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Microwave Irradiation Effect in Water-Vapor Desorption from Zeolites

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1. Introduction

In recent years, humidity control has been recognized as one of the important technologies in various fields; e.g., the system is required to maintain comfortable indoor air quality in household sector, and to improve the quality of products in industrial sector. In general, controlling the humidity through temperature, as in the case of conventional systems, appears to be an energy consuming process, and, depending to the operation conditions, does not assure the demand levels for humidity and temperature. Under these circumstances, desiccant humidity conditioner, which makes use of adsorption/desorption phenomena of porous adsorbent, has been gaining a great attention as an environmental friendly humidification/dehumidification system because of its advantages in the following points:

1. It consumes very little electrical energy, and for regeneration process it allows the use of solar energy and waste energy.
2. It is efficient when latent heat load is larger than the sensible load.
3. It is a clean technology, which can be used to condition the internal environment of buildings and operates without the use of harmful refrigerants.
4. The achieved control of humidity is better than that when using vapor compression systems.
5. In some cases the cost of energy to regenerate the desiccant is less than that when compared with the cost of energy to dehumidify the air by cooling it below its dew point.
6. Improvement in indoor air quality is more likely due to the normally high ventilation.
7. It has the capability of removing airborne pollutants.

The technology of adsorptive desiccant cooling presents interesting prospects as regards market penetration (Ando & Kodama, 2005; Davanagere et al., 1999; Elsayed et al., 2006, 2008; Ge et al., 2008; Halliday et al., 2002; Hamed, 2003; Kabeel, 2007; Kodama et al., 2001; Mavroudaki et al., 2002; Oshima et al., 2006).

The desiccants are natural or synthetic substances capable of absorbing or adsorbing water-vapor due to the difference of water-vapor pressure between the surrounding air and the desiccant surface. Typical adsorptive desiccant cooling process mainly consisting of a rotary dehumidifier (D-hum) and heat exchanger can be driven with low-temperature heat energy

like solar energy or waste heat, and it has been expected to be alternative air conditioning considering various energy/environmental problems such as global warming. In the solid desiccant system, a desiccant, which is coated with silica gel or zeolite, is generally used as an adsorber/desorber. During the desorption process of the system, hot air is generally used as a heating medium, and the heating of air is carried out using a solar collector, electric heater, and exhaust heat in some industrial factories. However, indirect heating with hot air, especially with the one generated by an electric heater, results in consumption of large amount of energy for regeneration, because of the fact that, the heating of air is required to heat entire desiccant rotor consisting of an adsorbent and rotor matrix. As a result, the system is characterized by low heating efficiency. In addition, excess temperature rise in the rotor during desorption process causes low water adsorptivity of an adsorbent in the following adsorption process. Moreover, when regeneration is performed with lower thermal energy below 80°C, humidification/dehumidification performance greatly decreases due to insufficient water desorption. This problem has been discussed and the optimization of adsorption-desorption process has been examined in the field of the desiccant air-conditioning, but it has not got an essential solution yet (Hamamoto et al., 2004; Harshe et al, 2005; Kodama et al., 2005).

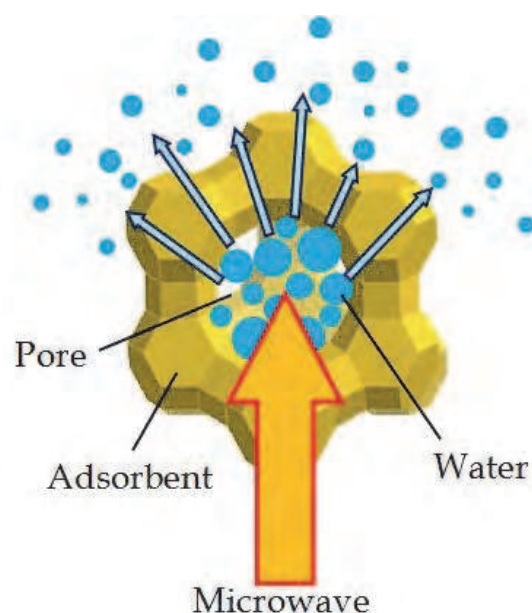


Fig. 1. Imaginative diagram of desorption by microwave selective heating

The method for application of the microwave irradiation as a regeneration heat source of the adsorption material is considered as a strategy to the above mentioned problems. Microwave irradiation has a great advantage of direct and rapid heating of material due to self-heating of a material under microwave irradiation, which consequently results in heating of only adsorbent containing adsorbed water without heating the surrounding air (as shown in Figure 1) (Bradshaw et al., 1997, 1998; Cherbanski & Molga, 2009; Kuo, 2008; Polaert et al., 2007, 2010; Yan et al., 2004, 2007).

Hence, in the process of water desorption, there is a possibility that adsorbed water is selectively heated by microwave irradiation rather than the adsorbent, resulting in an enhancement of desorption with lower energy consumption. Based on the above mentioned advantages of microwave heating, we have proposed the novel hybrid regeneration process,

which combines microwave heating and conventional hot air heating. By combining both heating methods, the hybrid system shown in Figure 2 is expected to achieve highly energy efficiency for regeneration due to direct and rapid heating by microwave irradiation as well as low thermal energy utilization provided by hot air heating. The system also has a feature of promotion of lower heat utilization by assisting water desorption with an additional energy of microwave.

