

EVALUATION OF ENERGY SAVING POSSIBILITIES IN CYCLIC FIXED-BED ADSORPTION PROCESS

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The cyclic Electrothermal Temperature Swing Adsorption (ETSA) process in a fixed-bed column with Supersorbon K40 activated carbon (AC) was applied to remove propan-2-ol (IPA) from air. The bed was electrothermally regenerated using direct resistive heating method. The tests were performed in the range of operating parameters: IPA loading 0.18-0.26 kg/kg, voltage 19.5 V, set-point temperature 393–403 K, nitrogen flow rate 0.12 m³/h.

The analysis revealed, that raising the bed temperature resulted in an increase of desorption degree of adsorbate, reduction of regeneration time and an increase in the energy consumption. The application of insulation enabled reduction of energy consumption and regeneration time by 27% and 10%, respectively.

Keywords: supersorbon K40, activated carbon, electrothermal heating, regeneration

1. INTRODUCTION

Volatile organic compounds (VOCs) are a group of carbon-containing compounds characterized by high vapour pressure greater than 0.01 kPa at 293.15 K (Council Directive, 1999). They include a variety of chemicals, such as toluene, acetone, methanol or propan-2-ol (IPA), that may cause short- or long-term adverse health effects. IPA is commonly used as a solvent, a cleaning agent, an antifreeze additive, a substrate of many chemical reactions and a component of cosmetics (Logsdon and Loke, 2000). The known effect of exposure to IPA vapours on human health are: throat and respiratory tract irritation, drowsiness, dizziness, skin dryness and cracking, eyes irritation (Logsdon and Loke, 2000). It can be removed from the gas streams using combustion, absorption or adsorption methods.

Adsorption on activated carbon (AC) is one of the most commonly employed methods for the removal of VOCs from diluted waste gases. Gas phase applications segment is the fastest growing part of the global activated carbon market. It is expected that the size of entire AC market reach over 3,500 Mg in 2021, compared to about 2,700 Mg in 2015 (Activated Carbon Market, 2017). Granulated Activated Carbon (GAC) accounts about 35% of its total global production (Activated Carbon Market Size, Share & Trends Analysis Report, 2019). GACs are generally manufactured from coal, coconut shell, peat and other carbon rich organic raw materials (Aygün et al., 2003).

The adsorbent characteristics (porosity, pore structure and surface chemistry), the nature of the adsorbate (molecular size, boiling point, polarity) and process conditions have a large impact on adsorption per-

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formance (Králik, 2014; Ribeiro et al., 2014). A detailed knowledge of adsorbate-adsorbent properties is helpful to design adsorption columns widely applied for VOC removal (Patel, 2019).

AC capacity gradually decreases during the process, which reduces its ability to remove VOC from the gas stream. The spent carbon may be regenerated on-site or off-site (Guyer, 2017). Otherwise, the carbon bed is replaced with a virgin one and the spent adsorbent is disposed as a hazardous waste. The on-site regeneration is widely used for the VOC recovery from air containing one or two compounds. The adsorbent capacity is governed by isotherms, thus the equilibrium conditions must be changed (temperature, pressure) to desorb the adsorbed compounds and re-use regenerated carbon for adsorption (Downarowicz, 2015; Ribeiro et al., 2014).

Strong-bonded VOCs can be recovered from the waste gas in Temperature Swing Adsorption (TSA) systems that operate in a three-stage cycle based on a periodic temperature change (Ambrożek, 2009). Waste gas composition determines required contact time in the adsorption column, AC loading and regeneration frequency. Ideally, the regeneration should restore the original carbon capacity. To ensure trouble-free gas stream purification in two column TSA system, adsorption stage must last longer than other stages (Downarowicz and Gabruś, 2009). The adsorbent regeneration method should ensure fast and energy-efficient restoration of the original adsorption capacity. The selection of this method is closely related to the type of contamination present in the gas and implemented adsorbent. In conventional adsorption systems, a saturated fixed bed is thermally regenerated using steam or hot inert gas (Bathen and Breitbach, 2001). The latter method is employed to desorb water-soluble solvents, such as e.g. aliphatic alcohols. However, it is a long-lasting and energy-consuming process which produces dilute gas mixtures (Patel, 2019).

