Pilot-scale Experiment for Purification of CO from Industrial Tail Gases by Pressure Swing Adsorption[*]

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Abstract Using pressure swing adsorption (PSA) technology to purify carbon monoxide (CO) discharged from industrial gases is a high-efficiency and economical method. In this article, a four-bed PSA experiment for CO purification was improved and optimized, in which a set of 120 $m^3 \cdot h^{-1}$ pilot-scale PSA device was developed to purify CO from industrial tail gases, a set of control systems suitable for industry production was developed, and the influences of the operating parameters on CO purification were investigated. The experimental results indicated that the pilot-scale PSA device could produce qualified product gas and get high CO recovery ratio under optimized conditions. The research may provide reliable fundamental data, for industrial scale utilization of CO, from industrial tail gases, and have strong market competitive power and a broad promoted application prospect.
Keywords pressure swing adsorption, purification, separation, carbon monoxide

1 INTRODUCTION

The demand for energy and synthetic materials is increasing gradually in the modern economic world. Petroleum, coal, and natural gas, which are flammable mineral resources, occupy 85% of the world's energy and organic synthesis industry [1]. However, all of these industrial tail gases include green-house gases [2], in which CO_2 contributes 55% to the green-house effect [3], which brings about global climate change, one of the greatest environmental problems of modern society [4].

With the shortage in energy and chemical materials, especially the increasing consumption of petroleum resources, C_1 chemical industry, which primarily includes coal and natural gas, is significantly important. In 2006, the output of organic products produced by CO was close to 20 thousand million RMB in China and was over one hundred billion RMB in the world. The demand for carbonyl synthetic products and the element material CO are increasing gradually [5]. High-purity CO is a very important production material in the C_1 chemical industry. However, many industrial processes discharge CO in tail gases [6, 7], and most of them are used as fuel because that CO concentration is too low to retrieve as the recovery of CO is expensive [8, 9]. If CO in tail gases can be purified and used in the C_1 chemical industry, it will not only achieve great economic benefit because of the lower production costs [10], but there will also be an environmental benefit, as there will be less discharge of greenhouse gases, CO and CO_2. For using CO from industrial tail gases effectively, high-efficiency CO purification technology needs to be developed and applied.

In this study, CO, purified by the method of PSA has higher purity, higher recovery ratio, and lower cost and energy consumption. Compared with the other methods of PSA to purify CO [11–14], the characters of the optimized four-bed PSA process for CO purification are as follows: (1) the pressure process is divided into direct and indirect pressure equalization, (2) the purge step is canceled in PSA circulation, resulting in high productivity and high CO recovery ratio, (3) the final pressurization step is canceled, which promotes productivity again.