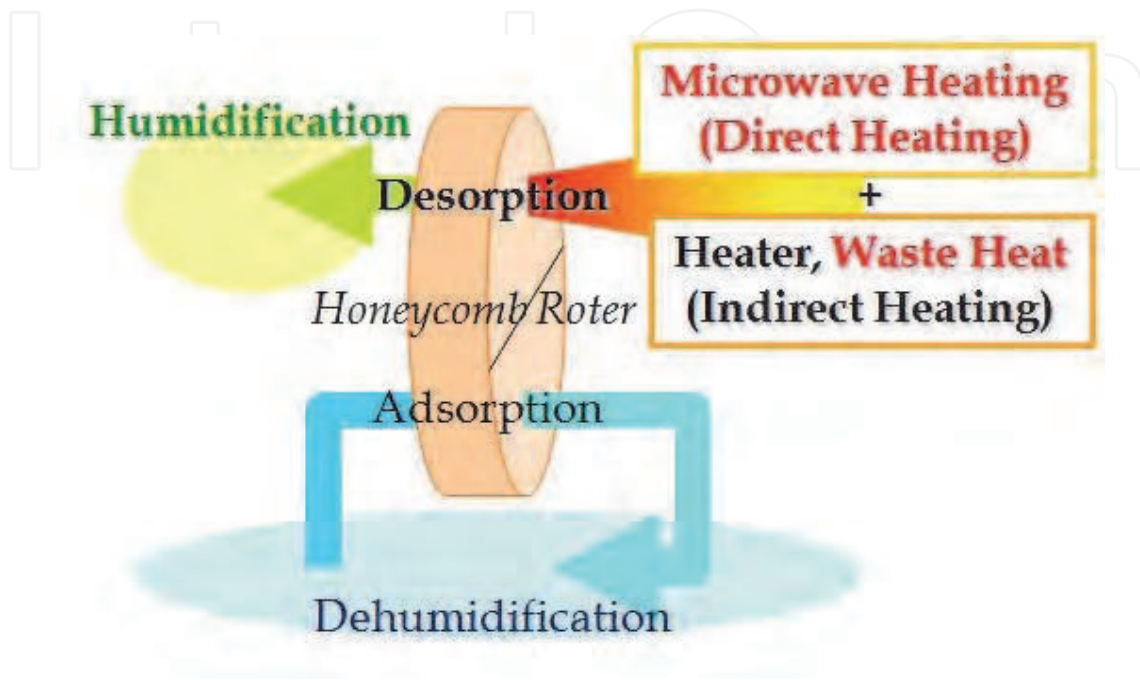


Fig. 2. Concept of the microwave heating hybrid system

Some researches have been carried out on the application of microwave heating to the regeneration of adsorbent for desiccant air conditioning. Ohgushi and Nagae have investigated heating and dehydration characteristics of various zeolites with microwave heating for the reusable desiccant in home, and reported that the mixture of Na-X and Ca-X was useful to prevent the thermal runaway (Ohgushi & Nagae, 2003). Concerning the durability of zeolite mixtures against microwave heating, they have also indicated 1.3% degradation of water adsorptivity after each MW irradiation (Ohgushi & Nagae, 2005).

In previous study, we paid attention to zeolite that showed strong water-vapor desorption capability as an adsorbent, and microwave irradiation effect was examined in water-vapor desorption of zeolite 13X (Saitake et al., 2007). As a result, the maximum desorption rate was found to be about 5 times higher for microwave heating at 800 W than that obtained for hot air heating. It was also observed that the amount of water desorbed from zeolite particles by microwave heating was 1.6-2.0 times larger than that by hot air heating, regardless of microwave power.

However, almost all experiments have been performed with powder or granular adsorbent, and there are very few researches on water desorption from desiccant rotor with microwave heating. Microwave desorption feature of these zeolite is almost unknown. Therefore, it is essential to grasp the influence of condition such as flow rate and temperature of air on desorption rate to establish the HM with microwave heating condition.

In this research, the examination of two items as follow was performed under the above-mentioned viewpoints that are; i) Experimental study was performed about the influence of

adsorption equilibrium and pore architecture in desorption of microwave heating by three zeolites. ii) Experimental study was performed about the influence of gas flow rate and temperature in desorption of microwave heating.

2. Experimental

2.1 Adsorbents

3 kinds of zeolite samples (4A, OXYSIV-5 and DF-9, average particle size fraction of 500 μ m) (made by UNION SHOWA K.K., Japan) were used. The pore size of samples is 0.4nm, 0.8nm and 1.0nm. The water-vapor adsorption and desorption isotherms were measured by water-vapor adsorption device (Belsorp aqua3, BEL JAPAN, INC.) at 30°C and shown in Figure 3.

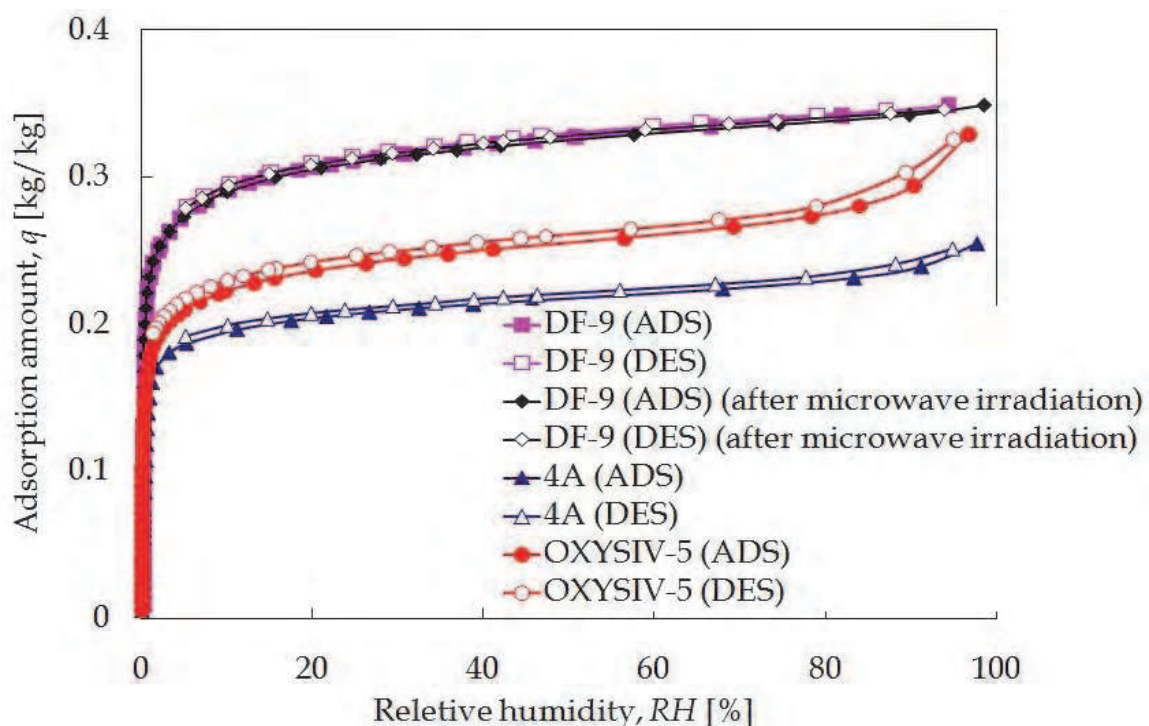


Fig. 3. Adsorption and desorption isotherms of water-vapor on zeolites at 30°C

The adsorption amount of water-vapor on 4A and OXYSIV-5 rise sharply in the range of the relative humidity, RH, below 5%, and then increased gradually. For 4A, the adsorption amount of water-vapor below 5% of RH accounts for 75% of the total adsorption amount. In addition, the adsorption amount of 4A is smaller than that of OXYSIV-5. For DF-9, the adsorption amount of water-vapor rise sharply in the range of the relative humidity, RH, below 10%, and then increased gradually almost similar to that of OXYIVE-5. The adsorption amount of water-vapor below 10% of RH accounts for 84% of the total adsorption amount which is 1.3 times larger than that of OXYSIV-5. In addition, the water-vapor adsorption and desorption of 4A and DF-9 showed desorption hysteresis, which is smaller than that of OXYSIV-5.

on, visual check and measurement of water-vapor adsorption and desorption isotherms was carried out after the microwave irradiation experiment. As a result, the damage and the transformation of the zeolites by microwave irradiation were not observed. Moreover,

change of adsorption and desorption isotherms by the existence or nonexistence of microwave irradiation was not observed as shown as Figure. 3.

