# INDIRECT AND DIRECT MICROWAVE REGENERATION OF Na-ETS-10

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**Abstract**

Indirect and direct microwave regeneration were assessed as potential techniques for desorbing a CO2/CH4 mixture from Na-ETS-10. Indirect microwave regeneration consists of desorption with water followed by microwave drying, while direct microwave regeneration consists of constant power microwave heating. Five adsorption-desorption cycles were completed to determine and compare the swing capacity, net energy consumption, and gas recovery obtained with these two regeneration techniques. During indirect microwave regeneration the swing capacity of Na-ETS-10 was stable at 0.3 mmol/g, with the exception of the first cycle where it reached 1.6 mmol/g. The first cycle swing capacity decreased however, by five fold during the subsequent desorption cycles. During direct microwave regeneration, the swing capacity remained stable at 0.7 mmol/g over all five consecutive cycles. On average, 2.46 KJ and 0.32 KJ were consumed during regeneration of one gram of Na-ETS-10 using indirect and direct microwave regeneration, respectively. With the exception of the first cycle, gas recovery for indirect microwave regeneration was only 20%. In contrast, an average of 50% gas recovery was achieved by direct microwave regeneration.

**Keywords:**  Microwave; Water; Desorption; Separations; Zeolites; Na-ETS-10

## 1. Introduction

Microwave heating is an emerging technology in the chemical process industries (Bykov et al., 2001) with the advantages of being cheaper and faster than conventional adsorbent regeneration techniques such as steam regeneration (Polaert et al., 2010). The fundamentals of microwave regeneration are unique and opposite to the mechanism of conventional thermal regeneration techniques (Das et al., 2009). In steam regeneration, the thermal energy is transferred from the surface to the bulk of the adsorbent bed. By contrast, the thermal energy in microwave regeneration is transferred from the inside to the outside of the adsorbent bed (Hashisho et al., 2005).

Finding an efficient method for regeneration of the adsorbents is important for chemical industries because it is a time-consuming process with high-energy requirements. In this context, microwave regeneration was shown to be successful in dehydrating and regenerating adsorbents saturated with volatile organic compounds (VOCs) (Roussy and Chenot, 1981; Cha and Carlisle, 2001; Hashisho et al., 2005). Dehydration of adsorbents using microwave heating was studied for zeolite 13X (Roussy and Chenot, 1981), zeolite 3A, 4A, 5A, (Ohgushi et al., 2001), Na-X, and Ca-X (Ohgushi and Nagae, 2003, 2005). Constant power microwave heating has also been used to regenerate dealuminated Y zeolite (DAY) (Reuβ et al., 2002; Turner et al., 2000), silicate(Meier, 2009), mordinate (Kim et al., 2005), faujasite (FAU) (Kim et al., 2007) and Engelhard titanosilicate (ETS-10) (Shi et al., 2010).

Natural gas is considered to be a cleaner alternative to other fuels and its supply currently meets one-fourth of the world’s energy needs. Typically, natural gas contains traces of impurities such as carbon monoxide, carbon dioxide, and nitrogen. In Australia and Germany, natural gas contains more than 10% carbon dioxide (CO2) impurity (Caventi et al., 2004). This percentage needs to be reduced to meet the ‘pipeline quality’ (< 2% CO2 impurity) set for methane (CH4) in order to protect equipment and pipeline infrastructures (Cavenati et al., 2004). ETS-10, a titanosilicate molecular sieve with pores large enough to accommodate CO2 and lighter hydrocarbons, is shown to purify CH4 by preferentially adsorbing CO2 over CH4. (Kuznicki, 1991; Anderson et al., 1994). ETS-10 can preferentially adsorb CO2 over C2H6 as well. (Anson et al., 2009). However like with adsorbents there is a need to efficiently regenerate and reuse ETS-10. Previous studies on regeneration of Na-ETS-10 with steam, conductive heating, and microwave heating techniques have shown that microwave regeneration is faster and more energy efficient than the other two techniques (Shi et al., 2010; Chowdhury et al., 2012). These studies reported that Na-ETS-10 provides maximum water desorption by mass action displacement but the energy requirement for drying wet Na-ETS-10 has yet to be determined (Shi et al, 2010, Chowdhury et al., 2012).