2 EXPERIMENTAL

2.1 Experimental devices and operational procedure

The PSA process with four parallel connected towers was applied in this study to purify CO from the purge gas in liquid nitrogen, in the washing section of the ammonia synthesis system in Yunnan Jiehua Chemistry Group Co., Ltd. (a kind of industrial tail gas containing CO). The designed gas flow rate was $120 \ m^3 \cdot h^{-1}$. The flowchart of the system is shown in Fig. 1. This system was composed of four parallel connected adsorbers filled with PU-1 adsorbent [15–19], (CuCl/zeolite adsorbent featured with a high adsorb capacity and selectivity), which had been made by the Peking University and the Beijing Peking University Pioneer Technology Co., Ltd. According to the principle that compounds can spontaneously disperse to the surface of supports to form a monolayer, CuCl might highly disperse to the surface of the zeolite. Inductors and sensors were connected to the adsorber and program-controlled machine (with computer), to monitor the change in the adsorption process and collect data. Computer programs were developed to analyze the data and control the valves in the process. Every
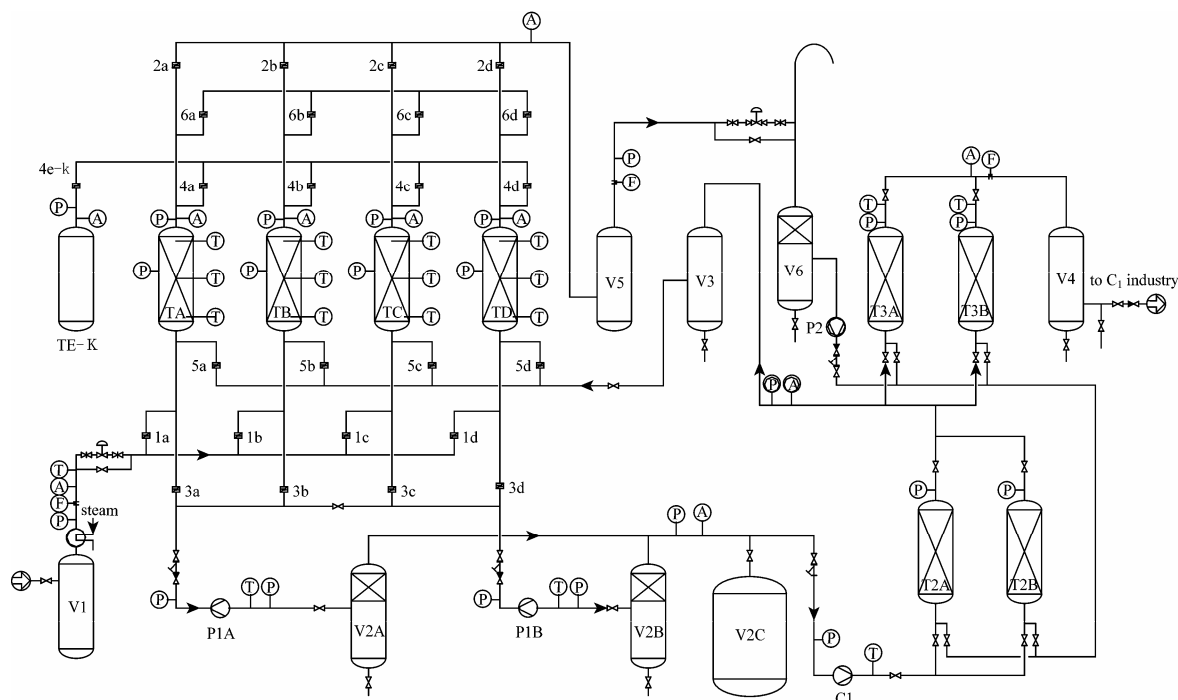
Figure 1 Flowchart of pilot-scale device for four-bed PSA process
TA/B/C/D—A/B/C/D adsorber; TE–K—PEB; V1—feed gas buffer; V2A/B—tank next to vacuum pump; V2C—CO container; V3—rinse gas buffer; V4—product gas buffer; V5—final buffer; V6—water segregator; P1A/B—product gas vacuum pump; P2—desiccant vacuum pump; C1—product gas compressor; T2A/B—drying tower; T3A/B—deoxidizing tower; —◁ needle valve; –⊠– gas-control valve; Ⓟ– pressure meter or pressure sensor ; Ⓣ– thermal couple; ⊖ mass flow meter; ⊖– sample or online gas analytical apparatus

adsorber had one pressure sensor and three K-type thermocouples. CO concentration in feed gas, waste gas, and product gas were analyzed by QGS-08D online CO analyzer, and the ingredients of the product gas were analyzed by a SC3000 chromatography workstation. Additionally, the pilot-scale device was equipped with one water circulation vacuum pump (WCVP) to regenerate the desiccant and two WCVPs to regenerate the adsorbent. Besides four adsorbers (A

to D), there were seven pressure equalization buffers (PEBs) to equalize the pressure in the adsorber between the adsorption and regeneration steps, under different pressures. The system was also equipped with some devices, including feed gas buffer, product gas buffer, ZW-1.5/7 product gas compressor, and so on.