A good alternative to these methods could be a direct resistive heating method (Downarowicz, 2015) in which the Joule effect is employed to heat the regenerated adsorbent (Lupi et al., 2015; Zou et al., 2019). In this method, adsorbent bed is placed between a pair of electrodes while voltage is applied to them. The method enables easy temperature control and a possibility to heat the AC bed to a relatively high temperature in a short time. It can be utilized to regenerate ACs with suitable electrical properties (Subrenat and Le Cloirec, 2006).

Although GAC is widely applied in TSA systems, the literature contains only a few reports on application of resistive heating methods to regenerate this adsorbent. In addition, most studies were dedicated to the activated carbon fiber cloth and activated carbon monolith (Emamipour et al., 2007; Luo et al., 2006; Subrenat and Le Cloirec, 2006; Subrenat et al., 2000; Yu et al., 2007). Regardless of the method, process conditions should be optimized to ensure the highest possible regeneration efficiency with low energy consumption.

The purpose of this article is to investigate the effect of desorption temperature and thermal insulation use on the regeneration efficiency, the energy consumption and the process time.

2. MATERIAL AND METHODS

2.1. Materials

The adsorbate was a liquid IPA analytical grade purity 99.7%, purchased from Chempur (Poland). It is a colourless alcohol with a characteristic smell, completely miscible with water and most organic solvents (Propan-2-ol safety data sheet, 2015). The flash-point is 12 °C, the auto-ignition temperature exceeds 450 °C (Propan-2-ol safety data sheet, 2015; Yaws, 2003). IPA vapors form a flammable mixture with air in the range of 2.2–10.9% vol. (Propan-2-ol safety data sheet, 2015). They can be released into the atmosphere during production, handling, storage and transport processes.

Extruded, steam activated carbon Supersorbon K40 (SK40) was selected as the adsorbent. It was manufactured from coal by Donau Carbon GmbH & CO KG (Germany). Owing to its properties (total surface area BET 1100 m²/g, bulk density 450 kg/m³) it could effectively be used in solvent recovery applications. Due to its favorable electrical properties it can be regenerated by the resistive heating method (Supersorbon K40 – Technical Data Sheet).

2.2. Fixed-bed experiment

The experiments were conducted in a glass adsorption column (0.042 m ID) with the Supersorbon K40 fixed bed with the mass of 0.098 kg and height of 0.16 m. The adsorbent bed was placed between two pyramid-shaped, perforated copper electrodes connected to a power source. A spring was applied to pressure the upper electrode to the bed. The scheme of adsorption column is presented in Fig. 1.