This study investigates an indirect method for regenerating Na-ETS-10 (water desorption followed by microwave drying) and compares it to direct microwave regeneration. In direct microwave regeneration CO2/CH4 mixture was adsorbed on a packed bed of Na-ETS-10 and then desorbed by water injection. The wet adsorbent was then dried and reactivated using microwave heating. In direct microwave regeneration, CO2/CH4 gas mixture was adsorbed on a packed bed of Na-ETS-10, and desorbed using constant power microwave heating. The swing capacity, gas recovery, and energy consumption achieved over five adsorption-desorption cycles were measured and compared between indirect and direct microwave regeneration.

## 2. Experimental

Na-ETS-10 was synthesized using the hydrothermal technique as described elsewhere (Kuznicki, 1991). Powder Na-ETS-10 was pelletized into 16-20 mesh pellets. A detailed method of pellet preparation can be found elsewhere (Shi et al., 2010).

Adsorption-desorption experiments were performed using an adsorbent bed 3.75cm long and 2.9cm in diameter containing 10g of Na-ETS-10 and a double ended cylindrical quartz column. Activation of the sample was completed at 200°C in a laboratory oven for 16h under 120ml/min helium gas flow. The feed gas mixture (Praxair) of 10%CO2 and 90%CH4 was introduced into the fixed bed column with a flow rate of 300 mL/min at 22°C and 101.325 kPa. The outlet gas was sampled and analysed by using a gas chromatograph (Agilent 5890) equipped with a thermal conductivity detector and Supelco matrix Heysep Q column. Adsorption proceeded under a continuous flow of feed gas until saturation when the outlet composition became the same as the feed composition. Na-ETS-10 became saturated with CO2/CH4 after 90 minutes.

In the indirect microwave regeneration, 5ml water was injected into the saturated adsorbent. The desorbed gas flowed to a downstream flask and was collected by water displacement. The experiment was carried out until no gas evolution was observed. The volume of the displaced water was equal to the volume of the gas that was collected at the outlet. After desorption with water, a microwave generation and propagation system was used to dry the adsorbent. The system consisted of a 2 kW switch-mode power supply (SM745G.1, Alter), a 2 kW variable output microwave generator (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). At the beginning of the experiment the tuner and the sliding short were manually adjusted for matching the impedance and improving the energy transfer to the adsorbent. To determine energy consumption, power was monitored using a dual channel microwave power meter (E4419B, Agilent), two power sensors (8481A, Agilent), and a dual directional coupler with 60 db attenuation (Mega Industries). To avoid interference with the electromagnetic field, a fiber optic temperature sensor with a signal conditioner (Reflex signal conditioner, Neoptix) was used to monitor the temperature of the adsorbent during microwave heating. A data acquisition and control (DAC) system (Compact DAC, National Instruments) equipped with a Labview program (National Instruments) was used to record the signals from the temperature sensor, and power meter and control the output of the power supply. The Labview program was used to monitor and control the heating. During the microwave drying, a 120 sccm nitrogen flow was used as purge gas to provide uniform heating. After microwave drying, the nitrogen flow was adjusted to 300 sccm for faster cooling of the bed to room temperature. Once the bed reached room temperature, further adsorption-water desorption-microwave drying cycles were initiated. A block diagram showing adsorption and regeneration by water desorption followed by microwave drying is presented in Figure 1.

