Regeneration of Na-ETS-10 using microwave and conductive heating

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Abstract

Desorption of ethylene/ethane and carbon dioxide/methane mixtures was performed by microwave heating and conductive heating on Na-ETS-10. Gas recovery, Na-ETS-10 regeneration efficiency and swing capacity were compared between the two methods. Na-ETS-10 regeneration occurred within 8 minutes for microwave heating and 22 minutes for conductive heating. For microwave heating the energy consumption was 0.7 kJ/g Na-ETS-10 and the gas recovery was 94% for C_2H_4/C_2H_6 and 70% for CO_2/CH_4. Conductive heating had an energy consumption of 7.7~7.9 kJ/g Na-ETS-10 and resulted in 71% gas recovery for C_2H_4/C_2H_6 and 57% for CO_2/CH_4. The adsorption capacity of Na-ETS-10 did not change over 5 adsorption-regeneration cycles for both heating techniques. Microwave desorption provided a faster heating rate and desorption rate, higher desorption and gas recovery and lower energy consumption compared to conductive heating. Hence, microwave heating can be used as a more efficient and energy saving regeneration technique for Na-ETS-10 for adsorptive separation of binary mixtures such as C_2H_4/C_2H_6 and CO_2/CH_4.

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1. Introduction

High purity ethylene (C_2H_4) is required for the production of polymers, rubber, fibre and various organic chemicals (Kniel et al., 1980). Generally, C_2H_4 is prepared through steam cracking or thermal decomposition of ethane (C_2H_6). The gas product of cracking contains un-cracked C_2H_6. Separation of un-cracked C_2H_6 from C_2H_4 is crucial in the polymer manufacturing production chain (Eldrige et al., 1993). Cryogenic distillation is the most reliable and commonly used technique for C_2H_4/C_2H_6 separation but it is extremely energy intensive (Shi et al., 2011).

Currently, natural gas provides one fourth of the world's energy needs for homes, vehicles and industries (Cavenati et al., 2004). Typically natural gas contains 80-95% methane; the rest is made of C_2^+ hydrocarbons, nitrogen, and carbon-dioxide impurities. High concentration of carbon dioxide in methane can lead to pipeline and equipment corrosion and therefore, reducing it to trace levels is necessary to achieve the pipeline quality methane (no more than 2% CO_2) (Cavenati et al., 2006). Typically the separation of CO_2 is accomplished by chemical absorption with amines which is energy intensive and requires high reagent costs (Rao et al., 2002).

Adsorptive separation is an effective alternative to cryogenic distillation or chemical absorption as it requires less energy and capital cost (Eldrige et al., 1993). Preliminary studies and model predictions suggest that Engelhard Titanosilicate-10 (Na-ETS-10) has great potential as an adsorbent in the separation of C_2H_4/C_2H_6 and CO_2/CH_4 mixtures (Anson et al., 2008, 2009). It has been reported that the adsorption separation of the binary mixture of C_2H_4/C_2H_6 using Na-ETS-10 can achieve a bed selectivity of 5 at ambient pressure and up to 11 at 2580 kPa (Shi et al., 2010, 2011).

ETS-10 is a large pored, mixed octahedral/tetrahedral titanium silicate molecular sieve possessing an inherent three dimensional network of interconnecting channels (Kuznicki et al., 1991; Anderson et al., 1994). The average pore size of ETS-10 has a kinetic diameter of ~8 Å. Hence C_2H_4, C_2H_6, CO_2 and CH_4 can enter the crystalline lattice as the pore size is larger than the molecular diameter of all four species stated (Shi et al., 2010). Therefore, separation selectivity of C_2H_4 over C_2H_6 or CO_2 over CH_4 would be based on the equilibrium competitive adsorption. Na-ETS-10 could preferentially adsorb ethylene in the binary mixture of C_2H_4 and C_2H_6 (Shi et al., 2010) and preferentially adsorb CO_2 in the binary mixture of CH_4 and CO_2 (Anson et al., 2009).

