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REMOVAL AND RECOVERY OF VOLATILE ORGANIC COMPOUNDS (VOCS) FROM WASTE AIR STREAMS IN THERMAL SWING ADSORPTION (TSA) SYSTEM WITH CLOSED-LOOP REGENERATION OF ADSORBENT

The cyclic thermal swing adsorption (TSA) process for volatile organic compounds recovery from the waste air is studied theoretically and experimentally. The TSA cycle is operated in three steps: an adsorption step with cold feed, a desorption step with hot inert gas and a cooling step with cold inert gas. The desorption and cooling are affected by a nitrogen circulating through a heater, an adsorber and a condenser. Toluene and isopropanol are chosen as the volatile organic compounds. Activated carbon Sorbonorit 4 is used as an adsorbent.

1. INTRODUCTION

Volatile organic compounds (VOCs) are the most common pollutants emitted by the chemical process industries [1]. For the removal and recovery of VOCs from the gaseous streams, the cyclic thermal swing adsorption (TSA) processes are widely used [2], [3]. A typical TSA system consists of two adsorption columns with fixed bed of adsorbent. The bed temperature is subject to cyclic variations. While the adsorption process takes place in one column, the bed in the other one is regenerated. During desorption, which is the first step of regeneration, hot purge gas, which can be a slipstream of the purified gas or another inert gas, flows through the bed. The adsorbate concentration in the purge gas is much higher than in the feed gas. This concentrated stream can be directed to an incinerator. It is also possible to recover the adsorbate by condensing it in the purge gas stream. After completion of the desorption step, the bed is cooled. The desorption and cooling steps are carried out using the closed-loop or open-flow methods.

Although we can found many theoretical and experimental works on cyclic TSA processes, there is little description of TSA system with closed-loop adsorbent regen-

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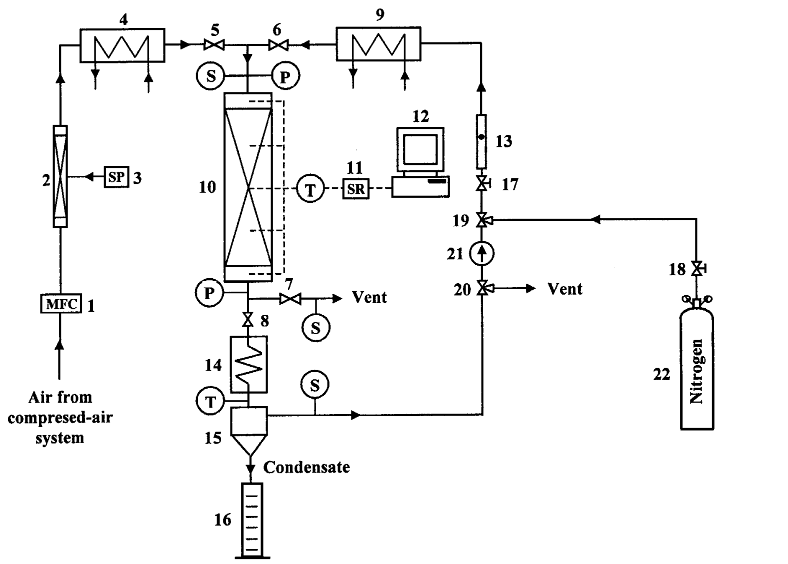
eration [4]. This paper presents the results of the experiments and the modelling of VOCs recovery from waste air in a cyclic TSA system with fixed bed of activated carbon. The TSA cycle is carried out in three steps: an adsorption step of organic compound in cold feed (waste air), a cocurrent desorption step with hot inert gas (nitrogen) and a cocurrent cooling step with cold inert gas (nitrogen). The closed-loop method is used to enhance the efficiency of VOCs recovery. During the desorption and cooling steps nitrogen is circulating through the heater, the adsorber and the condenser. In the condenser, organic compound is partially condensed. In both desorption and cooling steps, the inlet and outlet gas concentrations depend on each other. The results obtained for closed-loop regeneration are compared with the results obtained for the open-flow one. Toluene and isopropanol are chosen as the volatile organic compounds. Activated carbon Sorbonorit 4 is used as an adsorbent.

2. MATHEMATICAL MODEL

Mathematical model of the TSA process is used to simulate temperature and concentration data for adsorption, desorption and cooling steps. The following main assumptions are made: ideal gas behaviour, axially dispersed plug flow, negligible radial temperature and concentration gradients. The model consists of partial differential equations (PDEs) for mass and energy balances within the packed bed. The mass flux into the solid particles of adsorbent is represented by a linear driving force (LDF) model. The set of PDEs is solved using the numerical method of lines (NMOL). A spatial discretization is performed using the second-order central differentiating, and the PDEs are reduced to a set of ordinary differential equations (ODEs). The resulting set of ODEs is solved using the FORTRAN subroutine DIVPAG of the International Mathematical and Statistical Library (IMSL). The model as well as the numerical approach have been described in detail in [4]. Adsorption equilibria for the toluene-activated carbon and isopropanol-activated carbon systems are described by the Dubinin–Astakhov equation. The values of the parameters of this equation are given in [5].