The same microwave generation system was used for both indirect and direct microwave regenerations. After saturation of the adsorbent, the microwave generation system was turned on, and the heating was initiated using the Labview program. The adsorbent was exposed to a constant incident microwave power of 60W until the bed temperature reached its set-point, then the heating was stopped. During the regeneration, the desorbed gas flowed to a downstream flask and was collected by water displacement. Microwave regeneration was continued until no gas evolution was observed. Then, the adsorbent was cooled to room temperature by purging with nitrogen at 120 sccm. After the bed reached ambient temperature, further adsorption-microwave desorption cycles were initiated. A block diagram showing adsorption and constant power microwave regeneration is presented in Figure 2.

For water desorption, the swing capacity of Na-ETS-10 is defined as the amount of gas desorbed during water injection. For microwave regeneration, the swing capacity is defined as the amount of gas desorbed during microwave heating. Gas recovery was calculated based on equation 1:

Gas recovery (%) = (VW/M/VW ) x100 ..……….. (1)

where, VW/M = volume of gas desorbed by water desorption (W) or microwave (M) heating, and VW= the volume of gas desorbed by water desorption from the fresh adsorbent.



Figure 1: Block diagram showing adsorption and indirect microwave regeneration of Na-ETS-10

Figure 2: Block diagram showing adsorption and direct microwave regeneration of Na-ETS-10

## 3. Results and Discussion

### 3.1 Indirect microwave regeneration

Water desorption achieves complete (100 %) desorption through mass action displacement (Shi et al., 2010) within 7-8 minutes. A total of 410 ml gas was collected from approximately 10g of Na-ETS-10. Based on the GC-TCD analysis, the desorbed gas contained 89% CO2 and 11% CH4. Later, the wet Na-ETS-10 was dried by using microwave heating.

The wet Na-ETS-10 was dried by microwave heating for 20 minutes at 190°C. A total of 2,294 J microwave energy was consumed per gram of Na-ETS-10 and 20% of the adsorption capacity was restored. Microwave drying restored 20% of the gas adsorption capacity. Under these operating conditions, further drying required more energy and heating-time. Based on the amount of recovered gas, the swing capacity of Na-ETS-10 during water desorption was 1.58 mmol/g.

Typical temperature and power profiles during microwave drying as a function of time are shown in Figure 3. These temperature and power profile can be divided into four zones. In zone I, the temperature increased, but no desorption was observed. The net power consumption increased slightly at this stage. In zone II, continuous desorption occurred and the temperature showed slow change. The power consumption rapidly increased to a steady level. In zone III, the temperature sharply increased until the set-point was reached. At this stage, the energy consumption is mainly attributed to adsorbent heating. Therefore, both the energy requirement for heating and the power consumption decreased due to the precise control of the PID controller. Finally in zone IV, the temperature stabilized at the set-point value. At this stage, the heat gain and the heat loss became equal, and very little desorption was observed. The drying behaviour of Na-ETS-10 was consistent with reports in literature (Polaert et al., 2007, 2010).

Figure 3: Microwave drying following desorption with water of Na-ETS-10 saturated with CO2/CH4: temperature and power profile.

The higher the microwave frequency, the larger the dielectric loss of water, resulting in more microwave power to be absorbed. In this experiment a microwave frequency of 2.45 GHz was used. This is much lower than the 20GHz where the maximum dielectric loss of water is obtained, but offers the optimum heating of water since the microwaves’ penetration depth decreases with the increase in frequency (Michael et al., 1991).

Table 1 compares the adsorption capacities for H2O, CO2, and CH4 on Na-ETS-10 and classical zeolites (NaX, NaY) as well as the normalised energy consumption for drying the adsorbents using microwave regeneration. At similar dehydration levels (81 to 100%) the energy consumed (normalized by gram of the adsorbent) for Na-ETS-10 was considerably lower than for NaX and NaY (16% of NaX and 32% of NaY). The consumed energy for Na-ETS-10 (normalized by gram of the water desorbed) was equal to 35% of NaX and 45% of NaY. Although the adsorption capacity of Na-ETS-10 for water vapor was equal to 67% and 52% of NaY and NaX, it was easier to dry. The adsorption capacity of Na-ETS-10 for CH4 is comparable to that of NaX and NaY. However, its adsorption capacity for CO2 is equal to 40% of the adsorption capacities of either NaX or NaY.