Despite Na-ETS-10's great potential in adsorptive separation of C_2H_4, C_2H_6, CO_2 and CH_4, its regeneration cost presents a challenge because of the high heats of adsorption of the gases to be separated (Shi et al., 2010; Al-Baghli et al., 2005). In this context, microwave heating can be a promising alternative to the conventional pressure swing and temperature swing regeneration methods that are currently used in separation industry (Roussy et al., 1981, 1984). Although microwave heating was initially used for rapid heating of food, its unique selectivity and fast heating rate proved to be useful in other applications such as industrial drying (Tierney et al., 2005). In a conventional thermal regeneration process, the thermal energy is transferred from the surface to the bulk of the material. By contrast in microwave heating the energy is transferred from the inside to the outside of the material as microwaves propagate through molecular interactions between the material and the electromagnetic field (Das et al., 2010).

Microwave heating has been reported for the regeneration of zeolite 13X (Roussy et al., 1981), DAY (Reuβ et al., 2002; Turner et al., 2000), zeolite 3A, 4A, 5A, (Ohgushi et al., 2001), and Na-X and Ca-X (Ohgushi et al., 2003, 2005). A preliminary study of microwave regeneration of Na-

ETS-10 was recently completed using a kitchen microwave and showed that microwave heating is capable of regenerating Na-ETS-10 over several adsorption/desorption cycles (Shi et al., 2010).

Conventional thermal regeneration, known as temperature swing regeneration, is another widely used method for adsorbent regeneration in separation and purification industries. During temperature swing regeneration a hot gas stream or steam is used for bed heating and a cold gas stream is used for bed cooling (Clausse et al., 2004). There have been several reports of using temperature swing to regenerate zeolite 13X (Merel et al., 2006), 4A and 5A (Siriwardane et al., 2005) as well as an extensive review on temperature swing regeneration which can be found elsewhere (Ruthven et, 1984; Suzuki, 1990; Cherbanski et al., 2011).

The objective of this study is to investigate the performance of both conductive heating and microwave heating for the regeneration of Na-ETS-10. Two gas mixtures, ethylene/ethane (C_2H_4/C_2H_6) and carbon dioxide/methane (CO_2/CH_4), commonly used in industry, were separated on Na-ETS-10 in packed bed columns which were later regenerated by microwave heating and conductive heating. The Na-ETS-10 swing capacity, regeneration efficiency and energy consumption were determined and compared between microwave heating and conductive heating. The recovery and purity of the desorbed gases were also determined.

2. Experimental

2.1 Sample preparation

Na-ETS-10 was synthesized using the hydrothermal technique as described elsewhere (Kuznicki, 1991). A typical sample was prepared by thorough mixing of 50 g of sodium silicate (28.8%

SiO_2, 9.14% Na_2O), 3.2 g of sodium hydroxide (97+% NaOH), 3.8 g of anhydrous KF, 4 g of HCl (1M), and 16.3 g of $TiCl_3$ solution. The mixture was stirred in a blender (Waring) for 1h. Then it was transferred to a 125 mL sealed autoclave (PARR instruments) and heated at 215 °C for 64 h. The resultant material was carefully washed with de-ionized water and then dried in an oven at 100 °C. The material was reduced to fine powder (< 150 μm) and pelletized by mixing 6 g of the material (equilibrated at 100 °C) with 2 g of Ludox HS-40 colloidal silica (Aldrich). Morter and pastle were used to homogenize the mixture. Then the mixture was compressed using a pellet press at 10,000 psi for 3 min. The resulting cake was crushed and sieved to acquire 16-20 mesh particles. The prepared pellets were used in the adsorption-desorption experiments.