3. EXPERIMENTAL SYSTEM

A bench scale fixed bed adsorption unit is used for the experimental study. A flow diagram of the equipment is shown in figure 1. Air is used as a carrier gas in the adsorption step, while nitrogen is used as a purge gas in the desorption and cooling steps. The adsorption column made of stainless steel is 1.0 m long with 0.8 m packed section, and 0.048 m inside diameter. To minimize heat losses, the column is insulated with about 0.04 m thick layer of fiberglass. For the recovery of organic compounds a condensation unit is installed. For the temperature measurement, three J-type thermocouples are located in the bed, 0.2, 0.4 and 0.6 m from the top of the packed section. Moreover, two thermocouples are installed above and below the adsorbent bed, and one thermocouple is attached to the condenser to measure the temperature of effluent stream. The ambient temperature is also monitored. The thermocouples are connected to a data logger (Smart Reader Plus 6, ACR Systems Inc.)



1 Mass flow controller	**11** Data acquisition modules	**19, 20** Three-way valves
2 Static mixer	**12** Personal computer	**21** Diaphragm pump
3 Syringe pump	**13** Rotameter	**22** Nitrogen gas cylinder
4 Heat exchanger	**14** Condenser	**P** Pressure measurement
5-8 Plug valves	**15** Separator	**S** Sampling point
9 Heater	**16** Measuring cylinder	**T** Temperature measurement
10 Adsorption column	**17,18** Regulation valves	

Fig. 1. Schematic diagram of the experimental setup

controlled by a personal computer. The air flow rate during adsorption step is measured and controlled by a mass flow controller, type GFC 371 (Aalborg Instruments & Controls, Inc.). The adsorbate is introduced into the system using a syringe pump (KD Scientific). During the adsorption step the inlet and the effluent concentrations are monitored by the gas chromatograph. After completion of adsorption the regeneration conditions are established. During closed-loop regeneration, for the removal of air from the column, the adsorption system is first purged with small volume of cold nitrogen. Then the heater and the diaphragm pump are switched on. During the desorption step the adsorption column inlet is connected to the column outlet. Nitrogen is circulating through the heater, the adsorber and the condenser, where adsorbate is partially condensed. The flow rate of nitrogen is indicated by a rotameter. The volume of liquid organic compound recovered in a condenser is measured periodically to check the material balance and to determine the adsorbate concentration in the bed effluent. When the desorption is finished, the heater is switched off and cooling step begins. The direction of gas flow during regeneration (desorption and cooling) is co-current to the direction of gas flow during adsorption.

During open-flow regeneration nitrogen is fed into the adsorption column from gas cylinder through three-way valve (19). The adsorber effluent is cooled and fed to a separator. Gas leaving the separator is discharged, through three-way valve (20), to the ventilation duct.

4. RESULTS OF MODELLING AND EXPERIMENTS

The computer simulation results are used to study the effect of purge gas and condenser temperatures on the condensation efficiency of organic compounds. The cyclic steady-state cycles are obtained under various conditions by a cyclic iteration method. The complete cycles are run until the periodic state is achieved. The adsorption step is continued until breakthrough occurs. The breakthrough concentration is taken as 5% of the adsorption feed concentration. The desorption step is terminated when the outlet temperature exceeds 95% of the inlet temperature. Cooling time depends mainly on the required final outlet temperature. In this study, the value of 300 K is assumed. The final concentration and temperature profile in the adsorbent bed for each step defines the initial conditions for the next step.

It is assumed that the condition for a periodic state is satisfied when the amount of adsorbate removed from the bed during regeneration is equal to the amount that is accumulated in the bed during the adsorption step. The modelling results are compared with the experimental data to examine the feasibility of the model.

The results of the analysis performed indicate that the cyclic steady-state in the adsorption column is established after the second cycle.

The examples of the simulation and experimental results for the recovery of toluene and isopropanol, for the periodic state, are shown in figures 2 and 3. The effect of the purge gas and condenser temperatures (T_{o} and T_{cond}) during closed-loop desorption on the mass of the toluene collected in the condenser (m_{cond}) is shown in figure 2. The inlet temperature and toluene concentration during the adsorption step are 293 K and $3.69 \cdot 10^{-3}$ mol/mol (mole ratio), respectively. The system pressure equals 103.6 kPa. The superficial gas flow rate is the same for desorption and cooling step and equals 9.9 mol/(m^{2}s), while for adsorption step it reaches 13.1 mol/(m^{2}s). The mass of the toluene collected in the condenser corresponds to the amount of the toluene remaining in the bed, and is practically equal to the useful capacity of the adsorbent bed. The mass of the toluene recovered in the condenser decreases with the increase in the condenser temperature, whereas it increases with the increase of the gas temperature at the inlet to the bed.