Table 1: Comparison of adsorption and microwave drying properties of Na-X, Na-Y and Na-ETS-10 at laboratory scale

|  |  |  |
| --- | --- | --- |
|  | Microwave drying of adsorbent | Adsorption capacity  |
| Adsorbent | Energy consumption (KJ/g desorbed water) | Energy consumption (KJ/g adsorbent) | Dehydration (%) | Water (mmol/g) | CO2 (mmol/g) | CH4 (mmol/g) |
| Na-Xa | 53.8 | 14.5 | ~100 | 15.0a | 5.0b | 0.9c |
| Na-Ya | 41.1 | 7.1 | ~81 | 11.7a | 5.2 b | 0.7 c |
| Na-ETS-10 | 18.7 | 2.3 | 88 | 7.8 | 2.0d | 0.8 d |

a Polaert et al., 2010.

b Walton et. al., 2006

c Choudhary et. al., 1995

d Anson et. al., 2009

### 3.2 Direct microwave regeneration

In direct microwave regeneration, 10 g of saturated Na-ETS-10 was heated under 60 W of constant incident power. The heating started at 22 ºC and continued until the adsorbent bed reached a set-point of 190 ºC. The bed took 110 sec to reach 190 ºC. Constant power microwave regeneration required 320 J per gram of Na-ETS-10. A total of 6.7 mmol gas was desorbed, which represents 50% of the gas that had been adsorbed.

Figure 4 illustrates the temperature, power and desorption rate profiles as a function of time under constant power microwave heating. The adsorbent temperature linearly increased with a heating rate of 1.8ºC/sec while the net power consumption was constant at 35W. The desorption rate was 4.1-1.6 ml/sec. As long as adequate power was available to provide a thermal gradient, the desorption continued until it reached completion. The desorption rate therefore, depends on the absorbed power density (W/m3 bed). Similar findings have been reported for other zeolites (Polaert et al., 2007).

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| --- |
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Figure 4: Desorption of CO2/CH4 and regeneration of Na-ETS-10 with constant power microwave regeneration; (a) temperature and net power profile and (b) desorption rate

### 3.3 Discussion

Five adsorption-desorption cycles were successively completed in order to regenerate Na-ETS-10. A comparison of the gas recovery and energy consumption during the five adsorption-desorption cycles for indirect and direct microwave regeneration is shown in Figures 5 and 6 and Table 2. During water desorption a higher gas recovery was achieved only in the first cycle ~~only~~, while during constant power this was true over the 5 cycles. The swing capacity for direct microwave regeneration was higher than that for indirect microwave regeneration. During cycles 2-5 in water desorption, water was not totally desorbed from Na-ETS-10 after 20 minutes of microwave drying time. As a result, there was less CO2/CH4 mixture adsorbed in the 2nd and subsequent adsorption cycles, which led to a lower gas recovery. A longer microwave drying time could enhance water desorption and increase the swing capacity of the adsorbent increasing the gas recovery for cycles 2 to 5.

Indirect microwave regeneration was more energy intensive than direct microwave regeneration (Figure 6, Table 2). On average, 2,463 J microwave energy was needed to regenerate 1g of Na-ETS-10 in indirect microwave regeneration while direct microwave regeneration consumed 324 J per gram of Na-ETS-10 regenerated over five cycles of adsorption-desorption. Indirect and direct microwave regeneration required 694 J and 420 J per mmol of the desorbed CO2/CH4 mixture. Indirect microwave regeneration consumed more energy than direct microwave regeneration because Na-ETS-10 adsorbed more water than the CO2/CH4 mixture. The adsorption capacity of Na-ETS-10 for water is 0.14 g/g (7.8 mmol/g) while it is 0.09 g/g (2.0 mmol/g) and 0.01 g/g (0.8 mmol/g) for CO2 and CH4, respectively. If normalised by the number of moles of desorbed adsorbate, the energy consumed for drying ETS-10 (295 J/mmol desorbed water) is comparable to the energy consumed for desorbing CO2/CH4 from ETS-10 (419 J/mmol) through direct microwave regeneration.