2.2 Adsorption-desorption experiments

Adsorption-desorption experiments were performed by saturating 10 g of pelletized Na-ETS-10 (16-20 mesh) in a double-ended cylindrical quartz column. The adsorbent bed height was 3.75cm and its diameter was 2.9 cm. The sample was activated at 200 ºC in a laboratory oven for 16 h under 120 mL/min helium gas flow. During adsorption, feed gas flow was maintained at 22 °C and 101.325 kPa~~. Feed gas consisted of either C_2H_4:C_2H_6 mixture (59:41) or CO_2(10:90). The feed gas mixtures were introduced to the fixed bed adsorbent column at a flow rate of 180 mL/min (C_2H_4:C_2H_6) and 300 mL/min (CO_2:CH_4)~~. Feed gas consisted of either 59% C_2H_4/ 41% C_2H_6 mixture or 10% CO_2/ 90% CH_4. The feed gas mixtures were introduced to the fixed bed adsorbent column at a flow rate of 180 mL/min (C_2H_4/C_2H_6) and 300 mL/min (CO_2/CH_4). The feed gases (Praxair) were surrogate mixtures for the process gas streams of ethylene cracking and natural gas purification units. Outlet gas was sampled using 5 mL syringe at 5 minute intervals. Outlet gas composition was analysed using a 5890A Agilent Gas Chromatograph (GC) equipped with thermal conductivity detector (TCD) and a Supelco matrix Haysep Q column (well suited for hydrocarbon analysis). 0.5 mL samples were pulse injected and analysed with the GC-TCD. A continuous flow of feed gas was maintained until the outlet composition became the same as the inlet composition which occurred after approximately 16 minutes for C_2H_4/ C_2H_6 mixture and 90 minutes for CO_2/CH_4 mixture.

The microwave generation and propagation system consisted of a 2 kW switch-mode power supply (SM745G.1, Alter), a 2 kW microwave source (MH2.0W-S, National Electronics) equipped with a 2.45 GHz magnetron, an isolator (National Electronics), a three-stub tuner (National Electronics) and a waveguide applicator connected to a sliding short (IBF Electronic GmbH & Co. KG). The tuner and the sliding short were manually adjusted at the beginning of the experiment to improve the energy transfer to the adsorbent. The isolator was used to protect the microwave head by conducting reflected power into a water load. The power was monitored with a dual directional coupler with 60 db attenuation (Mega Industries), two power sensors (8481A, Agilent) and a dual channel microwave power meter (E4419B, Agilent). The temperature of the material was monitored using a fiber optic temperature sensor and a signal conditioner (Reflex signal conditioner, Neoptix). The temperature sensor, power meter and power supply were connected to a data acquisition and control (DAC) system (Compact DAC, National Instruments) equipped with a Labview program (National Instruments) to record the data and control power application. Labview program was used to monitor and control heating during desorption. After saturation, the microwave generation system was turned on and the heating was initiated using Labview program. ~~The desorbed gas was flown to a downstream flask~~ The desorbed gas flowed to a downstream flask and was collected by water displacement. The volume of the displaced water was equal to the volume of the gas that was collected at the outlet. The desorption experiment was continued until no gas evolution was observed. After desorption, the adsorbent was cooled to room temperature by purging with nitrogen at 120 mL/min. Once the bed reached ambient temperature, further adsorption/microwave desorption cycles were initiated.

In conductive heating technique, a double ended cylindrical steel column with an inner diameter of 1 cm and bed height of 7 cm was used as a reactor. Following saturation of the adsorbent bed, the column was wrapped with a heating tape (Omegalux^{TM}) followed by an additional insulation tape. The heating tape was connected to a 120 V AC power source through a solid state relay interfaced to a DAC system. A Labview program was used to initiate and control the heating. The bed temperature was maintained at 190 ºC. A shielded type K thermocouple (Omega) was used to measure the bed temperature. Data were recorded using a DAC and a Labview program as described in the microwave desorption experiments. Desorbed gas collection system and post desorption adsorbent cooling system were analogous to those used in the microwave desorption experiments. Heating was continued until no gas evolution was observed. A block diagram for adsorption and regeneration by microwave heating and conductive heating process is illustrated in Figure 1.

Swing capacity is generally defined as the adsorption capacity or working capacity of an adsorbent between two extreme states of the swing force (Anson et al., 2009). In this work, swing capacity of Na-ETS-10 is defined as the amount of gas desorbed during heating from 22 °C to 190 °C. The maximum swing capacity was achieved by water desorption (Shi et al., 2011). Gas recovery was calculated based on the Equation 1.

