Microwave Regeneration of Zeolites in a 1 Meter Column

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Although sorption is a common method of removing volatile organic compounds (VOC's) from processes, the adsorbent must be regenerated to repeat the process. The use of microwaves to regenerate the bed of adsorbent can be more efficient than conventional heating methods. Desorption of methanol from a silicalite zeolite was studied by the use of microwaves in cylindrical column. Temperature probes at five axial and three radial positions monitored the temperature profile in time. A significant amount of microwave energy passed through the dry zeolite bed, whereas a strong attenuation occurs if methanol is adsorbed. The radial temperature distribution conforms to theoretical predictions; however, microwave reflections cause a distortion of the predictions in the axial direction. Irregularities during the saturation of the bed indicated a chemical reaction during the irradiation with microwaves. A fraction of the methanol reacted under microwave irradiation to form dimethylether and water, and to methoxylate the zeolite surface. © 2009 American Institute of Chemical Engineers *AIChE J,* 55: 1906–1913, 2009

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Introduction

Sorption is central to many industrial processes. Many manufacturing industries employ organic chemicals as solvents, cleaners, or reactants. To clear these pollutants from waste gas, adsorption of these VOC's (volatile organic compounds) on zeolites (molecular sieves) or on active carbons is widely used. The adsorbed species can be recycled and reused by heating the adsorbate to induce desorption. In a conventional desorption process, the energy used for desorption is introduced into the system via the gas phase or through the walls, and is distributed by heat transfer (convection and conduction). This is an indirect way of heating, since the entire system (the gas phase as well as the adsorbent bulk phase) has to be heated to the desorption regeneration temperature.

Microwaves have the unique ability to heat materials selectively, depending on their dielectric properties. This makes microwaves interesting for use in sorption processes. With microwaves, energy can be delivered more efficiently to the actual sorption site, i.e., to an adsorbate on the surface of the adsorbent, provided that the adsorbate and/or the adsorbent surface can absorb microwave energy more efficiently than the adsorbent bulk phase.[1]

The use of microwaves in desorption processes is not yet commonly practiced. One of the documented applications is found in U.S. Patent 4,322,394.[2] It describes a process of rapid regeneration of noncarbon adsorbents by microwave energy.

Scaling up small-scale experiments to industrial scale is not necessarily straightforward when microwaves are involved. At a larger scale, microwave characteristics, like a

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varying field intensity and penetration depth, become important. Mehdizadeh[3] discusses problems that have to be considered in scaling up processes involving microwave-induced reactions. This indicates a need to study the behavior of microwave propagation combined with desorption in larger scale systems.

This study employs a pilot-scale cylindrical column (7.62 cm by 1 m) for desorption processes. It is intended to provide a basis to characterize the microwave propagation and sorption processes in a larger-scale system. A zeolite adsorbent was employed such that the adsorbent only weakly absorbs microwave energy, in contrast to activated carbon adsorbents.

This study will examine the instantaneous and transitory behavior of the temperature and regeneration of the column, and the reproducibility of adsorption. This includes: (1) how microwave energy propagates through a weakly absorbing bed, (2) how the absorptivity of microwaves change as desorption proceeds, and (3) how the reproducibility of adsorption changes depending on the component previously adsorbed in the column.

Background

Microwaves are electromagnetic waves in a given frequency range (0.3–300 GHz). The interaction between electromagnetic waves and matter is quantified by a material property called the complex dielectric permittivity ε:

$$\varepsilon = \varepsilon' - i\varepsilon''$$

The real part of the permittivity, ε', expresses the polarization of the medium in the electric field. The conversion of electromagnetic energy into heat depends on the imaginary part of the permittivity, ε''.[4] The permittivity is a function of frequency and temperature. In these experiments, a microwave frequency of 2.45 GHz was used.

The characteristic penetration depth of a medium is defined as the distance at which the field intensity decreases to $1/e$ of its incident value. Buffler[5] gives the following equation to calculate the penetration depth based on the complex permittivity:

$$d_p = \frac{\lambda_0}{\sqrt{2\pi}} \frac{1}{\sqrt{\varepsilon' \left[\sqrt{1 + \left(\frac{\varepsilon''}{\varepsilon'}\right)^2} - 1 \right]}}$$

Heating under microwave irradiation is different from conventional heating. Zeolites are in the bulk phase essentially transparent to microwave irradiation, i.e., they don't absorb microwave energy. However, depending on the structure of the zeolite, there can be hydroxyl-groups on the surface (also called silanol-groups), which absorb microwave energy. With time, the zeolite bulk and the gas phase will be heated due to heat transfer from the surface since the gas phase does not readily absorb microwave energy.