The equipment of the pilot-scale experiment is shown in Table 1. Main parameters of the stuff filled in the pilot-scale device are shown in Table 2. The

Table 1 The equipment of the pilot-scale experiment

No.	Name	Code number	Specification			Amount
1	feed gas buffer	V1	$\phi1200$ mm	$V=2.0$ m^3		1
2	tank next to vacuum pump	V2A/B	$\phi400$ mm	$V=0.5$ m^3		2
3	rinse gas buffer	V3	$\phi500$ mm	$V=0.25$ m^3		1
4	product gas buffer	V4	$\phi1200$ mm	$V=2.0$ m^3		1
5	final buffer	V5	$\phi500$ mm	$V=0.5$ m^3		1
6	water segregator	V6	$\phi400$ mm	$V=0.5$ m^3		1
7	adsorber (with insulation)	TA/B/C/D	$\phi500$ mm	$V=0.25$ m^3		4
8	PEB	TE–K	$\phi500$ mm	$V=0.25$ m^3		7
9	drying tower	T2A/B	$\phi500$ mm	$V=0.25$ m^3		2
10	deoxidizing tower	T3A/B	$\phi500$ mm	$V=0.25$ m^3		2
11	product gas vacuum pump	P1A/B	$Q=3$m$^3\cdot$h^{-1}	$N=5.5$ kW	$n=1450$ r$\cdot$min^{-1}	2
12	desiccant vacuum pump	P2	$Q=3$m$^3\cdot$h^{-1}	$N=5.5$ kW	$n=1450$ r$\cdot$min^{-1}	1
13	product gas compressor	C1	ZW-1.5/7; $Q=1.5$ m$^3\cdot$min^{-1}; $N=15$ kW; $n=730$ r$\cdot$min^{-1}; Suction pressure$=0.1$ MPa			1
14	CO container	V2C	50 m^3			1
15	noxious gases alarm system	–	–			5

Table 2 Main parameters of the stuff filled in the pilot-scale device

Parts	Tower volume/m³	Filled stuff	Mass of filled stuff per tower/kg	Density of filled stuff×10⁻³/kg·m⁻³	Bed voidage
adsorber TA/B/C/D	0.25	PU-1 adsorbent	226±1	0.904	0.33
drying tower	0.25	activated alumina	184	0.736	–
deoxidizing tower	0.25	deoxidizer	172	0.688	–

Table 3 The optimized four-bed PSA process for CO purification

Step	1	2 3 4 5 6 7	8	9 10 11 12 13	(1)
TA	AD	DD ID ID ID PG DD	EV	DR IR IR IR DR	(AD)
		1 2 3 4 5		5 4 3 2 1	
TB		EV	DR IR IR IR DR	AD	DD ID ID ID PG DD	(EV)
		5 4 3 2 1	DD ID ID ID PG DD 1 2 3 4 5		
TC	EV DR IR IR IR DR	AD	DD ID ID ID PG DD	(EV)
	5 4 3 2 1	1 2 3 4 5		
TD	PG DD	EV	DR IR IR IR DR	AD	DD ID ID ID
	5	5 4 3 2 1		1 2 3 4

Note: (1) AD—adsorption; DD—direct depressurization; ID—indirect depressurization; PG—purge; EV—evacuation; DR—direct repressurization; IR—indirect repressurization.
(2) Purge process and system connection should be kept, whereas, the purge process needs to be removed when the system is running.
(3) The values in the table are the indirect average pressure ($n=3$) as a result of the study.

Table 4 Experimental conditions of a PSA cyclic process

Adsorption pressure /kPa	Desorption pressure /kPa	Feed gas flow rate /m³·h⁻¹	CO concentration in feed gas/%	Temperature of feed gas /K	Time/s												The total cycle time /s
					DR5	IR4	IR3	IR2	DR1	AD	DD1	ID2	ID3	ID4	DD5	EV	
300	20	120	38.71–41.45	304	16	16	16	16	16	100	16	16	16	16	16	228	488

optimized four-bed PSA process and parameters are shown in Table 3 and Table 4 respectively.