~~$Gas\ recovery\ (\%) = \frac{V_{M/C}}{V_W}\ x\ 100$ (1)~~

~~Where, $V_{M/C}$ = volume of gas desorbed by microwave (M) or conductive (C) heating and V_W = the volume of gas desorbed by water desorption.~~

$$Gas\ recovery\ (\%) = \frac{V_{M/C}}{V_W} \times 100\% \qquad (1)$$

Where, $V_{M/C}$ is volume of gas desorbed by microwave (M) or conductive (C) heating and V_W is the volume of gas desorbed by water desorption which is equal to the adsorption capacity of the adsorbent.

3. Results and discussion

3.1 Ethylene/Ethane (C_2H_4/C_2H_6) desorption from Na-ETS-10

Desorption achieved by water desorption is considered as complete (100%) through the mass action displacement mechanism (Shi et al., 2010). Therefore, the saturated Na-ETS-10 was flushed with water and the desorbed gas was collected in a gas collection container. Desorption started immediately after water injection and lasted for 7-8 minutes. A total of 320 mL gas was collected from approximately 10 g of Na-ETS-10 through water desorption; therefore the maximum adsorption capacity is 30 mL/g Na-ETS-10 or 1.24 mmol/g Na-ETS-10. Based on GC-TCD analysis, the desorbed gas consisted of 88% C_2H_4 and 12% C_2H_6 which has been reported elsewhere (Shi et al., 2010).

A comparison of the temperature profiles for microwave heating and conductive heating is provided in Figure 2(a). The temperature profile of microwave heating shows a steep heating rate of 64 °C/min compared to only 13 °C/min for conductive heating. The difference in heating duration is because heating was stopped when gas evolution stopped.

The two heating techniques were also compared by power consumption as function of temperature in Figure 2(b). During microwave heating, power consumption fluctuates between 0-25 W before it stabilizes around 12 W, while temperature becomes stable around 190 ºC. During conductive heating, power consumption fluctuates between 0 and 112 W and finally stabilizes around 50 W, which is four times higher than that of microwave heating.

The comparison of desorption rates of adsorbed C_2H_4/C_2H_6 during microwave heating and conductive heating is shown in Figure 2(c). Although net power requirement is higher for conductive heating, the desorption rate is higher for microwave heating. During microwave regeneration, desorption starts immediately and reaches a maximum rate of 79 ml/min (3.25 mmol/min) within one minute. The rate decreases to 3 mL/min as the temperature stabilizes at 190 ºC. In conductive heating on the other hand, desorption starts within the first minute and reaches a maximum rate of 20 mL/min (0.82 mmol/min) during the second minute of heating and maintains it up to the tenth minute. Then the rate decreases as the power decreases until the temperature stabilizes at 190 ºC at which point the rate remains at 1 mL/min. Figure 2 illustrates that microwave heating performs better and quicker than conductive heating in terms of heating rate, net energy consumption and gas desorption rate for adsorptive separation of C_2H_4/C_2H_6.

The microwave desorption took 8 minutes and 28 mL gas was recovered from 1 gram of Na-ETS-10 (1.16 mmol/g). Based on GC-TCD analysis, the desorbed gas contained 87% C_2H_4 and 13% C_2H_6, which is consistent with adsorbed phase composition data reported elsewhere (Shi et al., 2010). When conductive heating was applied to regenerate the Na-ETS-10 saturated with the C_2H_4/C_2H_6 mixture, it took 22 minutes to evolve 21mL/g Na-ETS-10 of gas (0.87 mmol/g).

A total of five adsorption/desorption cycles for the C_2H_4/C_2H_6 mixture were completed on Na-ETS-10 for both microwave and conductive heating. No mass loss of the adsorbent was observed after each adsorption-desorption cycles, and the refreshed adsorbent bed has the same weight as the starting adsorbent. A comparison of microwave heating and conductive heating techniques over these five cycles is presented in Figure 3 and Table 1. The swing capacity of Na-ETS-10 during microwave heating and conductive heating was stable; 1.16 mmol/g Na-ETS-10 and 0.87 mmol/g Na-ETS-10 respectively over five cycles of adsorption/desorption (Figure 3). The results indicate that swing capacity of microwave heating is 1.33 times larger than that of conductive heating. The swing capacity also indicates that the adsorption capacity of Na-ETS-10 is not influenced by successive microwave/conductive heating cycles.