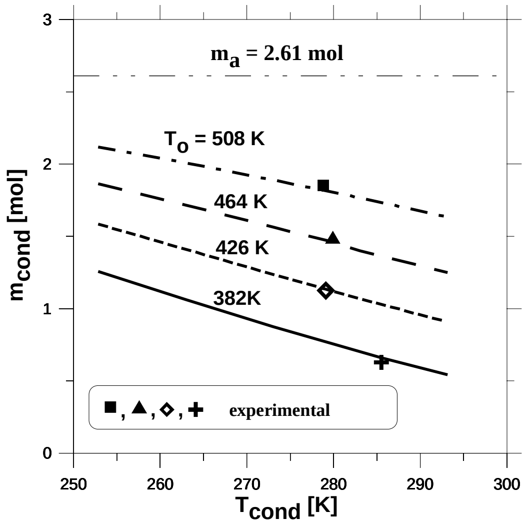


Fig. 2. Simulation and experimental results for the recovery of toluene in TSA system with closed-loop regeneration (m_a is the amount of organic compound adsorbed at the end of adsorption step)

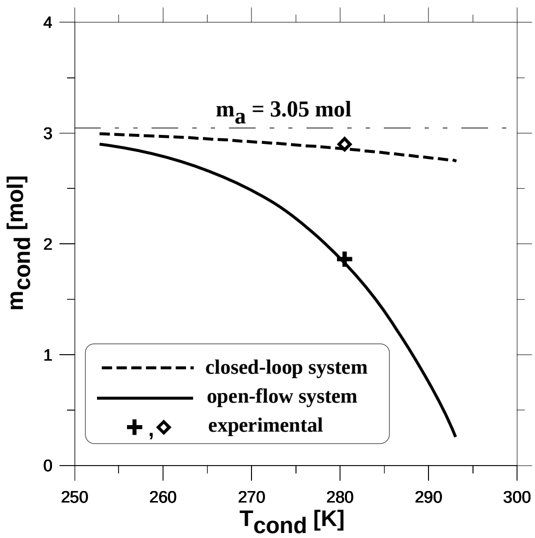


Fig. 3. Comparison of simulation and experimental results for the recovery of isopropanol in TSA systems with closed-loop and open-flow regeneration

The comparison of the simulation and experimental results for the recovery of isopropanol in TSA systems with closed-loop and open-flow regeneration is shown in figure 3. The process parameters are the same as for the recovery of toluene, except for the concentration of isopropanol in the inlet feed during adsorption, which is set at $2.57 \cdot 10^{-3}$ mol/mol. The inlet temperature of purge gas during desorption is 499 K. The mass of liquid isopropanol recovered in a condenser is much higher for closed-loop regeneration than for the open-flow one, especially for higher values of condensation temperature.

As may be seen from figures 2 and 3, the mathematical model employed in this work simulates the experimental data very closely.

5. CONCLUSION

- The possibility of recovering VOCs in cyclic fixed bed TSA system with closed-loop regeneration of adsorbent is studied based on experimental and simulated results. Toluene and isopropanol are chosen as adsorbates. Activated carbon of Sorbonorit 4 type is used as an adsorbent.
- The results obtained in closed-loop and open-flow systems are compared.
- In the closed-loop system, volatile organic compounds can be easily recovered at a condenser temperature equal to that of the surroundings.
- In the case of open-flow system, low condensation temperatures are required for the recovery of organic compounds.

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USUWANIE I ODZYSKIWANIE LOTNYCH ZWIĄZKÓW ORGANICZNYCH Z GAZÓW ODLOTOWYCH W INSTALACJI ADSORPCYJNEJ TSA Z ZAMKNIĘTYM OBIEGIEM GAZU PODCZAS REGENERACJI

Przedstawiono wyniki analizy teoretycznej oraz badań doświadczalnych nad odzyskiwaniem lotnych związków organicznych z powietrza w cyklicznym układzie adsorpcyjnym TSA, w którym regeneracja jest prowadzona w obiegu zamkniętym. Cykl adsorpcyjny składał się z trzech etapów: adsorpcji związku organicznego ze strumienia chłodnego powietrza, desorpcji zaadsorbowanego związku przy użyciu ogrzanego strumienia azotu oraz chłodzenia złoża adsorbentu za pomocą strumienia chłodnego azotu. Badania wykonano dla dwóch związków organicznych: toluenu i izopropanolu. Jako adsorbent stosowano węgiel aktywny Sorbonorit 4.