During the process, twelve steps occurred in every adsorber. An adsorber TA was taken as an example below. (1) AD: After being pressurized by a gas compressor, the feed gas went through buffer V1 and flowed into TA, in which CO was selectively adsorbed and other ingredients were released. (2) DD1: The gas-control valve in the TA entrance was closed and TA was connected to TC, which had completed the IR2 process, and then the first depressurization occurred. (3) ID2: TA was connected to the first PEB, and the second depressurization happened in TA. (4) ID3: TA was connected to the second PEB, and the third depressurization happened in TA. (5) ID4: TA was connected to the third PEB, and the fourth depressurization happened in TA. (6) DD5: TA was connected to TB, which had completed the EV process and depressurization for the last time was fulfilled. (7) EV: TA was connected to P1A and then vacuumized. CO adsorbed by PU-1 adsorbent was desorbed and flowed into V2A. If the product gas CO was up to standard, it would be sent into T3A and V4 and used for C_1 chemical industry. Else it would be sent into V3 and the adsorber. (8) DR5: TA was connected to TB, which had completed the ID4 process and had begun the first repressurization. The sequence was counted in inverted order to match the sequence of depressurization. (9) IR4: TA was connected to the third PEB and began the second repressurization. (10) IR3: TA was

connected to the second PEB and began the third repressurization. (11) IR2: TA was connected to the first PEB and began the fourth repressurization. (12) DR1: TA was connected to TD, which had finished the AD process and had begun the last repressurization of TA. Then TA went to the next circulation.

According to the results over a long run, the ingredients of the purge gas in the liquid nitrogen washing section of ammonia synthesis system are shown in Table 5.

Table 5 Ingredient analysis of the purge gas from liquid nitrogen washing section in ammonia synthesis system

Ingredient	Volume fraction/%
CO	25–45
N_2	41–64
CH_4	9.3–17.8
H_2	2.5–8

2.2 Processing method for experimental data

2.2.1 *Dynamic equilibrium adsorption quantity*

The dynamic equilibrium adsorption quantity can be measured by Dynamic Column Breakthrough Test (DCBT) [20]. The dynamic equilibrium adsorption quantity formula, under certain adsorption pressure and temperature, is shown in Eq. (1).

$$q_i^* = \frac{\left(V_{in} - V_{end}\right) \times \dfrac{273.15}{T_0} - V_d \times \dfrac{P}{101.325} \times C_i}{22400 \times m} \quad (1)$$

2.2.2 *Product gas recovery ratio*

The product gas recovery ratio represents the degree of recovery of active ingredients in feed gas and is an important index to measure the efficiency of the PSA device. It indicates the consumption of the active ingredient, and can be calculated by the ratio of absolute quantity of the active ingredient in product gas and feed gas. The formula is shown in Eq. (2).

$$\eta = \frac{V_P \times X_P}{V_F \times X_F} \quad (2)$$

3 RESULTS AND DISCUSSION

3.1 Breakthrough curve

Breakthrough curve is an important characteristic curve in the PSA process. It reflects adsorption equilibrium relations, adsorption kinetics, and mass-transfer mechanism in the mobile phase and fixed phase and is also the main basis of engineering design and operation [21]. Breakthrough curves of the four adsorption beds are shown in Fig. 2. It was obtained under the conditions where the adsorption temperature was 318 K, adsorption pressure was 300 kPa, CO concentration in the feed gas varied from 37.8% to 40% and the feed gas flow rates were 120 $m^3 \cdot h^{-1}$ (TA), 118 $m^3 \cdot h^{-1}$ (TB), 119 $m^3 \cdot h^{-1}$ (TC), and 118 $m^3 \cdot h^{-1}$ (TD), respectively. Influences of adsorption pressure on breakthrough curves of the adsorption bed were shown in Fig. 3, under the condition that CO concentration in the feed gas was about 40%. PU-1 adsorbent was first regenerated by the product gas vacuum pump. To prevent air going into the adsorber, the opening of the discharging gas-control valve was delayed by 10 s. As shown in Fig. 2, there were some differences in the breakthrough curves because of the differences in the adsorbent filled in the adsorber, filling ways, temperature, flow rate of feed gas, CO concentration, and so on. However there were no big differences in the breakthrough time of the four adsorption beds, which was around 130 s. At the beginning, there was a higher point of CO concentration in the waste gas. And then the breakthrough curve proceeded steadily. It could be explained that the adsorbent could not be desorbed completely and a certain amount remained
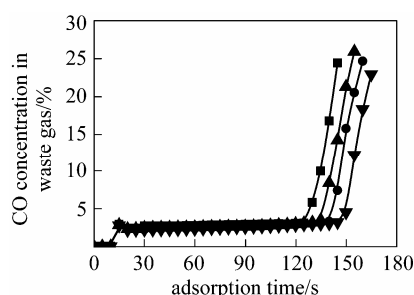
behind. When the next circulation began, part of these remains would be desorbed with the flushing action of the feed gas, under the condition that CO partial pressure was lower than CO balanced partial pressure, which led to high instant CO concentration in waste gas. Although, in the normal adsorption process (130 s), CO concentration in waste gas increased from 2% to 4%.

