Talu and Meunier's association theory before being introduced in the process simulation. A model with coupled heat and mass transfer is proposed to describe the multicomponent, non-equilibrium, non-isothermal D/C process. The simulations showed good agreement with the experimental data.

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Symbols used

C	[mol/m ³]	gas phase concentration
C _p	[kJ/(mol·K)]	heat capacity
H	[-]	coefficient
H _{ad}	[kJ/mol]	adsorption enthalpy
h	[kJ/(m ² ·K)]	heat transfer coefficient
K	[-]	coefficient
k	[m ³ /(kg·s)]	mass transfer coefficient
N	[mol/kg]	loading
N _m	[mol/kg]	maximal loading
P	[Pa]	partial pressure
s	[m ² /m ³]	surface of activated carbon
T	[K]	temperature
t	[s]	time
u	[m/s]	empty tube velocity

X	[mol/kg]	loading
y	[-]	mole fraction
z	[m]	axial distance

Greek symbols

ε	[-]	porosity
φ	[-]	coefficient
ρ	[mol/m ³ or kg/m ³]	density
ξ	[-]	coefficient

Subscripts

0	subscript for constant
1	subscript for constant
a	air
eq	equilibrium
g	gas phase
i	water/toluene
s	solid phase (carbon)
t	toluene
w	water

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