There are at least three different temperatures in our system: the zeolite surface temperature, the bulk adsorbent temperature, and the gas phase temperature.[1] Different temperatures will also be observed if there is a substance adsorbed on the zeolite surface, which can absorb more or less microwave energy.

A source of microwave reflection may appear where the microwave energy passes from one medium to another one with a different permittivity. In the system used, transitions in permittivity occur at the coax cable connections, and because the zeolite bed is not a homogeneous medium, reflection may also occur between solid particles and the surrounding gas phase. A network analyzing experiment revealed the reflections in the system are most likely from the beginning and the end of the adsorbate bed. Therefore, reflections within the bed may be neglected. A stubbed tuner was used to minimize reflections at the beginning and the end of the zeolite bed.

Experimental

The desorption column used in these experiments functioned as a circular waveguide, with a tube diameter of 76.2 mm and a frequency of 2.45 GHz. The dominant mode supported was the TE_{11} mode ($f_c = 1.459$ GHz).[4]

The radial field distribution of electric field should be a Bessel function of first kind, first order, and is shown in Figure 1 below.

A Sairem GMP 03 K/SM microwave generator was used, which can provide a variable power from 0 to 300 W in continuous mode at a frequency of 2.45 GHz. The microwave power was delivered to the desorption column through a 2 meter long coaxial cable.

The microwave generator also measured the amount of power that was reflected back to the source by the system. This reflected power was kept as low as possible. A stubbed tuning device was coupled into the transmission line between coax cable and column. Reflected power was typically 11–14% of forward power.

The column functioned as a waveguide. It was chosen to conduct the experiments in a waveguide and not in a microwave cavity because the electric field in a waveguide is well understood. In a cavity, the electric field intensity is significantly nonuniform.

Both the carrier gas stream and the microwaves were delivered to the same end of the column, from bottom to top. At the

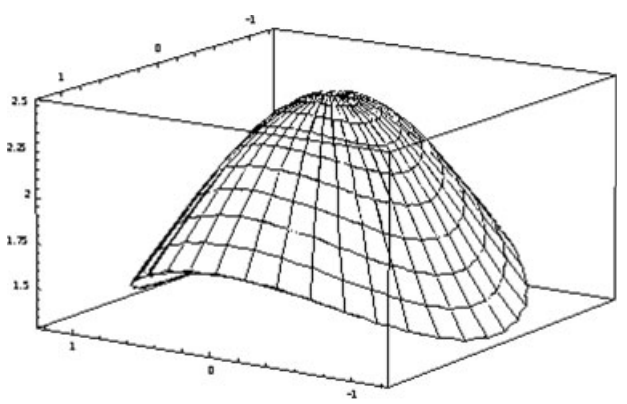

Figure 1. Theoretical radial power distribution for microwave propagation through a cylindrical waveguide.

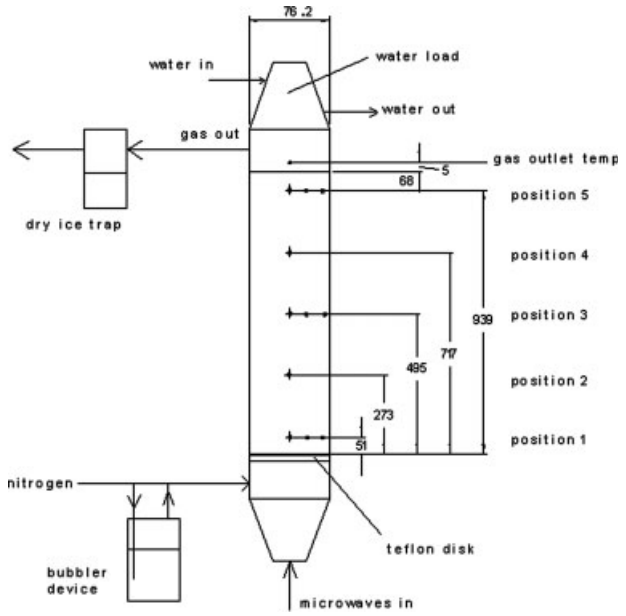

Figure 2. Diagram of the apparatus (distances in mm).

top end of the column, a water load (Raytheon G 2000 TA) absorbed the remaining microwave power in the column. The water load was supplied with water by a peristaltic tube pump (Verder VRE 200) that provided a constant water flow.

