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Ethanol-Water Separation by Pressure Swing Adsorption (PSA)

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Abstract

Single long spiral tube column pressure swing adsorption (PSA) unit, 25 mm diameter, and 6 m length was constructed to study the separation of water from ethanol at azeotropic concentration of 95 wt%. The first three meters of the column length acted as a vaporizer and the remaining length acted as an adsorber filled by commercial 3A zeolite. The effect of pressure, temperature and feed flow rate on the product ethanol purity, process recovery and productivity were studied. The results showed that ethanol purity increased with temperature and pressure and decreased with feed flow rate. The purity decreased with increasing productivity. The purity range was 98.9 % to 99.6 %, the recovery range was 0.82 to 0.92 and the productivity range was 0.3 to 1.05 kg ethanol/kg zeolite.h.

Keywords: PSA, azeotropic point, 3A zeolite, ethanol-water separation.

Introduction

The main problem of using ethanol as a car fuel is the presence of excess water. Simple binary distillation is used to separate ethanol-water up to maximum 95% as weight percent; further purity of ethanol cannot be got by distillation due to presence of azeotrope. There are many processes to get dehydrated ethanol beyond azeotropic point; pressure swing adsorption (PSA) process in vapor phase is the lowest energy consumption process [1].

In liquid phase water adsorption for ethanol-water mixture [2-4], the adsorbent is usually desorbed by solvent rinse or heating. Solvent rinse requires a suitable solvent and further separation and recovery of the solvent after the rinse. And the method of heating requires long operating period of heating for desorption and then cooling for adsorption, which lowers the productivity of the adsorbent beds. Heat energy is also required to evaporate the liquid remaining in the void of the beds and raise the temperature of the adsorbent and the beds. The gaseous phase adsorption process was proposed by Ladisch and coworkers [5]. PSA is widely used in the separation and purification of gas mixtures mainly because of the easy and quick desorption of the adsorbent only by depressurization [6].

All adsorption processes include two major steps, adsorption and desorption, and almost the process is named by the desorption step. There are two basic adsorption processes: Thermal swing adsorption (TSA) and pressure swing adsorption (PSA). Figure 1 shows the

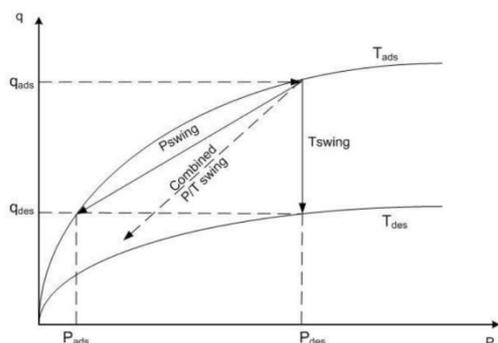principles of the two processes in both adsorption and desorption [7].

Fig. 1: Principles of Thermal Swing Adsorption (TSA) and Pressure Swing Adsorption (PSA) [7]

Desorption step takes rather long time (several minutes to hours) if the thermal swing is used due to slow heat transfer in packed columns while desorption steps takes short time (seconds to minutes) if the pressure swing is used.

Despite many researches on the adsorption of water on 3A zeolite and PSA simulation for ethanol-water mixture [8-14], there are limited studies on the experimental PSA process systems [15-18].

The aim of the present work is to construct a small scale pressure swing adsorption (PSA) unit for the separation of the vapor mixture of ethanol-water beyond azeotropic point, using long spiral bed column, packed with commercial 3A zeolite. The effects of the operating parameters, such as adsorption pressure, adsorption temperature and feed flow rate on the performance of PSA unit is to be studied, using 4-steps cyclic operations. The performance is characterized by ethanol product purity, ethanol recovery and ethanol productivity.

Experimental Work

Figure 2 shows the experimental set-up of the long novel spiral column PSA process. The spiral column is of stainless steel 25 mm diameter and 6 m length. The first three meters act as a vaporizer and the last three meters act as an adsorber filled with one kilogram of 3A zeolite. The spiral coil submerged in oil bath. Four solenoid valves of 6 mm diameter are used. The characteristic of the adsorbent is shown in Table 1. The ethanol purity is measured by Abbe Refractometer, Atago, Japan.
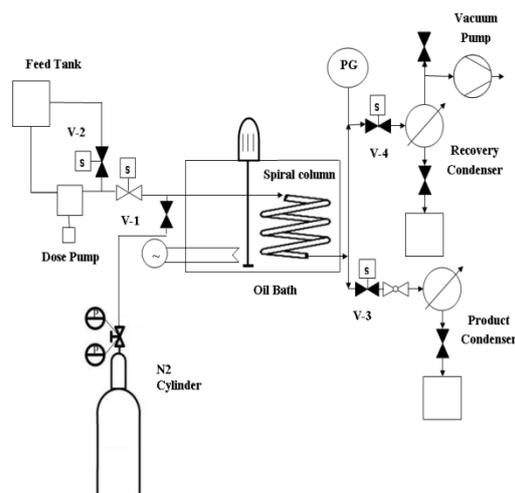

