

Influence of Microwave Irradiation on Water-vapor Desorption Capacity of Zeolites

Fujio WATANABE[†], Masanobu HASATANI[†], Hongyu. HUANG[†]

Abstract 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. However, the desorption rate decreases and the energy consumption increases in desorption process of D-hum was found. For the efficient development of D-hum, a microwave heating type D-hum was proposed. The microwaves have the characteristics energy is readily transformed into heat inside the particles by dipole rotation and ionic conduction and selectively heats water in the adsorbent. Consequently, processes based on microwave is now considered a promising rapid desorption technology compared to conventional heating. In this study, the effect of heat and microwave irradiation on adsorbed water-vapor desorption from various type zeolites was studied. Desorption performance of water from zeolites by microwave heating under the conditions of N₂ gas of 30°C with relative humidity 40%, microwave power of 800W was compared with that for hot-air heating at 40~80°C and microwave power of 50W. The effect of microwave irradiation was approved to be better than that of hot air heating in any zeolites.

1. INTRODUCTION

The performance of desiccant humidity conditioner is high, and the use of low temperature thermal energy is possible, and really a challenge for compactification. We suggested heat and microwave irradiation type hybrid desiccant humidity conditioner (HM) which enabled sophisticated and high speed desorption. In previous study [1], it 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 usual zeolite 13X. As a result, the desorption speed was admitted to increase five times or less that of warm air heating desorption in microwave heating of the thing and 800W with overplus desorption effect 1.6-2.0 times compared with warm air heating desorption in microwave heating desorption, and availability of desiccant humidity of this method was suggested.

On the other hand, there are various types different in adsorption property in zeolite [2]. This option was performed in desiccant humidity condition. For example, there are 4A and 5A which show adsorption performance is stronger than 13X, DF -9 shows that water vapor adsorption performance is more than normal 13X, etc. These can be applied as adsorbent of desiccant humidity conditioner for the high dew point manufacturing and for great capacity humidity conditioning. Furthermore, sophisticated and high speed desorption is made possible to use as adsorbent of HM system of this study.

[†] Research Institute for Industrial Technology, Aichi Institute of Technology

However, microwave desorption feature of these zeolite is almost unknown. Moreover, 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 about the influence of adsorption equilibrium feature on desorption feature of microwave heating by a zeolite of previous study [1] and two kinds of new zeolites and pore architecture was performed. ii) Experimental study was performed about the influence of gas flow rate exerted on microwave heating desorption attribute and temperature.

2. EXPERIMENTS

2.1 Sample

The zeolite sample (Average particle diameter 500 μ m) used 4A (pore size 0.4nm) and 13X (DF-9; pore size 0.8nm). These adsorption desorption isotherm measured by water vapor adsorption device (Belsorp aqua3, BEL JAPAN, INC.) is combined with the result of 13X (OXYSIV-5; pore size 1.0nm) shown in previous study [1], and it is shown in Figure 1. In 4A, below relative pressure $RH=5\%$ the rise of steep adsorption desorption isotherm is shown, after that the adsorption amount accrete gradually. The adsorption amount less than $RH=5\%$ accounts for 75% of the adsorption amount in $RH=98\%$. In addition, adsorption amount is smaller in comparison with OXYSIV-5, they are respectively 0.95 times

and 0.84 times $RH=5\%$ and 80% standard. In 4A, below relative pressure $RH=10\%$ the rise of steep adsorption desorption isotherm is shown. Afterwards, the increase of adsorption amount almost similar to OXYVIVE-5 is shown. The adsorption amount less than $RH=10\%$ accounts for 84% of the adsorption amount in $RH=98\%$. In addition, adsorption amount is better in comparison with OXYVIVE-5, they are respectively 1.35 times and 1.22 times $RH=10\%$ and 80% standard. The adsorption in the range of $RH=5-80\%$ is large with about 1.3 times of OXYVIVE-5. Although 4A and DF-9 show desorption hysteresis, it is small compared with OXYVIVE-5. In addition, visual check of a sample and measurement of water vapor adsorption isotherm was performed on the zeolite sample which is to be used to describe microwave irradiation experiment. As a result, the damage of the sample by microwave irradiation and the transformation were not observed. Moreover, change of adsorption isotherm by the existence of microwave irradiation was not checked.

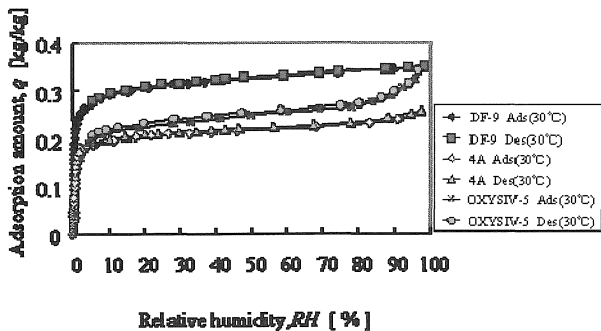


Fig. 1 Adsorption isotherm of water vapor on zeolite at 30°C

2.2 Experimental arrangement and methodology

The experimental arrangement used the microwave irradiation type adsorber which is similar to previous study [1]. The apparatus consisted of a microwave irradiator, a circulated packed adsorption column, an evaporator, a microwave adsorber, a heater and thermometers. The sample area (zeolites grain diameter 0.3-1.0mm, sample layer thickness; approximately 2.5mm (approximately 0.5g)) in adsorption column was put 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 layer upper which is a circulated packed adsorption column, it adjusted adsorbent layer temperature with heating the circulation gas with electric power (Figure 2). In this temperature control, beforehand, it filled up glass sphere in the position where it is