Nortech Fibronic model NoEMI-TS fiber-optic probes were used to measure the temperatures. The fiber-optic probes are made of a teflon-coated optical fiber which is essentially transparent to microwaves. The probes were positioned in the column at five equidistant axial positions, 222 mm from each other. The probes were fixed within the bed at five equidistant axial positions at the following heights above the bottom end of the bed: 51, 273, 495, 717, and 939 mm. The probes were placed at up to three radial positions: at the center axis, near the wall (38 mm from the center axis), and half way in between (19 mm from the center axis). Another probe measured the gas outlet temperature, situated on the center axis, 3 mm above the zeolite bed. Figure 2 shows the setup of the apparatus. The axial positions of the temperature probes were numbered from the bottom to the top of the column, along the direction of the forward microwave power.

A Nitrogen carrier gas was employed for the experiments, and was taken directly from a liquid nitrogen tank. This assures that the carrier gas stream contains no water. The gas enters the column at room temperature.

Adsorbates were introduced into the column, employing the N_2 carrier gas stream. The gas stream was saturated with adsorbate by passing it through a bubbler device. The temperatures along the column were monitored to determine when adsorption was complete. The heat release during adsorption indicated the location of the adsorption front. The adsorption was stopped when the adsorbate was detected in the gas outlet. The gas exiting the column was led through a dry ice trap, where adsorbate contained in the gas was condensed and collected in a graduated cylinder.

The maximum flow rate used was 5 SLPM (Standard Liter Per Minute). This flow rate gave a velocity of 0.0183 m/s in the column, and a Reynolds number of 178. Therefore, we always had laminar flow in the column.

The column was filled with 2880 g of silicalite zeolite. The zeolite was held inside the column by a perforated teflon disk. The zeolite used in these experiments was silicalite from United Catalyst T-4480 E Pentasil. It was a cylindrical extrudate, with a size of 1/16″ (1.3 mm). The packed bed density was 0.65 ± 0.03 kg/l. The SiO_2/Al_2O_3-ratio was >1000 (data given by the manufacturer). The dielectric properties of the silicalite were measured to be $\varepsilon' = 1.25$; $\varepsilon'' < 0.01$ at a frequency of 2.45 GHz. The low value for ε'' shows that the zeolite is almost transparent to microwaves.

To clean the adsorbent from preadsorbed substances (especially water, adsorbed from the ambient air), the zeolite was dried in the column with a microwave power of 300 W and a purge stream of nitrogen.

The adsorbate studied was methanol, which was obtained from EM Science with a purity of >99.9%. Its bulk liquid permittivity values at room temperature and 2.45 GHz are $\varepsilon' = 23.19$ and $\varepsilon'' = 13.84$. Gaseous methanol is essentially transparent to microwaves. The heat of adsorption of methanol on this silicalite is 43 kJ/mol.[6]

Results

First, the column was studied with no adsorbate on the zeolite. The pure heating curves were monitored. Two different powers (100 and 300 W) were chosen, as well as three different flow rates (1 SLPM, 3 SLPM, 5 SLPM) of the carrier gas.

The radial energy distribution in a cylindrical column of constant permittivity should be a Bessel function. This is superimposed onto an exponential attenuation along the column due to absorption of energy.[4]

The radial temperature distribution profile was observed and is shown in Figure 3. Because only three radial points were measured it cannot be proven without additional data that the profile is that of a Bessel function.

The steady-state temperatures were monitored after 3 hours. Generally, the temperatures increased along the column to a maximum at Position 3 or 4 (depending on the flow rate). Position 5 always showed a temperature lower

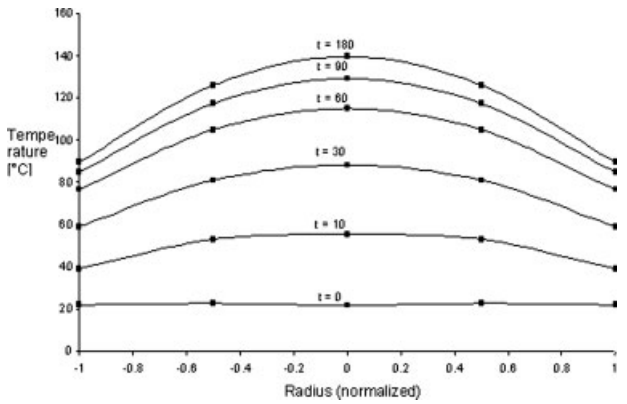

Figure 3. Radial temperature profile at 300 W, 1 SLPM, for position 3 with time.