Fig. 2: Experimental setup

Table 1: Adsorbent characteristics

Adsorbent Type	Zeolite 3A
Shape	Sphere
Weight	1 kilogram
Particle diameter	3-5 mm
Structure Formula	$0.45K_2O.0.55Na_2O.AL_2O_3.4.5H_2O$
Bulk density	$779kg/m^3$
Bed porosity	0.41

The parameters considered in the present work are:
- Operating Adsorption Temperature (T_{ads}): 150, 160 and 170 °C.
- Operating Adsorption Pressure (P_{ads}): 2, 3 and 4 bar.
- Feed flow rate (Q): 1, 2 and 3 l/h

- Cycle time: 12 minutes (6 minutes adsorption and 6 minutes desorption).
- Feed Concentration : 95 wt %
- Vacuum desorption pressure (P_{des}): 0.2 bar

The experiments were organized by a three level factorial design of the three operating variables (temperature, pressure, and feed flow rate).

The experimental procedure was:

1. Turn on the oil path and start the control board on manual mode to prepare the system by vacuum and N_2 purging.
2. Set the control board on automatic mode with the specified duration of each step and with the solenoid valves operation cycle as shown in Table 2 and Fig. 3 for 4-steps PSA operation.
3. Adjust the flow rates of feed by regulating the dose pump.
4. Take a sample of product each step and measure the product purity (EtOH %) by calibrated refractometer.

Table 2: Automatic solenoid valves operation of the 4-step PSA system

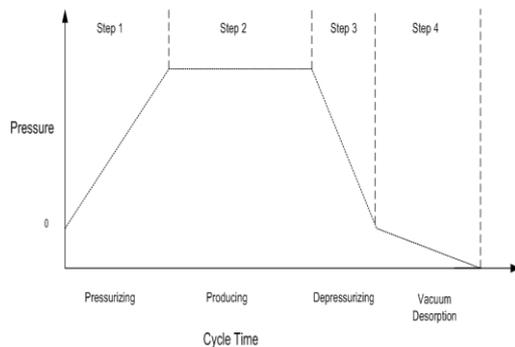| Process	Steps	Solenoid Valves			
		SV1	SV2	SV3	SV4
Adsorption	Pressurizing	Open	Close	Close	Close
	Producing	Open	Close	Open	Close
Desorption	Depressurizing	Close	Open	Close	Open
	Vacuum Desorption	Close	Open	Close	Open

Fig. 3: Sequence of the 4-steps Pressure Swing Adsorption operation

Results and Discussion

Figure 4 shows the effect of temperature and pressure on product ethanol purity at different levels of feed flow rate. No significance effect of temperature and pressure is noticed on the purity at low feed flow rate of 1 l/h. Whereas significance effects of both temperature and pressure are noticed at high feed flow rate of 2 and 3 l/h. Ethanol purity increases with increasing temperature because the increase of temperature leads to more heat for endothermic desorption process to be more complete at high temperature [10].

Ethanol purity decreases with the pressure increase, in contrast of what expected. The capacity of the adsorbent increases and the vapor velocity inside the column decreases with increasing the adsorption pressure. These lead to increasing the performance of the PSA process [10]. The reason of this unexpected result is due to that adsorbent exhibits more adsorbation in unit time in pressurizing step.