2.2 Experimental apparatus and method

The experimental apparatus, as shown in Figure 4, consisted of a microwave irradiator, a circulated packed adsorption, an evaporator, a microwave absorber, a heater and thermometers. To keep the apparatus on a constant temperature (30°C), the insulant was used to enclose the apparatus.

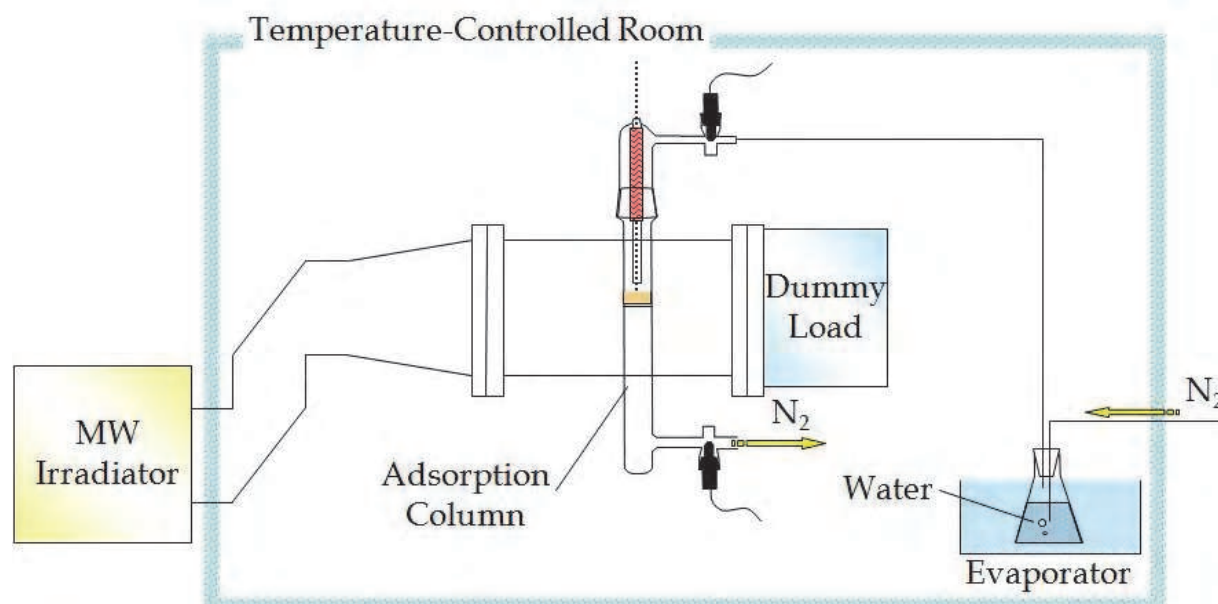


Fig. 4. Schematic diagram of experimental apparatus

Microwave was oscillated at 2450MHz, passed a cylindrical waveguide of 110mm in the diameter (TE₁₁ mode) through a rectangular waveguide (TE₀₁ mode), and was absorbed by the microwave absorber. The circulated type absorption column was vertically set at 140mm from the entrance and in the center of section of the circular waveguide. The position where the adsorption column was set up was the position where the electric field strength of microwave was the maximum. The humidity-temperature meters (PosiTector DPM, DeFelsko Corp.) were set up in each gateway of the circulation air was calculated from the measurement of temperature and humidity difference, and the amount of adsorption and desorption were calculated based on those. The fiber-optic thermometer was inserted in the center part of the adsorbent packed bed to measure the temperature, and this temperature was assumed to be a representative temperature of the adsorbent packed bed.

The samples bed (particle diameter: 0.3-1.0mm, adsorbent bed thickness: about 2.5mm (0.5g)) in adsorption column was set in the position where electric field intensity of microwave waveguide was the maximum. In this study, a micro heater (20W) is inserted in the sample bed upper which is a circulated packed adsorption column. The adsorbent bed temperature was adjusted with heating the circulation gas by electric power (shown in Figure 5). In microwave heating experiment, microwave irradiation was carried out with the supply of this measurement electric power.