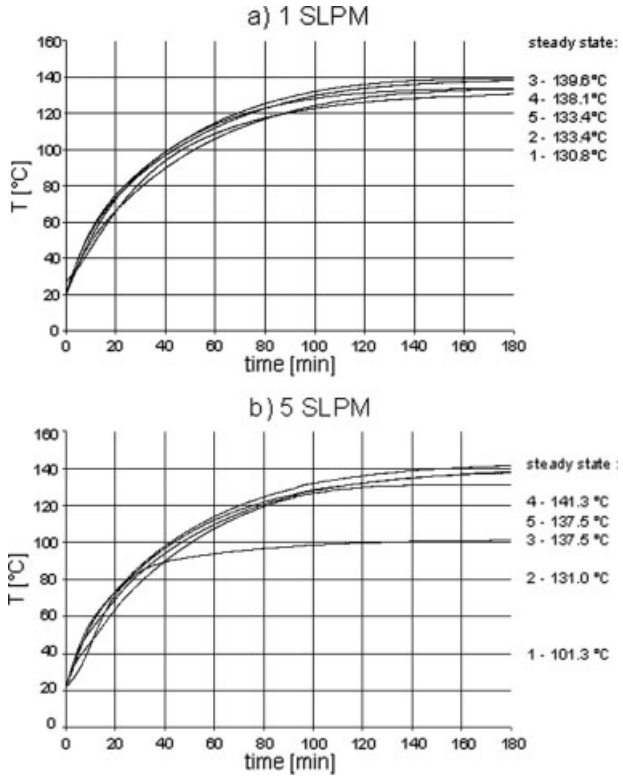

Figure 4. Center temperatures at 300 W (dry column) with time, a) 1 SLPM, and b) 5 SLPM.

than the maximum. This is shown in Figure 4. The lowest temperature was always found at Position 1.

About 30% of the forward power passes through the bed and is absorbed in the water load. For a solid bed of dry zeolite, the penetration depth is calculated to be 4.27 m, which is longer than the length of the bed; the actual penetration depth would be even longer if the void fraction was considered. A significant amount of energy passes through the column when no adsorbate is present.

The observed axial temperature profile may be explained by convective heat transfer through the gas flow, superimposed onto an attenuation of microwave energy further from the column.

The effect of the gas stream on the steady-state temperatures at a higher flow rate (5 SLPM) can be seen in Figure 4b. Carrier gas at room temperature enters the column, and, thus, cools the upstream portion. This cooling effect is in the lower part of the column (where both the gas and the microwaves enter the column).

The temperature of the gas stream increases at higher flow rates. A higher velocity results in a higher Nusselt number, improving convective heat transfer. Table 1 gives an overview of approximated gas outlet temperatures at different conditions.

Table 1. Gas Outlet Temperatures at Different Conditions

Flow rate [SLPM]	$T_{g,out}$ at 100 W [°C]	$T_{g,out}$ at 300 W [°C]	Nusselt number
1	43	66	3.82
3	Not measured	91	6.39
5	57	100	8.17

The desorption of methanol was then studied under the three following conditions: (1) 300 W and 1 and 5 SLPM carrier gas flow, (2) 100 W and 1 SLPM, (3) 200 W and 3 SLPM, with two different flow directions.

From Figure 5, the microwave energy was being strongly absorbed by the methanol. The temperature did not increase for all the temperature probe positions in the bed at the same time (as was the case in the experiments without adsorbate). There was a noticeable delay between the temperature increase along the length of the bed, indicating the time needed for the microwaves and/or cumulative convective heat transfer to reach each position, as the adsorption front moves down the column. It took about 100 minutes until the attenuation along the column was weak enough so that a significant amount of microwave energy reached the water load.

The temperature increase proceeds in two steps: first, there is an increase to between 60 and 67°C, then it increases to around 180°C. The boiling point of methanol is 64.5°C

This first temperature increase was due to convection. The gas stream, which has already been heated while passing the zone where methanol was being desorbed, heats the region that was not yet being reached by the microwaves. Position 1 was near enough to the microwave source so that it was irradiated almost instantaneously, so the temperature increases immediately after the microwave power was turned on at Position 1.

The gas outlet temperature increases quickly and remains around 60°C. It increases slightly, after the methanol is desorbed, to about 65°C. This is also the temperature observed without any adsorbate.

The temperature increased to around 180°C while the methanol is being desorbed. With time, the center temperature decreased. There was an interesting pattern observed in each temperature profile occurring about two hours after the beginning of desorption. This pattern was related to the behavior of the reflected power, which is also plotted in Figure 5.

Comparing these conditions to a run at a higher flow rate (5 SLPM), only minor differences were seen:

- Desorption happens faster due to the enhanced convection.
- The maximum temperatures are about 20 K lower due to greater cooling by the carrier gas at the higher gas flow rate.