From Fig. 3, it can be seen that there were no big differences in breakthrough time of the same adsorption bed under different pressures, which indicated that the influences of adsorption pressure on breakthrough curves of the adsorption bed were acceptable.
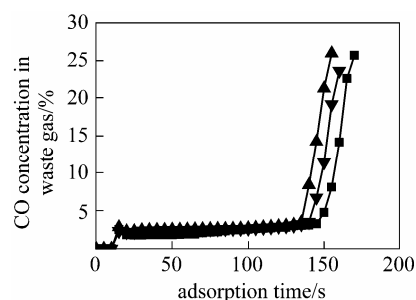

Figure 3 Influences of adsorption pressure on breakthrough curves of adsorption bed
■ $P=250$ kPa, $Q=112$ $m^3 \cdot h^{-1}$; ▲ $P=300$ kPa, $Q=119$ $m^3 \cdot h^{-1}$;
▼ $P=350$ kPa, $Q=120$ $m^3 \cdot h^{-1}$

3.2 Influences of feed gas flow rate on relative CO adsorption quantity

It can be seen from Fig. 4 that there were some differences in adsorption time under different feed gas flow rate. The adsorption time was 200 s and the relative adsorption quantity of PU-1 adsorbent was 0.35 $mol \cdot kg^{-1}$ when adsorption pressure was 300 kPa, feed gas flow rate was 78 $m^3 \cdot h^{-1}$ and CO concentration in feed gas was 40.5%. Although, the adsorption time was 135 s and the relative adsorption quantity of PU-1 adsorbent was 0.33 $mol \cdot kg^{-1}$ when the adsorption pressure was 300 kPa, the feed gas flow rate was 119 $m^3 \cdot h^{-1}$ and CO concentration in feed gas was 37.2%. Therefore, it showed that the relative adsorption quantity of PU-1 adsorbent declined with the increase in feed gas flow rate. It could be explained that with the increase in feed gas flow rate, gas residence time (GRT) decreased and the breakthrough point occurred in advance, though generally relative adsorption quantity was in positive correlation with GRT.
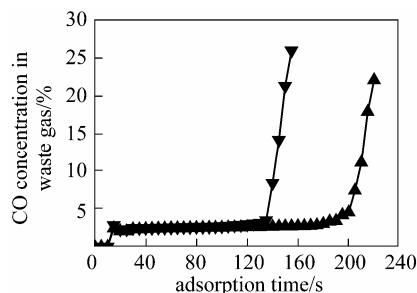

Figure 2 Breakthrough curve of the four adsorption beds
■ TA; ● TB; ▲ TC; ▼ TD

Figure 4 Influence of feed gas flow rate on experimental result
▲ $P=300$ kPa, $Q=78$ $m^3 \cdot h^{-1}$; ▼ $P=300$ kPa, $Q=119$ $m^3 \cdot h^{-1}$

3.3 Influence of CO concentration in feed gas on product gas purity

Usually, CO concentration in feed gas varies with time because of the discontinuous operation of the ammonia synthesis system. Actually CO concentration in feed gas varies from 25% to 45%. The influence of the fluctuation on product gas purity is shown in Fig. 5. It can be seen that the influence of CO fluctuation on product gas purity is slight, because when CO concentration in feed gas is low (25%), the product gas purity is still high. It can be explained by the mass transfer zone (MTZ) in the adsorber, which reaches the controlling station when the adsorption step is complete and results in little quantities of other ingredients getting into the product gas. However, when the CO concentration in the feed gas is higher (45%), because of the control of the MTZ station, the increasing quantity of CO in waste gas is less and a large quantity of it is saved in the adsorber and PEB, which is adsorbed by the next circulation. CO concentration in product gas can be kept at more than 98%, with fluctuation, over a long run. According to the actual demand of CO concentration in product gas, higher purity product gas can be obtained by the purge process with lower CO recovery ratio.
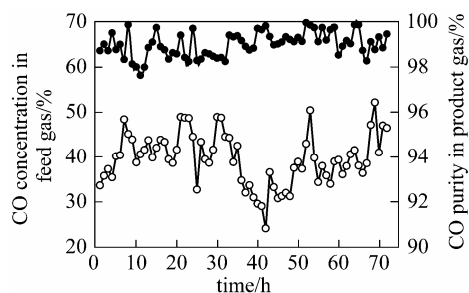