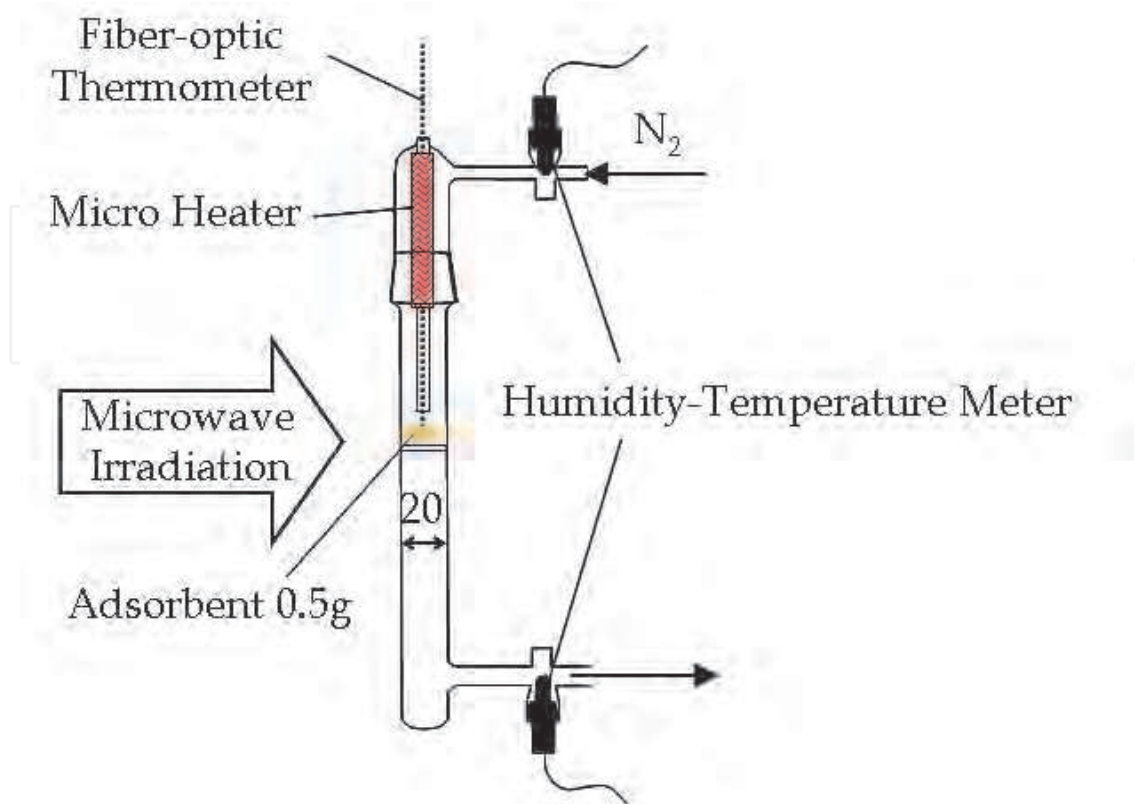


Fig. 5. Schematic diagram of adsorption column

The following two desorption experiments were carried out.

1. After zeolites were packed into the adsorption column, the adsorption column was heated and kept at 350°C. The N₂ gas of 99.99% was circulated enough to dry the adsorption column as a pretreatment. Adsorption process was carried out by circulating N₂ gas of relative humidity 40% to adsorption column at 3.18m/min. Desorption process was carried out by microwave heating under conditions of N₂ gas of 30°C with relative humidity 40%, gas flow rate of 1.62-6.36m/min and microwave power of 800W as comparison.
2. After the adsorption of DF-9 became equilibrium like 1) (flow rate: 3.18m/min), the desorption experiments was carried out by supplying power (electric power can heat 40 to 80°C of adsorbed bed achieving temperature.) and microwave heating (microwave output; 50W).

In experiments, temperature in the center of adsorbent bed and humidity of exit of adsorbent bed were measured, and the desorbed amount was calculated by using this result.

3. Results and discussions

3.1 Comparison of microwave heating desorption effect in different zeolites

As shown in Figure 6, for 4A and DF-9, heat and mass transfer behavior of microwave heating desorption process under the condition that adsorption column inlet temperature is 30°C and relative humidity is $RH=40\%$ showed similar behavior compared with previous study on OXYSIV-5. The good repeatability under the same experimental condition was confirmed.

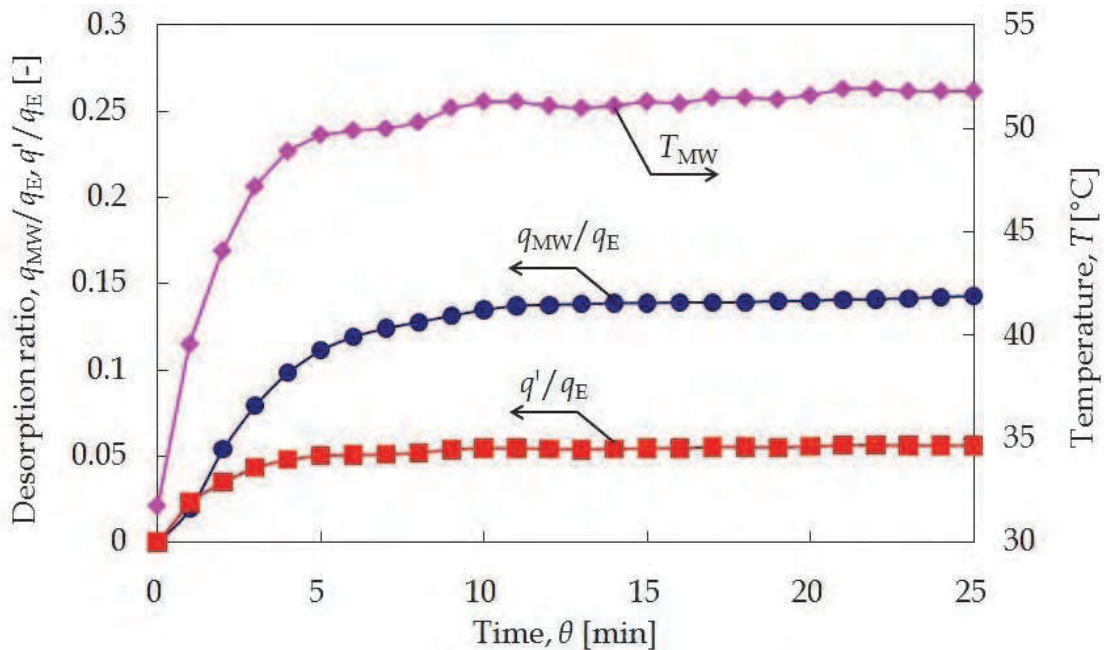


Fig. 6. Desorption ratio and temperature of zeolite DF-9 bed during microwave heating (microwave output: 800W, flow rate: 3.18m³/min)

However, for the different type of the zeolites, temperature (T_{MW}) rise, maximum achieving temperature (T_{MAX}) of adsorbent bed and desorption amount (q_{MW}) by microwave irradiation heating were different. To compare the difference of desorption of the zeolites, equilibrium adsorption amount (q_E) at 30°C, $RH=40\%$, q_{MW} , desorption ratio (q_{MW}/q_E) and T_{MAX} after desorption begins to 15 minutes (microwave output 800W), hypothetical temperature desorption amount (q'), hypothetical temperature of heat source (T'), hypothetical temperature desorption amount ratio $R_q(=q_{MW}/q')$ and hypothetical temperature of heat source rise $T_D(=T'-T_{MW})$ calculated using adsorbed equilibrium relation of Figure 3 in each zeolite sample are shown in Table 1 together with the results of OXSIV-5 in microwave irradiation time of 15min. Moreover, temperature rise rate ($\Delta T/\Delta \theta$) of adsorbent bed, relationship between desorption rate ($\Delta q_{MW}/\Delta \theta$) and adsorption ratio ($1-q_{MW}/q_E$) is shown in Figure 7 and Figure 8, respectively.