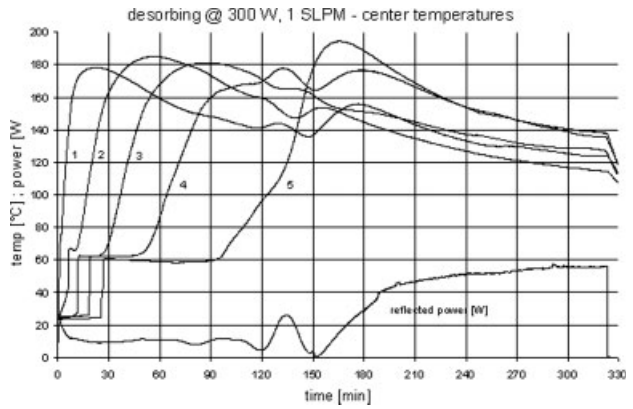

Figure 5. Center temperatures during desorption, 300 W and 1 SLPM.

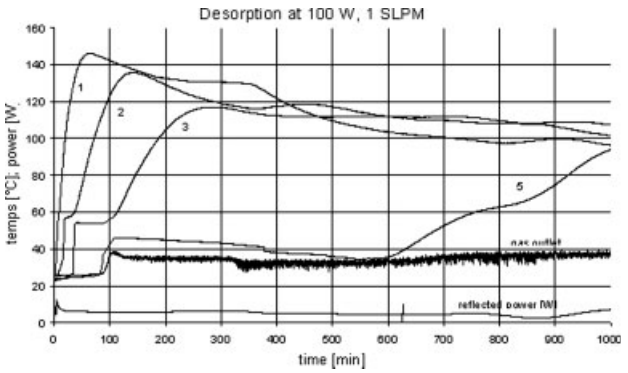

Figure 6. Center temperatures during desorption, 100W and 1SLPM.

• The first temperature increase does not reach 60°C; rather, it stays between 50 and 60°C and slowly decreased with time.

There were obvious differences for the lower microwave power (100 W) shown in Figure 6:

• The power was a third of the power in the other run, but desorption took much longer than three times as long (e.g., 1000 minutes compared with 150 minutes to reach the temperature at position 5).

• The maximum temperatures at 100 W were lower than at 300 W, and decreased at longer distances from the microwave source.

These differences were not all evident in a run at 200 W and 3 SLPM. It appears that a power of 100 W is so weak that the gas stream is not fully saturated. Note that the gas outlet temperature stays more than 20 K below the boiling point of methanol.

The temperatures were essentially independent of radial position before the microwaves reach each position, as seen in Figure 7a. The wall temperature was slightly lower, which was due to the heat transfer through the nearby wall. When desorption was started, a radial profile was observed that was similar to when no adsorbate was present (similar to a Bessel function). This pattern is established at the beginning of desorption as seen in Figure 7b and persists also after the methanol is desorbed and temperatures are decreasing again as shown in Figure 7c.

An experiment with only the lower half of the adsorbent bed filled with zeolite was performed to examine a change in the distribution of methanol, and is shown in Figure 8.

The behavior of the reflected power was always similar during desorption. The absolute values differed with different flow rates or different microwave powers, and desorption took longer at lower powers; qualitatively, the curves showed the same features, and the reflected power was categorized into four zones:

• Zone A: Immediately after turning on the microwave power, the reflected power started at a high level and went to a lower level within a few minutes.

• Zone B: The reflected power stayed lower than observed in the experiments with no adsorbate (around 5% of forward power).

• Zone C: The reflected power decreases, then increases quickly higher than in zone B, then goes down to a global minimum.

• Zone D: The reflected power increases to a steady-state level while desorption was being completed.

The behavior in zone A may be explained simply by changes in the properties in the microwave transmission, due to heating of the coax cable after starting the microwave generator. Losses might increase with temperature, causing reflections to be dissipated instead of being transmitted back to the generator where they are measured.