Figure 5 shows the effect of feed flow rate on product ethanol purity for different levels of pressure and temperature of 150 ^{o}C. The purity decreases with increasing the feed flow rate because solid adsorbent exhibits more adsorbation for unit time which makes the column approaches sooner the breakthrough point and saturation early. The same trends were noticed at temperatures of 160 and 170 ^{o}C.
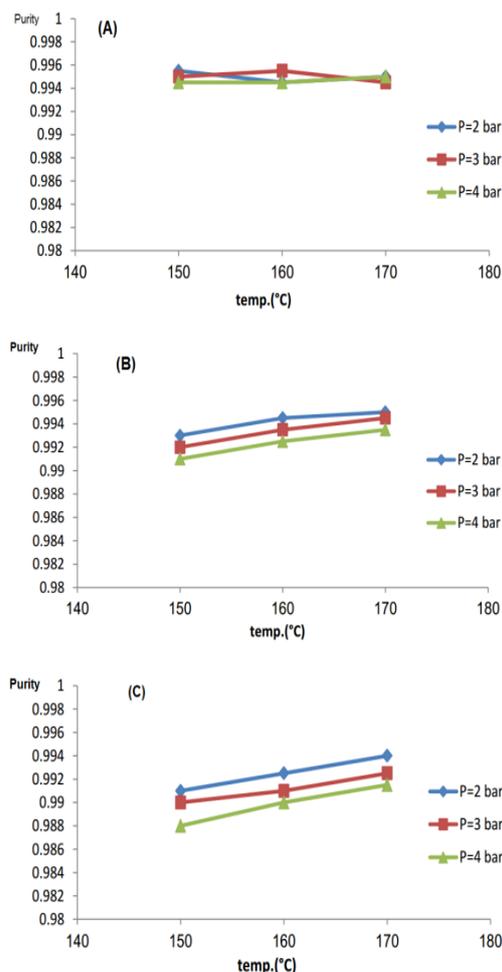
Fig. 4: Temperature and pressure effect on ethanol purity (A: Q=1 l/h, B: Q=2 l/h, C: Q=3 l/h)
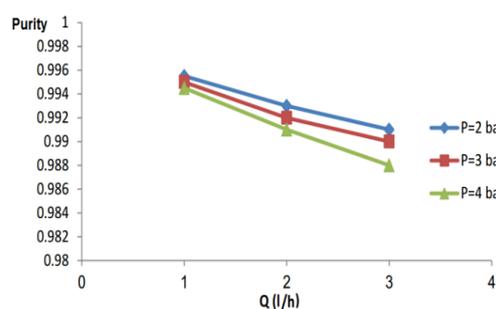

Fig. 5: Effect of feed flow rate on ethanol purity (T=150 °C)

Figure 6 shows the effect of temperature and pressure on ethanol recovery at feed flow rate of 1 l/h. Ethanol recovery decreases with increasing temperature and pressure because ethanol losses during desorption step increases with increasing temperature and pressure. The same trends were noticed at feed flow rate of 2 and 3 l/h.
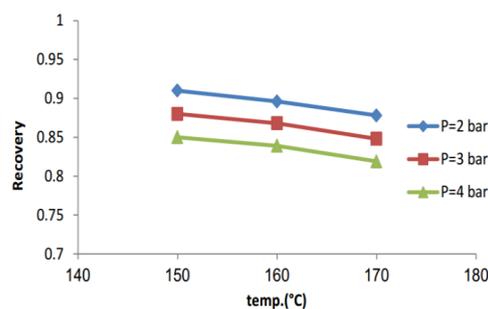

Fig. 6: Temperature and pressure effect on recovery (Q=1 l/hr)

Figure 7 shows the effect of temperature and pressure on the system productivity at different levels of feed flow rate. The productivity decreases with increasing temperature and pressure because ethanol losses during desorption step increases with temperature and pressure increase.
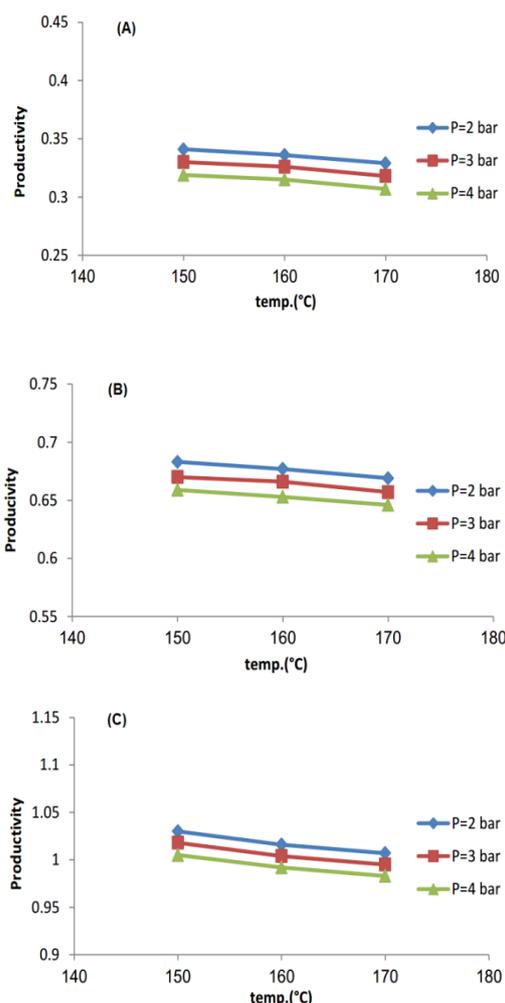