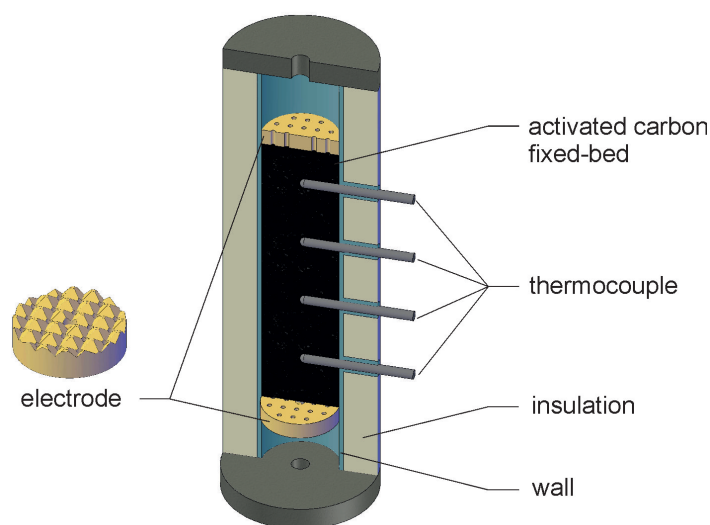


Fig. 1. A schematic diagram of adsorption column

The experimental set-up is presented in Fig. 2. A stream of compressed, dried air was supplied to the installation. The gas flow rate was regulated by a mass flow controller (model GFC 47, Aalborg, accuracy: $\pm 0.25\%$). A liquid propan-2-ol was continuously dosed to a static mixer with a syringe pump (model 100, KD Scientific, accuracy: $\pm 1\%$). The resulting vapor-air mixture was then cooled to ambient temperature. The mixture flowed into the adsorption column where the IPA vapor was adsorbed. The stability of the inlet and outlet alcohol concentrations was controlled by means of a gas chromatograph (SRI 8610C) equipped with a flame ionization detector (FID). Peak Simple Chromatography software (SRI Instruments, ver. 3.93) was employed for peak analysis.

In the adsorption stage, an IPA-laden air stream with specified concentrations C_0 up to 10 g/m³ was purified in the fixed-bed columns. The superficial gas velocity based on an empty column was 0.25 m/s. The breakthrough (BTC) tests were conducted until complete bed exhaustion had been reached, indicated by the column outlet concentration equal to that at the inlet. The duration time of adsorption stage was always higher than 4 hours. After each BTC test, the saturated adsorbent was regenerated by the direct resistive heating method. Pure SK40 was resistively heated up to 393 K and then cooled down to room temperature (cycle A0).

The regeneration stage was carried out in a nitrogen atmosphere to minimize the possibility of self-ignition of the bed. The nitrogen flow rate was controlled by a GFC 47 mass flow controller. The superficial gas velocity was maintained at 0.02 m/s. The nitrogen flow was counter-current with respect to the flow

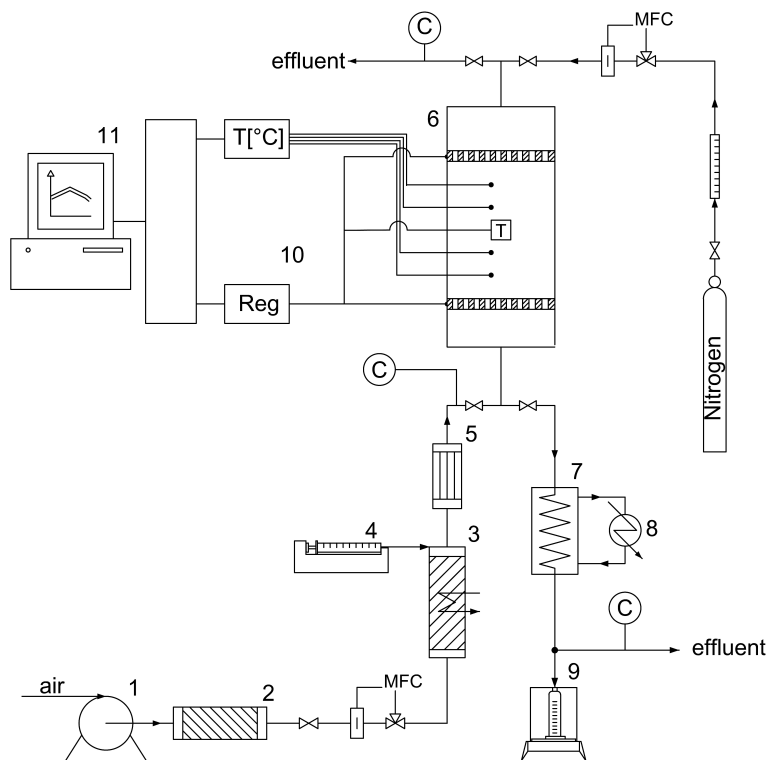


Fig. 2. Experimental setup; 1 – compressor; 2 – desiccant dryers, 3 – static mixer, 4 – VOC syringe pump, 5 – water cooler, 6 – adsorption column, 7 – condenser, 8 – refrigerating circulator, 9 – electronic balance, 10 – regulator, 11 – computer, T – temperature sensor, C – sampling point

direction during adsorption. The concentrated gas flowed through a condenser. Then, propan-2-ol vapors were separated from the inert gas. IPA liquid was collected and weighed, pure gas was removed from the system.