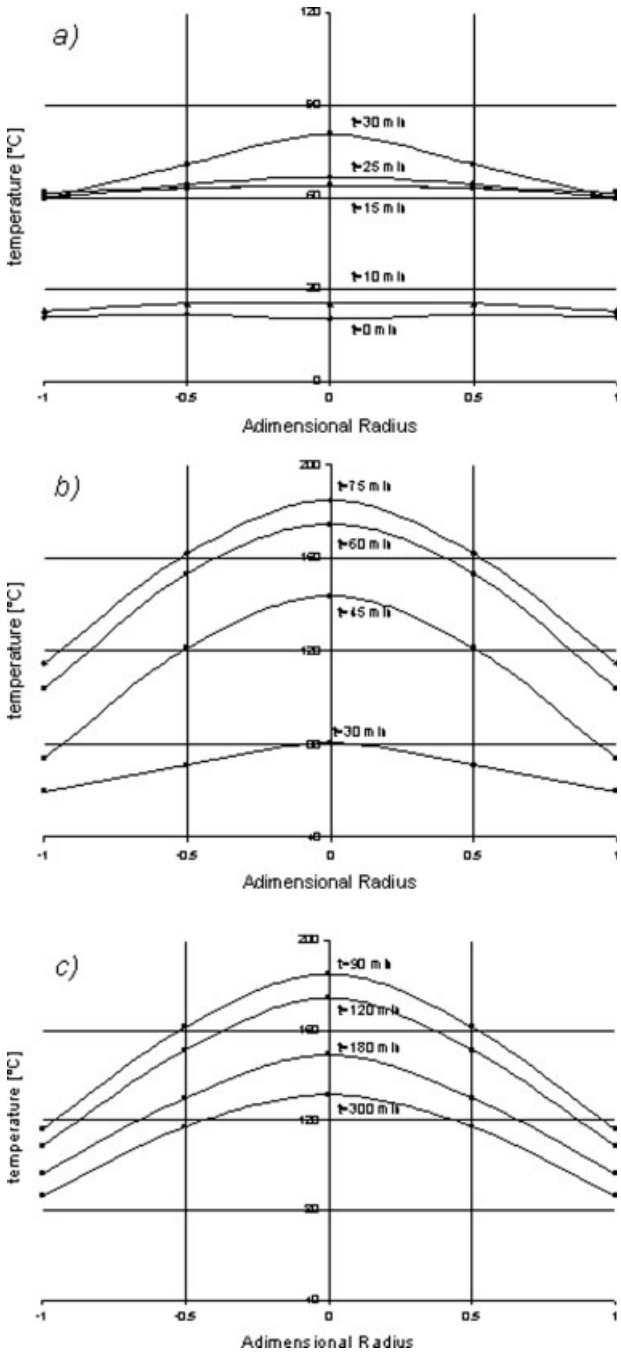

Figure 7. Radial temperature profile at 300 W, 1 SLPM, and position 3, a) before microwaves reach the position, b) during desorption, c) after methanol depletion.

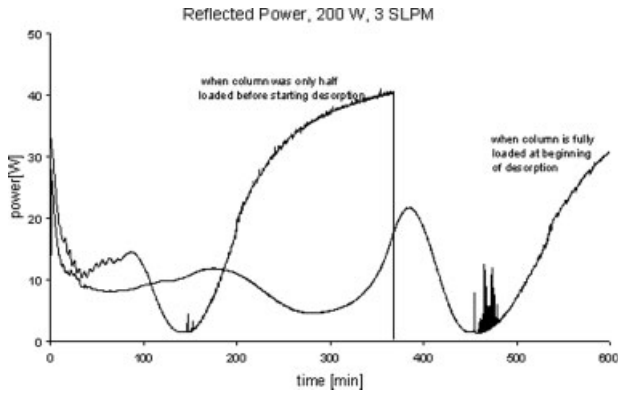

Figure 8. Reflected power for methanol desorptions with different amounts of adsorbate initially adsorbed.

Zone B exhibited a lower reflection because the microwaves were absorbed before they could be reflected at the end of the bed, causing a fairly constant level of reflected power. It can also be seen that the microwaves don't reach the end of the column, because the temperatures further down the column increase only from convection. Accordingly, an increase in temperature at the water load was observed at the end of zone B.

In zone C, the reflected power started to vary. As can be seen from the temperature profiles, this zone started when the microwaves penetrate the entire column. The increase in reflected power happened when a certain fraction of the methanol has been desorbed and was dependent on the amount of methanol remaining in the column.

For desorption with only the lower half of the column filled with adsorbate, zone C was reached much faster.

Also, a desorption run was performed with a reversed direction of the gas flow. This was done to study the effects of convective transport in the direction of the flow. This experiment was conducted at 200 W and 3 SLPM. This run took a much longer time to desorb. The same behavior of the reflected power occurs. From these observations, the amount of methanol has a much higher influence on the reflection than has the distribution within the column.

Since the distribution of methanol in the column did not seem to influence the reflections, the zeolite bed can be considered as a homogeneous medium with an average dielectric constant ε', being an average of ε'(zeolite) and ε'(methanol); However, ε' decreases with time as methanol is being carried out of the system. Wavelengths change with different ε', according to

$$\lambda = \frac{\lambda_0}{\sqrt{\varepsilon'}}$$

As the microwaves are being reflected from the end of the zeolite bed, they interfere on their way back to the generator with the other microwaves from the beginning of the bed. If the two waves are in-phase, the sum of them (which is monitored by the generator) grows, if they are out-of-phase, the sum decreases.