Figure 5   Influence of CO concentration in feed gas on product gas purity
● product gas purity;   ○ CO concentration in feed gas

3.4 Stability analysis of experimental operation

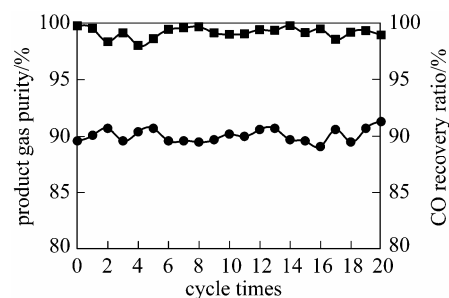The purification influence of feed gas on the pilot-

Figure 6   Influences of cycle times on product gas purity and CO recovery ratio
■ product gas purity;   ● CO recovery ratio

scale device is shown in Fig. 6. As presented in Fig. 6, in any period of 20 cycles, when the adsorption pressure was constant at 300 kPa, the CO concentration was 25%–45%, and flow rate was 115–123 $m^3 \cdot h^{-1}$. The CO concentration of product gas fluctuated from 97.98% to 99.74%, and correspondingly CO recovery efficiency fluctuated from 89.1% to 91.3%. This indicated that CO purification of the purge gas from the ammonia synthesis system had higher stability by PSA. This high stability was because of the efficiency of the pilot-scale facility and the optimization of the operation procedure.

3.5 Temperature variation of adsorption bed during a PSA cyclic process

It is well known that there was an exothermic phenomenon in the adsorption process and an endothermic phenomenon in the desorption process. Therefore the fluctuation of temperature in the adsorption bed was unavoidable, which was disadvantageous to CO purification. It was indicated in many studies [22] that the best separation condition was isothermal, in the adsorption bed. Consequently, it was necessary to study the influences of the thermal effect on the adsorption bed temperature during the adsorption and desorption process. Temperature variation of the adsorption bed during a PSA cyclic process is shown in Fig. 7, under the conditions that are shown in Table 4. It showed that the temperature of the adsorption bed
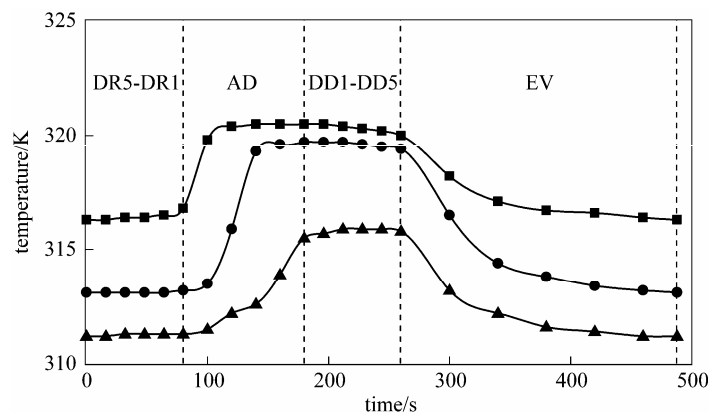

Figure 7   Temperature variation of adsorption bed during a PSA cyclic process
■ lower part of tower;   ● middle part of tower;   ▲ upper part of tower

was less increased in the repressurization process as MTZ had not yet reached the temperature measurement point of the lower part of the adsorption bed. However, in the adsorption process, the temperature of the adsorption bed increased gradually from the lower part to the upper part with MTZ moving forward, adding up to 6.5 K, and the adsorption bed had already reached the breakthrough point, whereas, the upper-part temperature kept on rising. With decreasing of pressure in the depressurization process, the temperature of the lower part and middle part of the adsorption bed fell a bit, whereas, the upper-part temperature continued to increase. Moreover, the temperature decreased in the evacuation process, which indicated that there was an endothermic phenomenon in the desorption process. The temperature decreased rapidly in the beginning 80 s of the evacuation process, which showed that the desorption quantity was large, and later gradually became constant when the desorption time was prolonged.