The process was carried out at a constant voltage. The temperature of the activated carbon bed was controlled by means of a Pt100 sensor coupled with a thermoregulator. DASYLab (National Instruments, Ireland) software was implemented in a data acquisition system to record the experimental data with a 1 s sampling rate.

Two series of cyclic adsorption-regeneration tests were carried out. The studies in the A series were performed in the exposed column at 393 K, while in the B series at 403 K. In addition, in the B3_{INS} cycle the column was covered with a 40 mm layer of mineral wool to avoid excessive heat loss. The experiments were performed in the following invariability range of operating parameters: voltage 19.5 V, nitrogen flow rate 0.12 m³/h, respectively.

After completion of successive adsorption and regeneration steps, the adsorption column was weighed. This procedure enabled to determine the total adsorbate loading in the fixed bed as well as the residual bed capacity after the regeneration step which did not exceed 2% of the virgin capacity of the adsorbent.

3. RESULTS AND DISCUSSION

3.1. Electrothermal characteristic of fixed-bed regeneration

Figure 3 depicts the temperature characteristics for SK40 bed heating at 19.5 V. As can be seen, the process consists of heating-up and desorption steps. During the first step the AC bed temperature increased

monotonically until the set-point temperature was reached. In the second step, the heating process was controlled by an on/off control system that switched off the power source. It stabilized the bed temperature during the process phase.

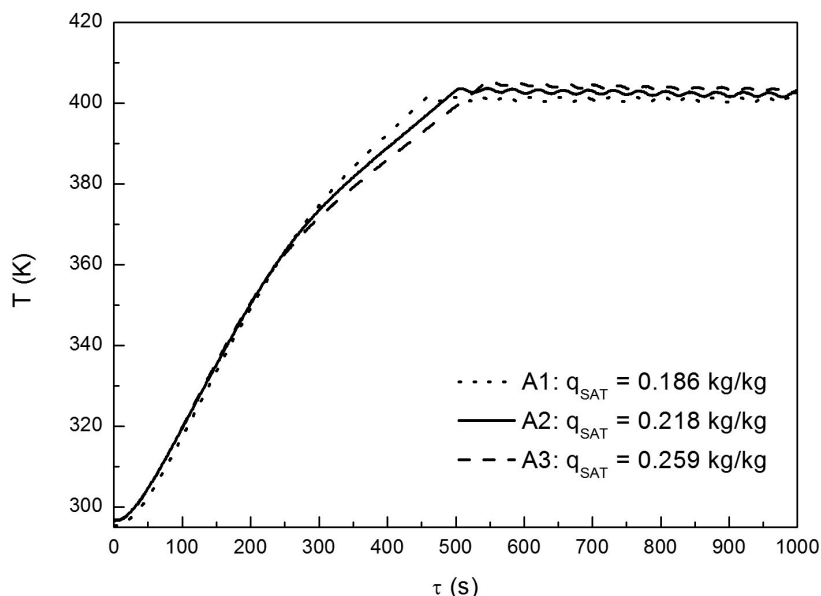


Fig. 3. Bed temperature profiles for series A at constant voltage of 19.5 V

At temperatures up to 365 K, the shape of the kinetic curves was almost the same, while at higher temperatures the heating rate decreased as the IPA loading increased. This is the effect of IPA desorption, which takes place at temperatures above the boiling point (355 K).

Energy efficiency was analyzed based on electrical power and heating energy characteristics. Electrical power (P_{el}) generated in the carbon bed is directly proportional to the electric current (I) and voltage (U) (Luo et al., 2006):

$$P_{el} = I \cdot U \tag{1}$$

The power characteristics for various initial IPA loadings are shown in Fig. 4.