Table 1 shows that on average 94% of the adsorbed gas was recovered with microwave desorption while only 71% was recovered with conductive heating. However, with both techniques, the adsorption capacity remained steady over repeated adsorption-regeneration cycles. In microwave desorption, an average net energy of 0.73 kJ/g was consumed to achieve such desorption, however, approximately 7.9 kJ/g was consumed in the case of conductive heating.

On average, 25 J microwave energy and 370 J conductive energy was needed to desorb 1mL of the adsorbed gas (mixture of ethylene/ethane) in each of the five cycles performed (Figure 4). While both systems display steady energy consumption during the five cycles of adsorption and desorption, the conductive heating requires 14.8 times more energy than microwave heating to desorb the same volume of gas. In the conductive heating experiments, the reactor was heated first and then the energy was transferred to the adsorbent through conductive heating. However, in microwave heating, the energy is transferred from the inside to the outside of the material as microwaves propagate through molecular interactions between the material and the electromagnetic field (Das et al., 2009). Hence, more energy loss occurred during the conductive heating, which explains why microwave heating is faster and consumes less energy.

Desorbed gas composition of each cycle was analyzed by GC-TCD which was presented in Table 2. It shows that 87~87.5% C_2H_4 and 12.5~13% C_2H_6 could be obtained during the microwave desorption and 85~85.5% C_2H_4 and 14.5~15% C_2H_6 could be obtained during the conductive heating. Both methods gave the similar desorbed gas composition as adsorbed phase gas.

3.2 Carbon dioxide/methane (CO_2/CH_4) desorption from Na-ETS-10

Complete (100%) desorption of CO_2/CH_4 from Na-ETS-10 was obtained by water desorption, generating a total of 407 mL of gas from 10 g of Na-ETS-10, indicating a maximum desorption capacity of 39 mL/g. Based on the GC-TCD analysis, the desorbed gas contained 89% CO_2 and 11% CH_4.

Comparisons of temperature profile, power consumption profile and desorption rate of adsorbed CO_2/CH_4 for both methods are shown in Figure 5. For microwave heating, power consumption fluctuated between 0-20 W and stabilized around 12 W while temperature stabilized at 190 ºC. For conductive heating power consumption fluctuated between 0-101 W and stabilized around 44 W. Desorption rate for conductive heating is slower than for microwave heating and also net power requirement is higher. Desorption rate during microwave heating reached a maximum of 100 mL/min in the first minute then decreased reaching close to zero at the eighth minute. During conductive heating, the desorption rate reached a maximum of 26 mL/min in the seventh minute, remained constant up to the tenth minute and then decreased and stabilized at 1 mL/min at twenty second minute of heating time. Figure 5 illustrates that microwave heating is more efficient and faster than conductive heating in terms of heating rate, net energy consumption and gas desorption rate for adsorptive separation of CO_2/CH_4.

Microwave heating was successful in desorbing CO_2/CH_4 mixture from Na-ETS-10. 27 mL of desorbed gas per gram of Na-ETS-10 was recovered after 8 minutes of microwave heating. The desorbed gas consisted of 82% CO_2 and 18% CH_4 as determined by GC-TCD analysis. After heating, the bed was cooled under N_2 flow at 120 mL/min. Regeneration of CO_2/CH_4 saturated Na-ETS-10 with conductive heating took 22 minutes to evolve 22 mL/g of gas.

A total of five adsorption/desorption cycles for the CO_2/CH_4 mixture were completed on Na-ETS-10 for both microwave and conductive heating. A comparison of microwave heating and conductive heating over 5 cycles is presented in Figure 6 and Table ~~2~~3.