Zeolites	4A	DF-9	OXSIV-5
q_E [kg/kg]	0.218	0.322	0.242
q_{MW} [kg/kg]	0.016	0.044	0.032
q_{MW}/q_E [-]	0.074	0.136	0.132
T_{MAX} [°C]	49.1	50.1	46.7
q' [kg/kg]	0.007	0.017	0.019
T' [°C]	62.4	93.7	61.4
R_q [-]	2.22	2.59	1.57
T_D [°C]	13.3	43.6	14.7

Table 1. Desorption amount and temperature rise

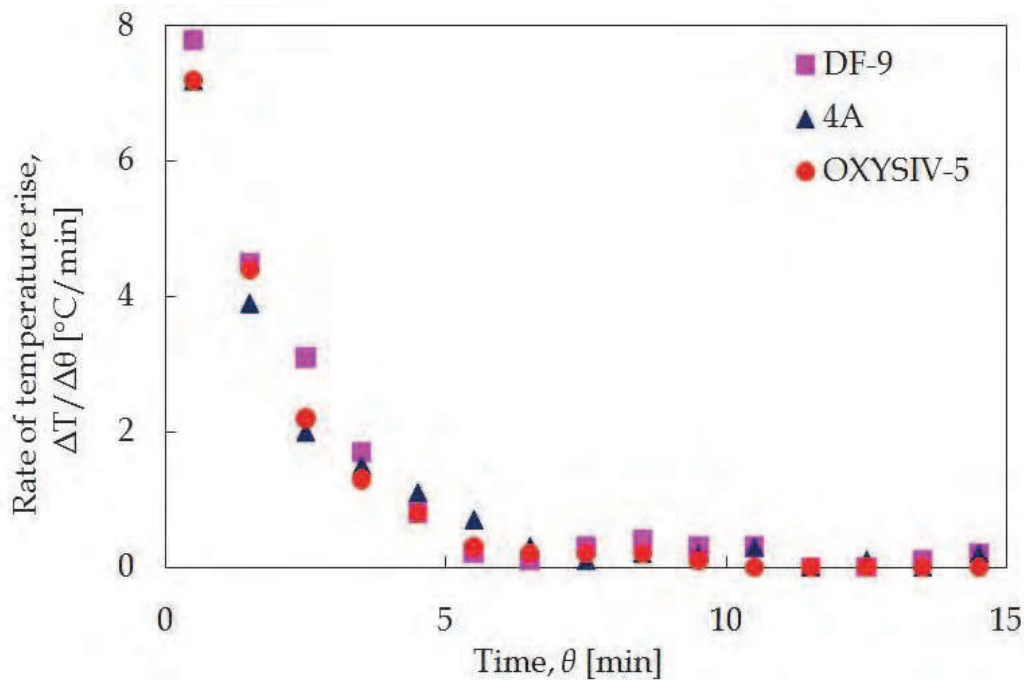


Fig. 7. Rate of temperature rise for zeolite bed

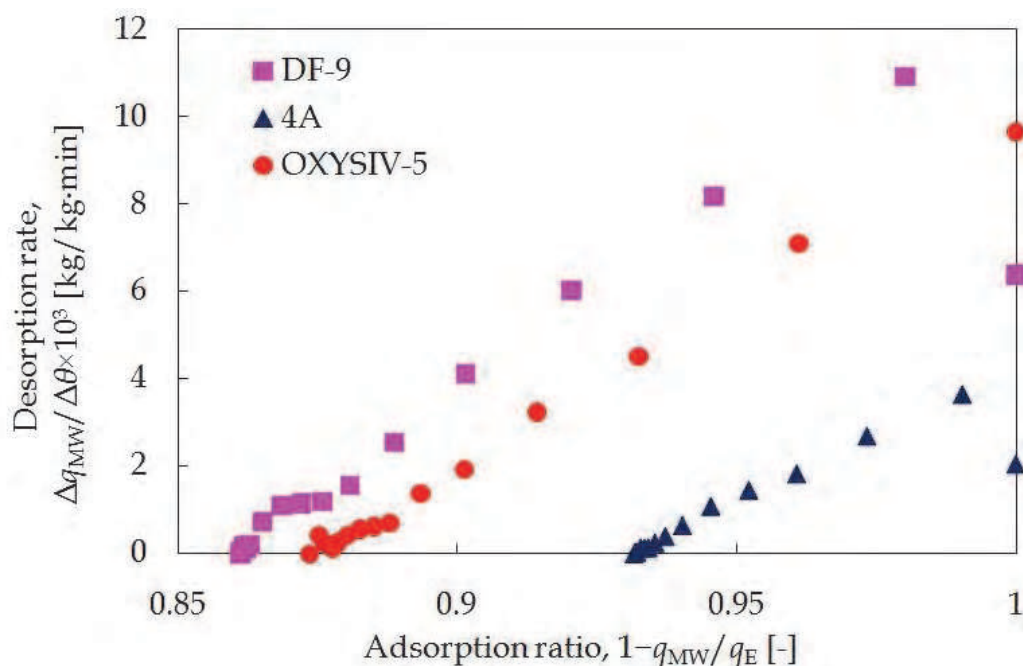


Fig. 8. Relationship of desorption rate and adsorption ratio

q' and T' are defined as follows. In this experiment, adsorbent temperature rises with microwave irradiation. On contrast, adsorbent bed inlet temperature are constant at 30°C and $RH=40\%$ (absolute humidity: $H=0.0106\text{kg}/\text{kg}\cdot\text{air}$). Therefore, relative humidity in the bed decreases because adsorption bed temperature rises, q' is equilibrium adsorption amount difference corresponding to this temperature rise on isotherms. In this calculation, Clausius-Clapeyron Equation (Aroġba, 2001; Kolaczkiwicz & Bauer, 1985; Komdaurov, 2004) was used based on adsorption isotherm of two temperatures of various zeolites.

The following was observed from Table 1, Figure 7 and Figure 8.