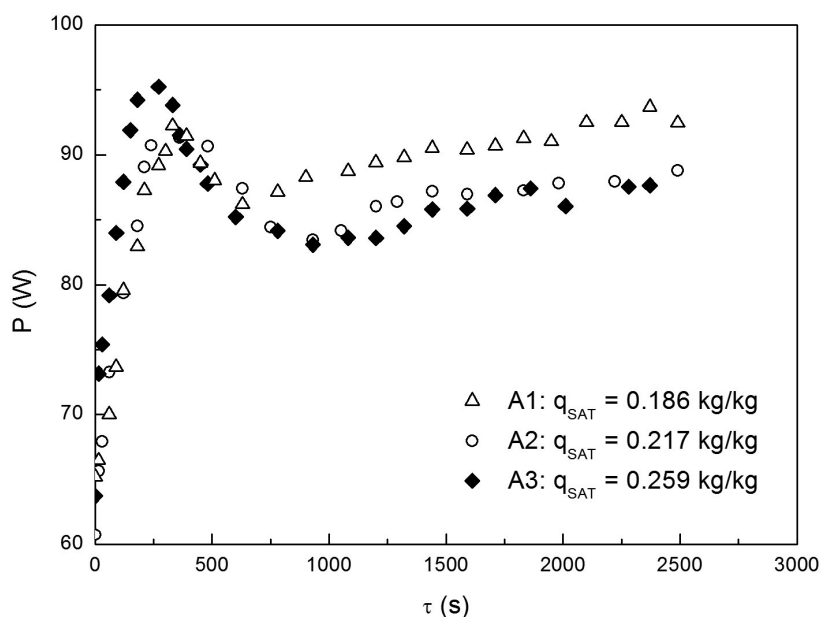


Fig. 4. Electrical power characteristic at 19.5 V for various initial IPA loading

In the initial phase, a linear increase in electrical power was observed. The maximum electrical power values for the A1 and A3 cycles were 92 W and 96 W, respectively. The higher power generated in the fixed bed resulted in faster desorption (Table 1).

3.2. Evaluation of regeneration efficiency

The electrical energy E_{el} consumed by the system is directly proportional to electrical power and the process time. It can be calculated from the area under the electrical power characteristic:

$$E_{el} = \int P_{el} dt \quad (2)$$

Figure 5 illustrates the electrical energy characteristics for cycles A0–A3.

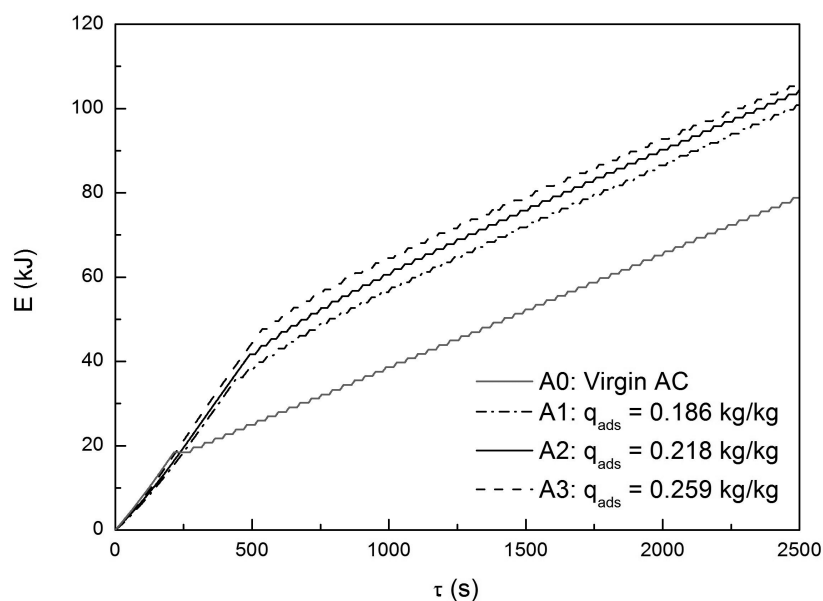


Fig. 5. Electrical energy characteristic at constant voltage of 19.5 V

According to Fig. 5, an increase in the adsorbate loading from 0.186 to 0.259 kg/kg caused an increase in the energy demand and heating time for the A series by ca. 21% and 32%, respectively. The values of heating-up times and energy consumption for initial step and whole process were collected in Table 1.