Fig. 7: Temperature and pressure effect on productivity (kg Ethanol/kg Zeolite.hr) [A: Q=1 l/h, B: Q=2 l/h, C: Q=3 l/h]

Figure 8 shows that ethanol recovery slightly increases with increasing the feed flow rate. This is because ethanol losses in the desorption or depressurizing step are not affected by the change of the feed flow rate.
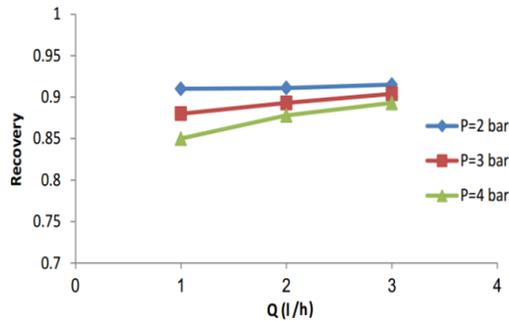

Fig. 8: Effect of feed flow rate and pressure on ethanol recovery (T=150 °C)

Figure 9 shows that the system productivity is directly proportional to the feed flow rate. The relationship of product purity and the productivity is shown in Fig. 10. The product purity decreases with increasing the productivity. This result is in agreement with the published literature [15].
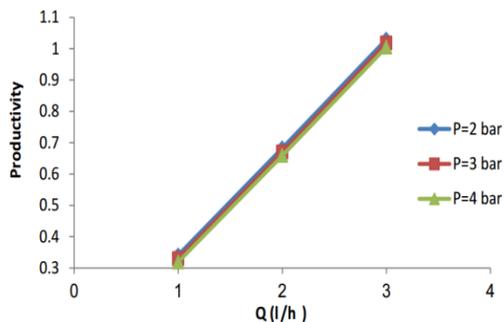

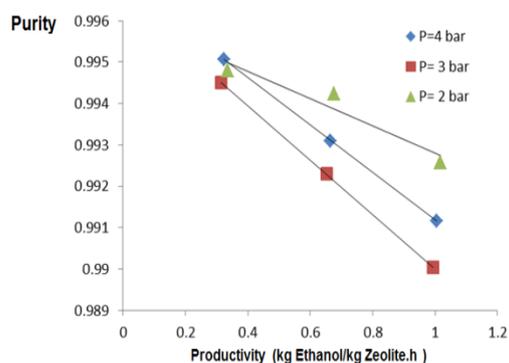Fig. 9: Effect of feed flowrate and pressure on productivity (T=150 °C)

Fig. 10: Purity- Productivity Relationship

Conclusion

1. Pressure Swing Adsorption (PSA) experiments using zeolite 3A shows high performance in ethanol-water separation and produce high purity ethanol of about 99.5 wt%; that can be used as a car fuel.
2. No significance effect of temperature and pressure on ethanol purity at low feed flow rate of 1 l/h, while there was significant effect at high feed flow rate of 2 and 3 l/h. The purity increases with temperature increase. Whereas the purity decreases with pressure increase.
3. Ethanol purity decreases with increasing the feed flow rate.
4. Recovery is slightly changed, with a range of 0.82 to 0.92 for all operating conditions.
5. Productivity is directly proportional to the feed flow rate. It is of a wide range of 0.3 to 1.05 kg ethanol/kg zeolite. h.
6. Ethanol purity decreases with increasing productivity.

Nomenclature

P   Operating Pressure, bar
P_{ads} Operating Adsorption Pressure, bar
P_{des} Operating Desorption Pressure, bar
Q   feed Flowrate, l/h
q   Adsorbent Capacity at Operating conditions, kg water/kg Adsorbent
q_{ads} Adsorbent Capacity at Adsorption conditions, kg water/kg Adsorbent
q_{des} Adsorbent Capacity at Desorption conditions, kg water/kg Adsorbent
T   Operating Temperature, ^{o}C
T_{ads} Operating Adsorption
T_{des} Operating Desorption Temperature, ^{o}C

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