1. Amount order of q_{MW} is DF-9 > OXYSIV-5 > 4A. q_{MW} of 4A and OXYSIV-5 are 0.36 times and 0.73 times of DF-9, respectively.
2. Amount order of q_{MW}/q_E is also DF-9 > OXYSIV-5 > 4A. This value was slightly smaller than DF-9 in OXYSIV-5. In contrast, q_{MW}/q_E of 4A was 0.54 times of DF-9.
3. Amount order of T_{MAX} is DF-9 > 4A > OXYSIV-5.
4. R_q of each zeolite samples shows more than 1. R_q of 4A and DF-9 was about 1.4 times and 1.6 times of OXYSIV-5. T_D of 4A and DF-9 is about 0.9 times and 3.0 times of OXYSIV-5.
5. Temperature rise rate ($\Delta T/\Delta \theta$) is a little different with the type of the zeolites in desorption early time, but there is no big difference in change. The beginning desorption is the maximum, and then this value decreases afterwards.
6. On the other hand, adsorption rate shown the maximum in 0.98-1.0 of $(1-q_{MW}/q_E)$ for DF-9 and 4A, and then decreased with the decrease of $(1-q_{MW}/q_E)$ afterwards.

The above mentioned result of 1) shown that q_{MW} of the adsorbent became small with small equilibrium adsorption amount under this experimental conditions. But, equilibrium adsorption amount of OXYSIV-5 is 0.76 times of DF-9, and this value is approximately same as 0.73 times in q_{MW} . However, although equilibrium adsorbed amount of 4A is 0.67 times of DF-9, q_{MW} decreases greatly with 0.36 times.

In the range of this experiment, the water-vapor adsorption can be considered influenced by pore size with same shape of temperature rise rate of adsorbents (Figure 7). This is also shown in 6), the maximum desorption rate of 4A (pore size: 0.4nm) is 0.33 times of DF-9 (pore size: 0.8nm) and 0.38 times of OXYSIV-5 (pore size: 1.0nm). On the other hand, pore size of DF-9 is slightly small compared with that of OXYSIV-5, but desorption rate is considered depending on the adsorption amount, and the desorption rate is quick up with the large equilibrium adsorption amount.

For the result of R_q in result 4), the same desorption effect of 4A and DF-9 are shown same as that of OXYSIV-5. And the effect is different with the type of zeolites. Concretely, R_q is 1.4 times while beginning adsorbed amount of 4A when OXYIVE-5 is 0.88 times in the same experiment condition. This shows that desorption by microwave heating is more advantageous than by hot air heating in zeolite with small pore size. On the other hand, the beginning adsorption amount of DF-9 is 1.3 times and R_q is 1.6 times compared with OXYIVE-5. This shows that microwave heating is effective in desorption of zeolite which is adsorbing water-vapor in large quantity.

To confirm desorption effect of the microwave more clearly, other experiments results of OXYSIV-5 are shown in Figure 9 and Figure 10. These figures shows the adsorbent bed temperature and the desorption amount change in microwave heating desorption (microwave output: 800W) and hot air heating desorption at the adsorption column inlet temperature of 30°C and RH=40%. T_{MW} in figure is the adsorbent bed temperature by microwave heating. In addition, T_{HE} is adsorbent bed temperature under hot air supply condition that performed hot air heating to show increased temperature as same as T_{MW} . And, q_{HE} is desorption amount of water by hot air heating. T_{HE} and T_{MW} almost draw the same curve according to Figure 9. On the other hand, adsorption ratio of microwave heating $(1-q_{MW}/q_E)$ is larger than adsorption ratio of hot air heating $(1-q_{HE}/q_E)$ at all time. It is especially remarkable in beginning desorption. As for this, microwave heating causes desorption more than hot air heating desorption. It is shown that it is more effective under the condition with much adsorption water.

T_D of 3) shows decreasing effect of heat source temperature by microwave heating. This value also changes with the type of the zeolites. This value is especially excellent in DF-9. Specifically, It corresponds to desorption amount when desorption of hot air temperature 93.7°C uses hot air temperature 50.1°C together with microwave heating, and the decrease of 43.6°C of temperature of heat source becomes possible.

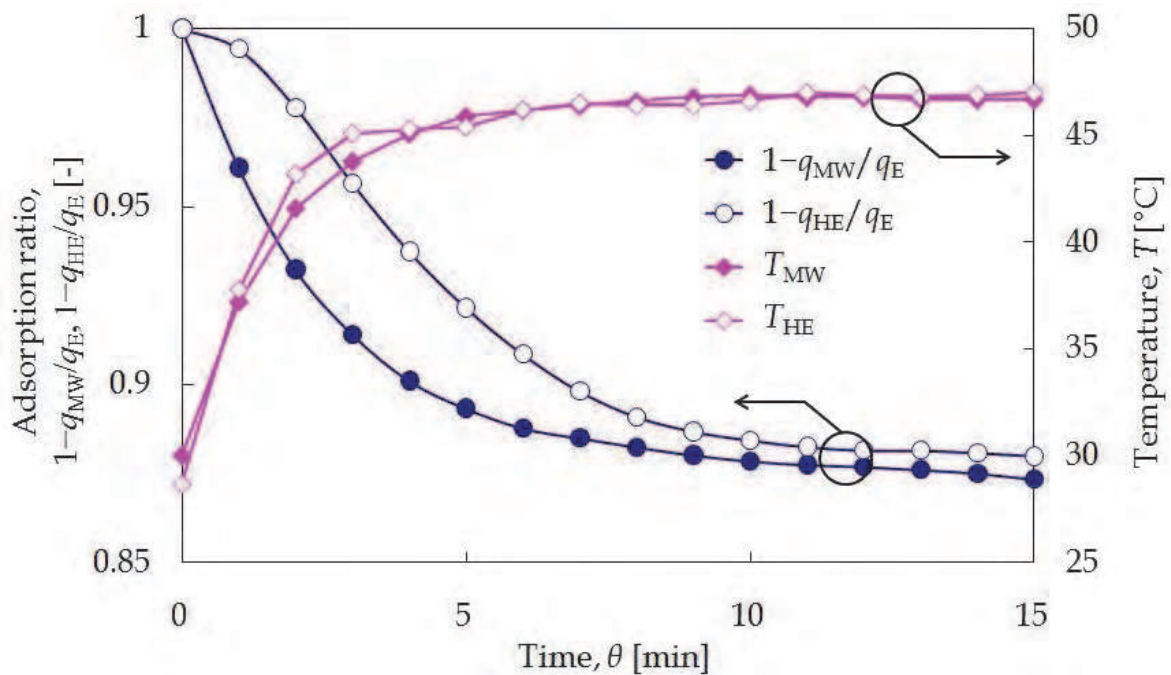


Fig. 9. Adsorption ratio and temperature during microwave irradiation and heating for zeolite OXYSIV-5 (microwave output: 800W, flow rate: 3.18 m/min)