Table 1. Summary of time and energy consumption for heating-up phase and total energy for regeneration stage

Cycle	$\tau_{heat-up}$	$E_{heat-up}$	E_{total}
A0	215	18.4	108.4
A1	445	36.2	129.6
A2	534	43.7	134.9
A3	538	47.7	135.1
B1	528	47.6	148.0
B2	597	55.9	146.6
B3 _{INS}	592	54.3	106.5

Table 1 reveals that heating a saturated adsorbent requires time twice as long as that for pure adsorbent. The AC bed and the flowing gas consume energy only to heat up.

Effects of bed temperature and application of insulation on electrical energy consumption are presented in Fig. 6.

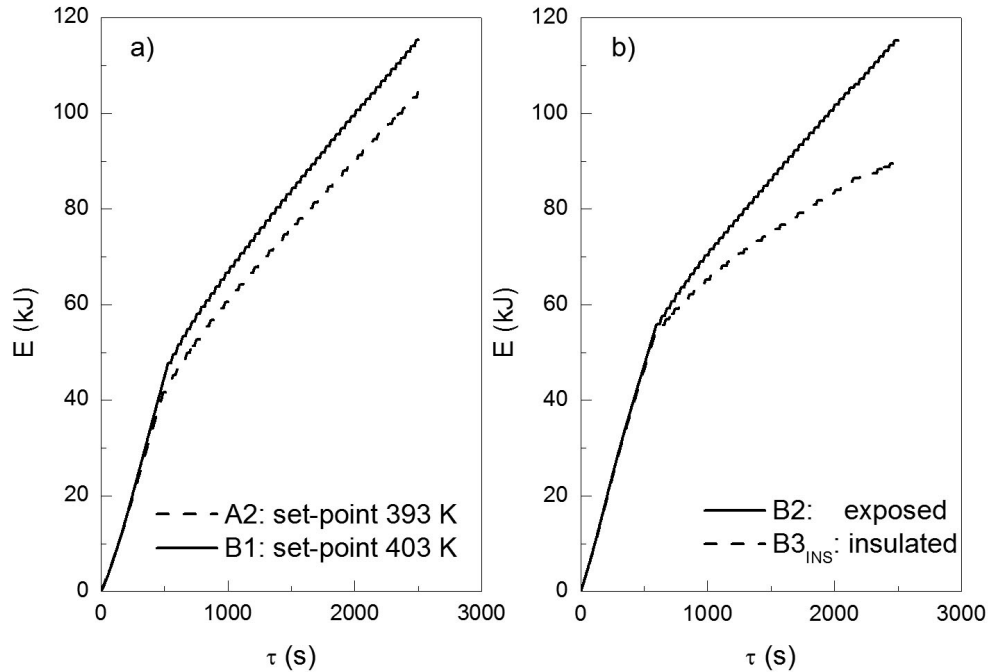


Fig. 6. Electrical energy characteristic for: a) various bed temperature ($q_{SAT} = 0.218$ kg/kg); b) exposed and insulated column ($q_{SAT} = 0.278$ kg/kg)

As can be seen from Fig. 6a, an increase of the set-point temperature by 10 K resulted in an increase in the heating-up and total energy by 6.3 kJ and 14.5 kJ (13% and 11%), respectively. The change in set-point temperature did not significantly affect the $E_{heat-up}/E_{total}$ ratio.

Figure 6b presents that the impact of using insulation on energy consumption in the initial phase is insignificant (ca. 3%). However, for the desorption step, energy consumption decreased by about 42%. For the B3_INS cycle, more than 50% of total energy was consumed in the initial regeneration step.

The desorption degree η_{des} (%) was evaluated using the following equation:

$$\eta_{des} = \frac{m_{des}}{m_{sat}} \quad (3)$$

The energy efficiency degree η_{el} (kJ/mol) was calculated as:

$$\eta_{el} = \frac{E_{el}}{n_{des}} \quad (4)$$

The energy consumption and desorption degree for the A3 cycle was depicted in Fig. 7.

The inflection points on the graphs of η_{des} and E determine the start of on/off control system. The duration time of the proper desorption step was about 9 times longer than that of heating-up, but the energy consumption for both steps was almost the same. As can be seen, roughly 30% of total energy was indispensable in the initial step of the process. It was indispensable to heat the AC bed, inert gas stream, the adsorption column, and to desorb about 18% of the adsorbate.