Figure 5: Variation in gas recovery (%) over 5 cycles during water desorption and direct microwave heating of CO2/CH4 on Na-ETS-10.

Figure 6: Energy consumption normalised by number of mmoles desorbed or adsorbed during direct microwave regeneration was significantly lower than in indirect microwave regeneration of Na-ETS-10

Table 2: Comparison of indirect and direct microwave regeneration for desorbing CO2/CH4 from Na-ETS-10 over five cycles.

|  |  |  |
| --- | --- | --- |
| Indirect microwave regeneration (water desorption-microwave drying) | Cycles | Average and standard deviation over 5 cycles |
| 1 | 2 | 3 | 4 | 5 |
| Swing capacity (mmol/g) | 1.58 | 0.29 | 0.33 | 0.28 | 0.32 | 0.56 ±0.57 |
| Gas recovery (%) | 100 | 18 | 21 | 18 | 20 | 35±36 |
| Energy consumed per gram adsorbent regenerated (J/g) | 2,294 | 2,565 | 2,550 | 2,494 | 2,420 | 2,465±111 |
| Energy consumed per mole gas desorbed (J/mmol) | 1,453 | 8,903 | 7,691 | 9,030 | 7,650 | 6,945±3,138 |
| **Direct microwave regeneration (constant power microwave desorption)** | **Cycles** | **Average and standard deviation over 5 cycles** |
| 1 | 2 | 3 | 4 | 5 |
| Swing capacity (mmol/g) | 0.64 | 0.69 | 0.78 | 0.75 | 0.72 | 0.72 ±0.05 |
| Gas recovery (%) | 40 | 44 | 55 | 48 | 46 | 47±6 |
| Energy consumed per gram adsorbent regenerated (J/g) | 300 | 309 | 368 | 332 | 313 | 324±27 |
| Energy consumed per mole gas desorbed (J/mmol) | 387 | 399 | 476 | 428 | 404 | 419±35 |

The swing capacity, gas recovery, and net energy consumption over five cycles of adsorption and desorption remained relatively constant for direct microwave regeneration. Hence, the repetitive microwave heating did not affect the adsorption capacity of Na-ETS-10 which supports the previous findings (Shi et al, 2010, Chowdhury et al, 2012).

Water has higher adsorption strength than CO2 and CH4 (Li et al., 2009). In addition, since the amount of water adsorbed is higher than the amount of gas CO2/CH4 adsorbed, higher microwave energy is required to reactivate the adsorbent in water desorption. Microwave power can induce dipole moments into adsorbates that are typically non-polar, have low adsorptive strength but carry quadrupole moments. CO2 is quadrupolar and therefore can introduce polar behaviour into the desorption experiment. This issue requires further investigation (Li et al., 2009; Maryott and Birnbaum, 1962).

## 4. Conclusions

In this work, indirect microwave regeneration (water desorption followed by microwave drying) was studied and compared to direct microwave regeneration (constant power microwave regeneration) of Na-ETS-10. Based on five adsorption-regeneration cycles, direct microwave regeneration achieved on average 22 % higher swing capacity, and 24 % higher gas recovery compared to indirect microwave regeneration. The energy consumption per mole of gas desorbed was on average 16.6 times larger for indirect microwave regeneration compared to direct microwave regeneration. Indirect microwave regeneration was found to be energy-intensive most likely due to the high adsorption capacity and heat of adsorption of water. The results from this study are important because they provide a quantitative assessment of the performance of potential techniques for regeneration of Na-ETS-10.

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