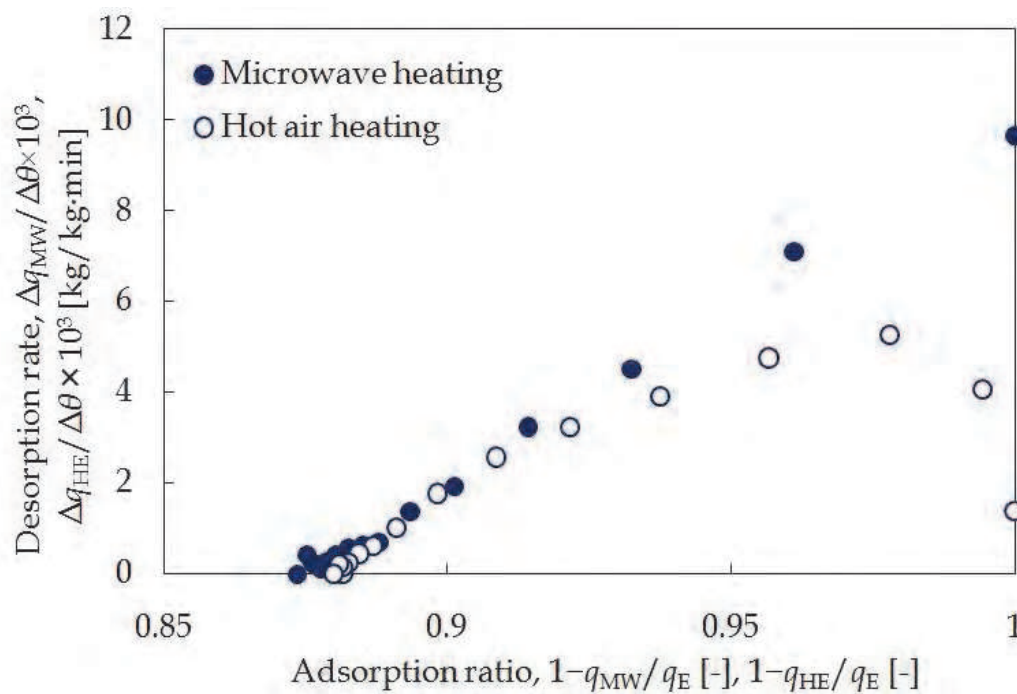


Fig. 10. Relationship of desorption rate and adsorption ratio

3.2 Influence of flow rate and hot air temperature exerted on microwave heating desorption

Desorption ratio (q_{MW}/q_E) and adsorbent bed highest achieving temperature (T_{MAX}) obtained from results of DF-9 under the conditions of three flow rates are shown in Table 2. It's observed that q_{MW} shows the minimum by flow rate and T_{MAX} decreases with increase of flow rate. It is considered as following, i) Desorption rate increases with increase of flow rate and adsorption temperature. ii) The increase of flow rate inhibits the temperature rise of adsorbent bed, and reduces the desorption rate.

Flow rate [m/min]	q_{MW}/q_E [-]	T_{MAX} [°C]
1.62	0.166	58.6
3.18	0.141	51.3
6.36	0.160	46.2

Table 2. Experimental results of desorption ratio and temperature for zeolite DF-9

In order to clarify the influence of hot air temperature exerted on microwave heating desorption (preset temperature: 45°C), adsorbent temperature change and desorption amount change of DF-9 desorption experiment by hot air and microwave hybrid system are shown in Figure 11. The following are observed from this figure.

1. Adsorbent bed centre temperature by hot air heating is almost adjusted to preset temperature. Moreover, adsorbent bed temperature T_{MW} of microwave irradiation heating shows higher than T_{HE} .
2. The maximum amount change of hot air desorption shows up in about 4 minutes, and go back to the initial value in about 17 minutes. Desorption amount change of microwave heating begins to decrease after becoming the maximum in about 2 minutes, and the value is maintained for about 2 minutes longer, and appears larger than the initial value in 17 minutes.