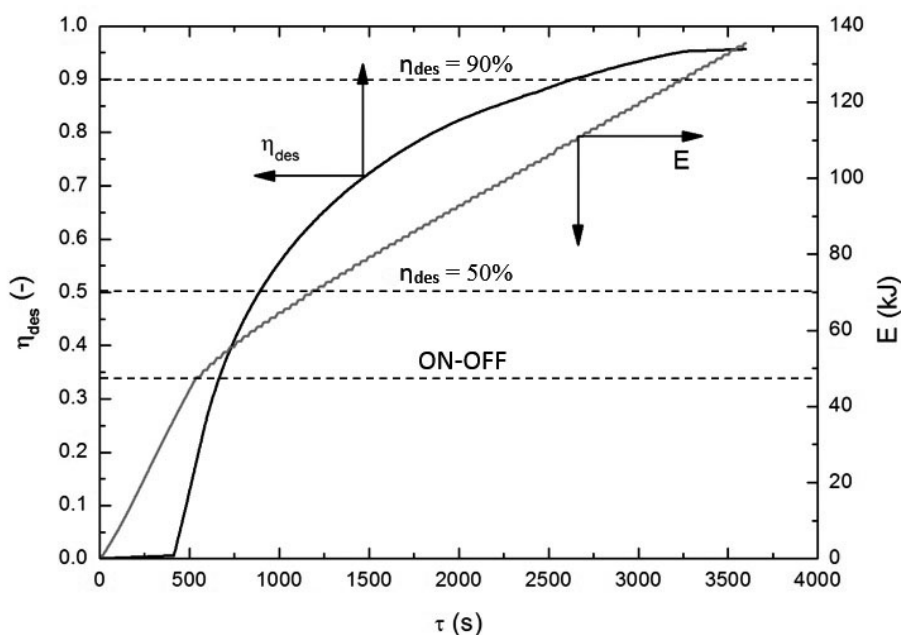


Fig. 7. Changes of desorption efficiency and energy consumption during regeneration cycle A3

Figure 7 displays that an increase in the regeneration time resulted in an increase of desorption efficiency and energy consumption.

The results for series A and B were summarized in Table 2.

Table 2. Electrothermal regeneration process parameters in series A and B

Cycle	q_{SAT} , kg/kg	U , V	$T_{set-point}$, K	$\tau_{90\%}$, s	η_{des} , -	η_{el} , kJ/mol
A1	0.186	19.33	393	2766	0.91	465
A2	0.218	19.24	393	2567	0.92	409
A3	0.259	19.40	393	2591	0.92	345
B1	0.216	19.93	403	2152	0.95	442
B2	0.279	20.02	403	2027	0.95	336
B3 _{INS}	0.278	20.13	403	1841	0.97	239

Table 2 shows, that an increase in the adsorbate loading resulted in an increase of energy efficiency, as indicated by the lower η_{el} values. Higher desorption degree values were obtained from the B series. This was the effect of increasing bed temperature. The duration time of desorption needed to achieve a required desorption degree ($\eta_{des} = 90\%$) was also shorter. The process was most effective in the B3_{INS} cycle when the column was thermal insulated.

The effect of using insulation for cooling time was demonstrated in Fig. 8.

As can be seen from Fig. 8, the cooling time in the B3_{INS} cycle was longer three times than in B2 cycle. It was caused by higher heat storage capacity of the insulated bed, which resulted in lower cooling rate.

To ensure continuous work of a two-column adsorption installation, the total time of the regeneration and cooling stages should be shorter than the time of the adsorption stage.