The wavelength of the reflection within the column also changes in time. This results in a varying phase shift when the second reflected wave finally leaves the column. Thus, the generator observes a sinusoidal reflected power in zone C. In addition to this varying phase shift, the intensity of the second reflection changes, because more microwave power reaches the end of the bed and is reflected there. This also will affect the observed total reflected power.

The reflected microwaves interfere with the forward traveling waves in the bed, giving rise to standing waves. This means that the intensity of microwave irradiation varies periodically along the column as a function of axial location, and also varies in time as the wavelength changes. It is possible that a temperature probe is exposed to a higher (or lower) microwave power level than before. Temperatures at other positions change with the same periodicity.

In zone D, all of the methanol has been desorbed, and, therefore, no more variation in the reflected power is occurring. The reflected power rises to a constant level. Surprisingly, this constant level was about twice as high as it was in the experiments without adsorbate. This observation was a first hint that something else happened in the column during desorption. Some of the methanol reacted with silanol groups present on the zeolite surface to form methoxyl groups. Having methoxyl groups on the surface changes both the adsorption behavior, and how the surface interacts with microwaves. Methanol reacted in the presence of microwaves. The dehydration of methanol formed dimethylether and water.

The reproducibility of desorption of methanol was then examined. Figure 9 shows how the center temperatures change in time during the first adsorption of methanol. Figure 10 shows the second adsorption of methanol, after the first desorption had taken place and the methanol was completely desorbed using microwaves. Now a significant difference can be seen: a distinct second peak. The same temperature profiles were found in all other subsequent methanol adsorptions after the first one.

To confirm that the single peak at Position 1 was not a result of a more intense microwave irradiation during desorption that could have changed the zeolite properties, an experiment was performed with the direction of the gas flow reversed. From the result shown in Figure 11, it does not

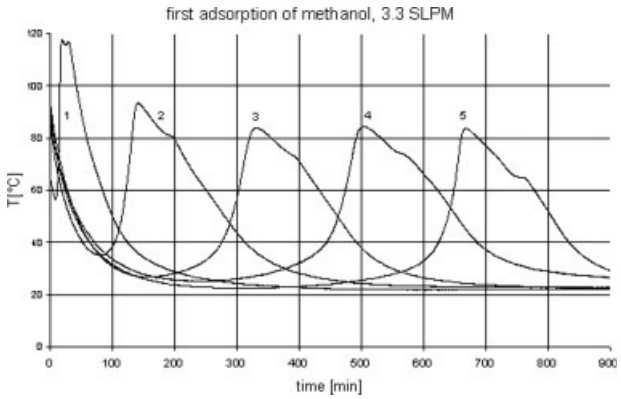

Figure 9. First adsorption of methanol.

matter from which direction the microwaves come from during the desorption step.

Pure water was then adsorbed on the column and desorbed with microwaves, and another methanol adsorption was done. This revealed a most interesting behavior; the methanol adsorption after this showed the same pattern as the first overall methanol adsorption. Subsequent methanol adsorptions again showed a two-peak-pattern like the one in Figure 10. Microwave desorption of methanol may modify the sorption properties of the silicalite, and the surface modification due to the adsorption of methanol may be reversed by sorption and microwave desorption of water.

It was revealed that there was water in the column during methanol adsorption, when the gas stream coming out of the column was led through the dry-ice trap; a short time after the beginning of the adsorption, water could be observed crystallizing out from the gas stream at dry ice temperature (−45°C). Water was then suspected to be a product of a reaction that was occurring with methanol in the presence of microwaves. A sample was examined through a gas chromatograph and GC/MS. This revealed three substances: methanol, water, and dimethylether.

The suspected reaction was the dehydration of methanol to dimethylether:

$$2\,CH_3OH \rightarrow CH_3OCH_3 + H_2O$$

Methanol is reported to form dimethylether on cationic zeolite catalysts like Y-zeolites or ZSM5-zeolites that have a low SiO_2/Al_2O_3-ratio, at temperatures above 180°C.[7] Y and ZSM5 zeolites have a far lower SiO_2/Al_2O_3-ratio than was used in these experiments. Bandiera and Naccache[8] report experiments of dehydrating methanol on a dealuminated H-mordenite with Si/Al $>$ 80 above 473 K.

This reaction explains how water does get into the column. However, studying the extent of this reaction in the system and its exact conditions was beyond the scope of this work, as no reaction was anticipated. Considering the conditions in the column and the literature, it is assumed that only a small portion of the methanol reacts. The reaction takes place via a surface methoxy intermediate.[7] This suggests that a partly methoxylated surface could still be present after desorption. Bandiera and Naccache[8] also report that at 473 K, which is in the lower region of their studied temperature range, reaction products remain adsorbed.