Based on the gas being recovered, the swing capacity of Na-ETS-10 over 5 adsorption-desorption cycles during microwave heating and conductive heating were stable around 1.10 mmol/g Na-ETS-10 and 0.91 mmol/g Na-ETS-10 (Figure 6). Figure 6 illustrates that the adsorption capacity of Na-ETS-10 was unchanged during both microwave heating and conductive heating. The results also indicate that swing capacity of microwave is 1.21 times larger than that of conductive heating.

Table ~~2~~3 shows that 70% of the adsorbed CO_2/CH_4 was recovered by microwave heating while only 57% by conductive heating. In microwave desorption, an average net energy of 0.67 kJ/g was consumed to achieve such desorption, however, approximately 7.7 kJ/g was consumed in the case of conductive heating.

On average 25 J of microwave energy and 348 J of conductive energy are needed to release 1 mL of gas adsorbed on Na-ETS-10. Throughout the five adsorption-regeneration cycles, conductive heating requires 14 times more energy than microwave heating in order to desorb the same volume of gas. The higher energy requirement in conductive heating is due to high heat loss as discussed in section 3.1. Figure 7 illustrates the consistency in energy consumption over 5 cycles of CO_2/CH_4 desorption for microwave heating and conductive heating.

Table 4 summarizes the purity of the recovered CO_2/CH_4 gas for these two heating techniques over five cycles of adsorption/desorption. Based on GC-TCD analysis, the purity of the gas desorbed by microwave heating consisted of 82~83% CO_2 and 17~18% CH_4 while the purity of the gas desorbed by conductive heating contained 81~81.8 % CO_2 and 18~19% CH_4.

Comparing these two different binary systems (C_2H_4/C_2H_6, CO_2/CH_4), the recovery percentage of C_2H_4/C_2H_6 was higher than CO_2/CH_4. In C_2H_4/C_2H_6 separation system, the adsorbed phase is highly enriched C_2H_4 which has a polarizability of 42.52×10^{25} cm^3, while in CO_2/CH_4 separation system, the adsorbed phase is highly enriched CO_2 which has a polarizability of 29.11×10^{25} cm^3 (Li et al., 2009). Considering in the case of physical adsorption, the adsorbed phase is in a liquid-like phase (Myers et al., 1965), so the adsorbed C_2H_4 consumed the microwave more efficiently than CO_2. By supplying the same amount of microwave energy, a higher recovery rate could be obtained in C_2H_4/C_2H_6 separation system.

4. Conclusion

In this work, two binary gas mixtures C_2H_4/C_2H_6 (59:41) and CO_2/CH_4 (10:90) were separated by adsorption on Na-ETS-10 at 22 °C and 101.325 kPa. Na-ETS-10 was regenerated using microwave and conductive heating desorption and the desorbed gas was collected. Results show that microwave desorption can regenerate Na-ETS-10 more efficiently than conventional temperature swing regeneration such as conductive heating. Swing capacity achieved in microwave heating is higher than that of conductive heating. For both heating techniques swing capacity is not affected by successive heating cycles. During microwave desorption, 94% of the adsorbed C_2H_4/C_2H_6 and 71% of the adsorbed CO_2/CH_4 mixture were recovered. On the other hand, during desorption with conductive heating, 71.4% C_2H_4/C_2H_6 and 57.2% CO_2/CH_4 were recovered. Microwave desorption required an average of 0.7 kJ/g Na-ETS-10 during 8 minutes of heating while conductive heating required 7.7~7.9 kJ/g Na-ETS-10 during 22 minutes of heating. Results show that microwave desorption is characterized by faster heating, higher desorption rate, and lower energy consumption compared to desorption with conductive heating. Therefore, microwave heating can potentially be used as a cheaper energy source to regenerate Na-ETS-10 for adsorptive separation of binary gas mixtures such as C_2H_4/C_2H_6 and $CO_2/CH_{4.}$

The regeneration results can be further improved by using a sweep gas that can purge the adsorbent bed during heating. Using steam as purge gas can be a practical approach to enhance the heating both during microwave heating and conductive heating. Another approach can be using previously recovered C_2H_4 / CO_2 to ensure purging without diluting the product gas. It is expected that using C_2H_4 / CO_2 as purge gas would speed up the desorption process and would improve heating and therefore, requires further investigation.

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