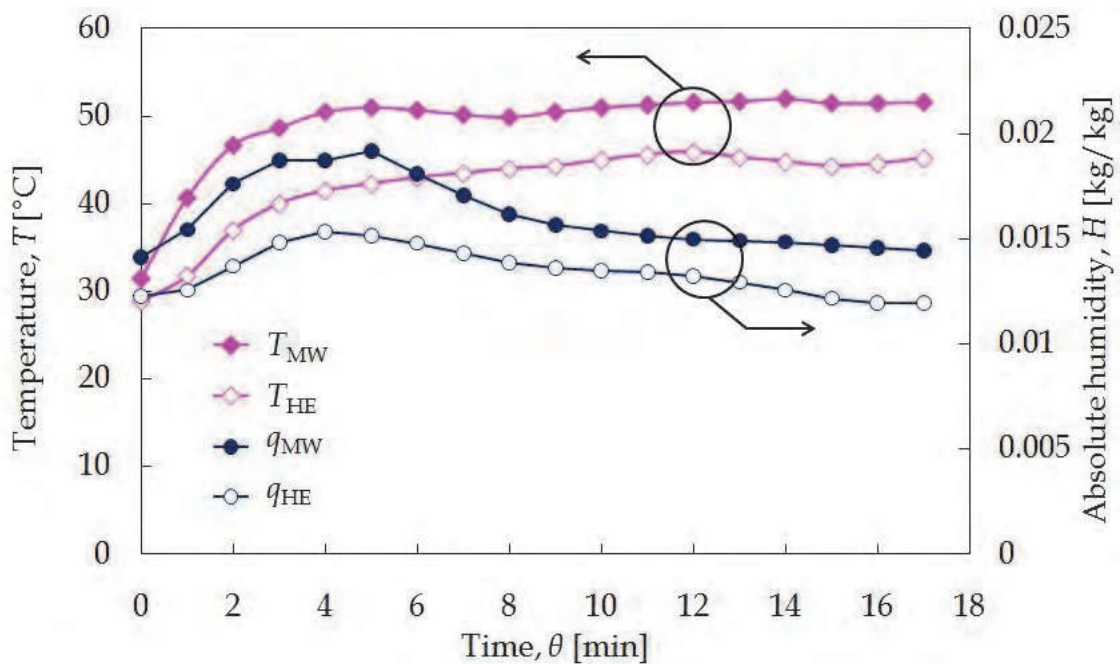


Fig. 11. Changes of absolute humidity and temperature for zeolite DF-9

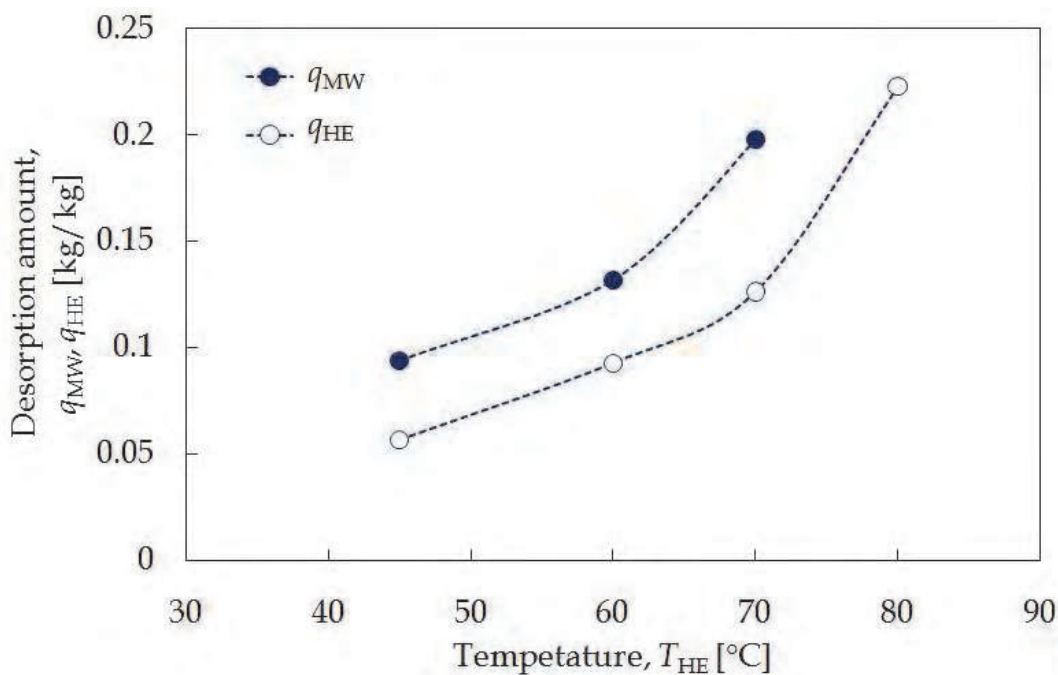


Fig. 12. Relationship between desorption amount and air temperature for zeolite DF-9

These tendencies were similarly observed through changing hot air temperature. Then the q_{MW} and q_{HE} corresponding to adsorbent bed temperature T_{MW} and T_{HE} were calculated and shown in Figure 12 after the desorption started 10 minutes. The temperature of the equal desorption amount is $T_{MW} < T_{HE}$. In addition, the difference of the temperature ΔT ($=T_{HE}-T_{MW}$) is equivalent to the temperature of heat source decrease effect. T_{MW} shows the decrease effect of 16.5°C, 13.2°C and 8.6°C that obtained respectively at 45°C, 60°C and 70°C. Heat source temperature decrease effect decreased with the increase of T_{MW} . The reason is considered as the loss coefficient of the microwave for the water decreased with increase of temperature (Koshijima et al., 2004). Moreover, ΔT is smaller than T_D . This depends on the microwave output being small.

4. Conclusions

The water-vapor desorption characteristics due to microwave heating adsorption equilibrium characteristics and pore size different types of zeolites were evaluated, and the following results were gained.

1. The effect of microwave irradiation was approved to be better than that of hot air heating in any zeolites. This effect is remarkable for zeolite that has small pore size (4A) or large adsorption amount of water-vapor (DF-9). The desorption amount of water-vapor from zeolites by microwave irradiation was 2.22 (4A) and 2.59 (DF-9) times larger than that by hot air heating.
2. The desorption rate increased along with large pore size of zeolites. Moreover, the desorption rate of similarly sized zeolites increased with the increase of the adsorption amount.
3. The desorbed effect of microwave irradiation according to the results of the temperature rise experiments during hot air heating was confirmed as same as the effect of microwave irradiation.

4. The minimum value of desorption ratio appeared with the air flow rate change.
5. On the same desorbed amount standard, the temperature of hot air and microwave irradiation hybrid type is lower than that of hot air heating. The microwave irradiation showed the effect of maximum 16°C decrease of the heat source.

As shown above, in this research, the speeding-up of desorption and the effect of heat source temperature decrease can be confirmed by microwave irradiation.

As challenges for the future research, it is necessary to study the uniform heating method of the adsorbent. So it's necessary to consider the shape of the adsorbent and microwave irradiation method.

When applying for microwave irradiation in the desiccant humidity conditioner, it's necessary to examine microwave irradiation method and the rotor shape etc. In addition, grasp of the microwave irradiation effect by low-humidity environment is necessary for upgrading the desiccant humidity conditioner.

5. Nomenclature

H	absolute humidity	[kg-H ₂ O/kg-dry air]
q	adsorption amount of water	[kg-H ₂ O/kg-zeolite]
q'	desorption amount of water calculated from temperature rise of zeolite bed	[kg-H ₂ O/kg-zeolite]
q_E	equilibrium adsorption amount of water	[kg-H ₂ O/kg-zeolite]
q_{HE}	desorption amount of water by hot air heating	[kg-H ₂ O/kg-zeolite]
q_{MW}	desorption amount of water by microwave heating	[kg-H ₂ O/kg-zeolite]
RH	relative humidity	[%]
R_q	ratio of q_{MW} and q'	[-]
T	temperature	[°C]
T'	temperature of zeolite bed calculated from desorption amount of water	[°C]
T_D	$T' - T_{MW}$	[°C]
T_{MAX}	maximum temperature	[°C]
T_{HE}	temperature of zeolite bed during hot air heating	[°C]
T_{MW}	temperature of zeolite bed during microwave irradiation	[°C]
ΔT	$T_{HE} - T_{MW}$	[°C]
θ	time	[min]

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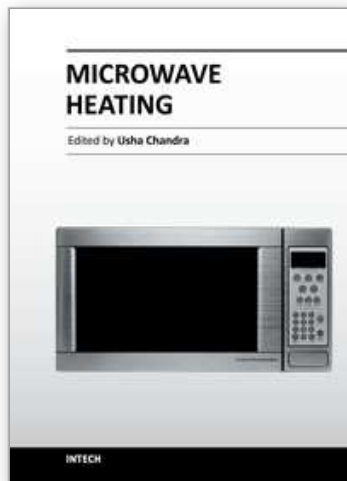
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