4 CONCLUSIONS

(1) There was no big difference in breakthrough time in the four adsorption beds, which was around 130 s, and CO concentration in waste gas was kept within 4% when adsorption temperature was 318 K, adsorption pressure was 300 kPa, feed gas flow rate was 120 $m^3 \cdot h^{-1}$, and CO concentration in feed gas was kept at 40%. Furthermore, there was no big difference in breakthrough time of the same adsorption bed under different pressure.

(2) Product gas purity was higher than 98% and CO recovery ratio was about 90% under normal operations, which indicated that CO purification by PSA was very stable.

(3) The temperature of adsorption bed was less increased in the repressurization process. As in the adsorption process, the temperature of the adsorption bed increased gradually from the lower part to the upper part, and the adsorption bed had already reached the breakthrough point, whereas, the upper-part temperature kept on rising. In the depressurization process, the temperature of the adsorption bed's lower part and middle part fell a bit, whereas, the upper-part temperature continued to increase. The temperature decreased in the evacuation process, rapidly in the beginning 80 s, which showed that the desorption quantity was large, and then gradually became constant as the desorption time was prolonged.

(4) The relative adsorption quantity of PU-1 adsorbent reduced with an increase in the feed gas flow rate, which was 0.33 $mol \cdot kg^{-1}$ under normal operations.

The conclusions are reliable fundamental data for industrial design, on the condition that adsorption temperature is 318 K, adsorption pressure is 300 kPa, and breakthrough time is 130 s, the relative adsorption quantity of the adsorbent is about 0.33 $mol \cdot kg^{-1}$, product gas purity is higher than 98% and CO recovery ratio is about 90%.

ACKNOWLEDGEMENTS

NOMENCLATURE

C_i	volume percent of adsorption ingredients, %
m	filled mass of adsorbent in adsorber, kg
P	total pressure in adsorber (absolute pressure), kPa
q_i^*	equilibrium adsorption quantity of ingredient i under partial pressure P_i, $mol \cdot kg^{-1}$
T_0	initial adsorption temperature, K
V_d	dead space of adsorption bed, may be directly measured by the emptying method, ml
V_{end}	volume of adsorption ingredient in waste gas, be integral calculated by breakthrough curve, ml
V_F	volume of feed gas, m^3 (normal conditions)
V_{in}	volume of adsorption ingredient in gas intake, ml
V_P	volume of product gas, m^3 (normal conditions)
X_F	the required ingredient concentration in feed gas, %
X_P	the required ingredient concentration in product gas, %
η	product gas recovery ratio

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Single Crystal Growth of Semiconductors from Metallic Solutions covers the four principal growth techniques currently in use for the growth of semiconductor single crystals from metallic solutions. Providing an in-depth review of the state-of-the-art of each, both experimentally and by numerical simulations. The importance of a close interaction between the numerical and experimental aspects of the processes is also emphasized. Advances in the fields of electronics and opto-electronics are hampered by the limited number of substrate materials which can be readily produced by melt-growth techniques such as the Czochralski and Bridgman methods. This can be alleviated by the use of alternative growth techniques, and in particular, growth from metallic solutions. The principal techniques currently in use are: Liquid Phase Epitaxy; Liquid Phase Electroepitaxy; the Travelling Heater Method, and; Liquid Phase Diffusion. **Single Crystal Growth of Semiconductors from Metallic Solutions** will serve as a valuable reference tool for researchers, and graduate and senior undergraduate students in the field of crystal growth. It covers most of the models developed in recent years. The detailed development of basic and constitutive equations and the associated interface and boundary conditions given for each technique will be very valuable to researchers for the development of their new models.