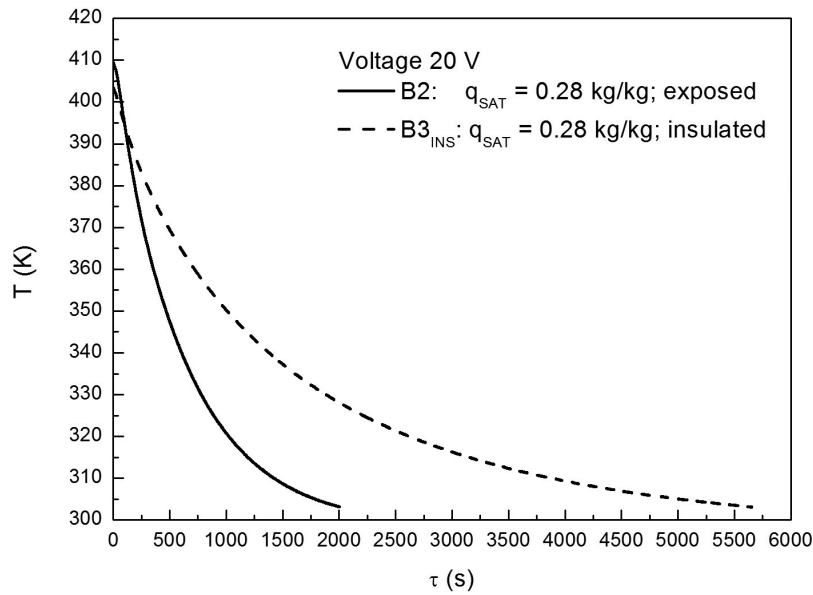


Fig. 8. Temperature profiles for fixed bed cooling stage

4. CONCLUSION

- The studies were carried out in the adsorption column filled with extruded Supersorbon K40 AC. The fixed-bed worked in a three-stage system based on cyclic temperature changes. The direct resistive heating method was used to regenerate the exhausted adsorbent.
- Electrothermal desorption was most effective in recovery when the IPA loading in adsorbent increased. It was the result of a lower electrical resistance of a more saturated AC bed. For adsorption capacities $q_{SAT} = 0.186$ kg/kg (cycle A1) and $q_{SAT} = 0.259$ kg/kg (cycle A3), the energy efficiency degrees amounted to 465 kJ/mol and 345 kJ/mol, respectively. This also resulted in a slight reduction (about 6%) of time needed to achieve $\eta_{des} = 90\%$.
- Energy consumption increased by over 13 kJ (ca. 10%) in the B1 cycle compared to the A2 cycle (with 10 K increase in set-point temperature) for an initial IPA loading ($q_{SAT} = 0.218$ kg/kg). However, increase of the bed temperature resulted in an increase in the rate of desorption. In cycle B1 achieving a required desorption degree ($\eta_{des} = 90\%$) occurred about 420 s (ca. 16%) earlier than in cycle A2. Degree of desorption measured at the end of the process (after 1 hour) was 0.92 and 0.95 for cycles A2 and B1, respectively.
- The application of thermal insulation resulted in a decrease in energy demand by 27% and an increase in energy efficiency by 29% compared to the values obtained for the exposed column. In addition, the same values of desorption degree were obtained faster by 10%. However, this resulted in 3 times longer cooling time. The results are consistent with our earlier research for IPA-BPL 4x6 system (Downarowicz and Kowalski, 2020).
- The time of the adsorption stage was longer than the other stages, which is required to ensure the continuity of operation of the two-column installation.
- However, the choice of the operation parameters should be balanced in order to compromise between the reduction of electricity consumption and the total process time.

SYMBOLS

C_0 initial concentration, g/m³
 E_{el} electrical energy, kJ

$E_{heat-up}$	energy demand during heating-up phase, kJ
E_{total}	total energy demand, kJ
I	electric current, A
m_{des}	desorbate mass, g
m_{sat}	total adsorbate mass in saturated adsorbent, g
n_{des}	number of moles of desorbate, mol
P_{el}	electrical power, W
$T_{set-point}$	set-point temperature, K
q_{SAT}	adsorbate loading, kg/kg
U	voltage, V

Greek symbols

η_{des}	desorption degree, %
η_{el}	energy efficiency degree, kJ/mol
τ	process time, s
$\tau_{90\%}$	time to achieve desorption degree of 90%, s
$\tau_{heat-up}$	heating-up time, s

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