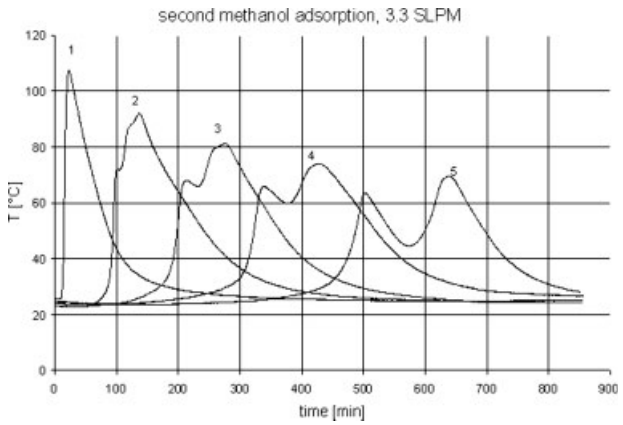

Figure 10. Second adsorption of methanol.

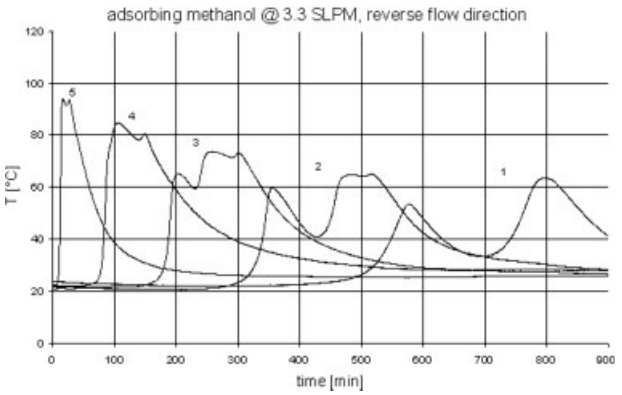

Figure 11. Reverse gas flow direction.

Comparing the two different methanol adsorption conditions, it can be seen that the second run was faster (at the same flow rate), and the area under the curves was smaller in the second run. This suggests that not as much methanol was adsorbed as before, since the adsorbent surface had been modified. After the reaction takes place, not all methanol can be removed by microwaves, and a partly methoxylated surface remains after desorption. The methoxyl groups occupy the adsorption sites that give the greatest heat of adsorption and are therefore the most difficult to be removed. When these sites are free and can adsorb, as was the case in the first methanol run, a larger heat release was observed.

Conclusions

In these experiments, the instantaneous and transitory behavior of the temperature and the propagation of microwave energy were studied during desorption from a zeolite in a 1-meter waveguide column. Methanol and/or water were used as the adsorbates. From the experiment performed, the following conclusions were made:

- The axial temperature profile observed for the column without adsorbate present was explained by convective heat transfer through the gas flow, superimposed onto an attenuation of microwave energy along the column.
- With adsorbate present, the microwave energy is attenuated within a short distance. When methanol adsorbate was present, there was a time delay of the temperature increase along the length of the bed. This indicated the position of the adsorption front as it moved along the column.
- The temperature increased along the column in two steps; the first temperature increase was due to convection, and the second due to microwave irradiation.
- The amount of methanol has a much higher influence on the microwave reflection than the distribution of methanol within the column.
- The microwave desorption of methanol modified the sorption properties of the silicalite, and the surface modification due to methoxylation was reversed by the sorption and microwave desorption of water.

• Methanol reacted in the presence of microwaves to form dimethylether and water

The gas flow rate and microwave power used have an influence on the time necessary for completion of desorption. Higher flow rates carry the desorbed methanol out of the column more quickly, and a higher microwave power induces desorption more quickly. The longer the desorption process takes, the more time in which heat is transferred from the zeolite surface to the zeolite bulk phase and the gas phase. This heat doesn't directly contribute to desorption, and, therefore, the overall efficiency is expected to go down.

The power reflected from the column varied during desorption, influencing the axial temperature profile. Standing waves developed in the column when reflection occurs at the end of the zeolite bed.

These studies demonstrated that an adsorbent such as methanol could easily be regenerated by the use of microwaves. It is still unknown if the use of microwaves was the cause of the reaction of the methanol to form dimethylether and to methoxylate the zeolite, or if the reaction also would have occurred using conventional heating under the same conditions. Considerations about efficiency would require quantification of the amount of methanol desorbed with time, and of the changes in heat loss for various system components with time.

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