Figure 3 Desorption ratio and temperature of zeolite DF-9 bed during MW heating (MW: 800W, flow rate: 3.18m/min)

suitable to adsorbent layer, under the condition which supplies the circulation gas, the supply capability where packed bed central temperature becomes specified arrival temperature was measured. In the microwave irradiation heating experiment, the microwave irradiation was done with the supply of this measurement electric power. Furthermore, microwave is attenuated by sample distance, but because with the adsorbent layer thickness and fill ration is little, we assumed that most of it is uniformly irradiated without being attenuated.

Experimental method is similar to past research. And, the following two items for desorption experiments were conducted.

1) After reaching adsorption equilibrium in the same conditions as previous study [1] (adsorbent fill ration;

about 0.5g, adsorbent layer thickness; about 2.5mm, flowing gas; 30°C, $RH=40\%$, fluid velocity; 0.81-6.36m/min) in two sorts of new zeolite, a microwave heating desorption experiment with the microwave output 800W is conducted.

2) After adsorption equilibrium attainment like 1) in which a sample uses DF-9, a desorption experiment by microwave output 50W (constant) is made in addition to measured supply of electric power so that circulation gas of fluid velocity 3.18m/min can heat 40 to 80°C of adsorbed layer achieving temperature.

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

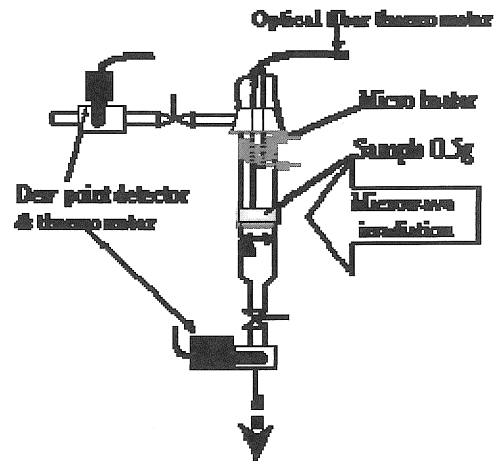


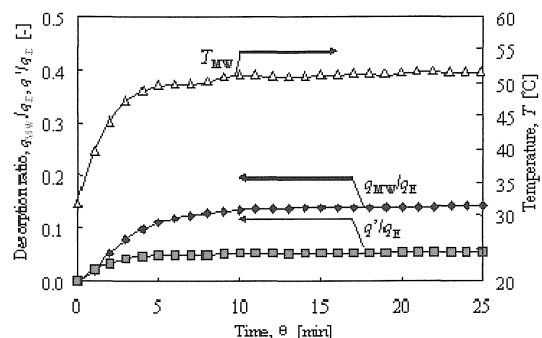
Fig. 2 Schematic diagram of adsorption column

3. EXPERIMENTAL RESULTS AND CONSIDERATION

3.1 Comparing microwave heating desorption effect accelerated by different zeolites

As shown in Figure 3, heat and mass transfer behavior of microwave heating desorption process in adsorption column inlet temperature 30°C and relative humidity $RH=40\%$ that used the zeolite 4A and DF-9 showed similar behavior compared with previous study on zeolite OXYVIVE-5[1]. This behavior confirmed repeatability was shown by repeated experiment. However, by the type of the zeolites, temperature (T_{MW}) rise, maximum achieving temperature (T_{MAX}) of adsorption layer and desorbed amount (q_{MW}) by microwave irradiation heating were different. To compare the difference of desorption by this kind of the zeolite, equilibrium adsorbed amount (q_E) at 30°C, $RH=40\%$, and like previous report,

Table 1 Amount of desorbed water and temperature rise



zeolite	4A	DF-9	OXY SIV-5
q_E [kg-H ₂ O/kg]	0.218	0.322	0.242
q_{MW} [kg-H ₂ O/kg]	0.016	0.044	0.032
q_{MW}/q_E [-]	0.074	0.136	0.132
T_{MAX} [°C]	47.8	50.1	46.7
q' [kg-H ₂ O/kg]	0.007	0.017	0.019
T' [°C]	62.4	93.7	61.4
R_d [-]	2.22	2.59	1.57
T_D [°C]	13.3	43.6	14.7

microwave output 800W, q_{MW} in 15min after desorption begins, desorption ratio (q_{MW}/q_E), T_{MAX} , hypothetical temperature desorbed amount (q'), hypothetical temperature of heat source (T') and hypothetical temperature desorbed amount ratio $R_d(=q_{MW}/q')$, hypothetical temperature of heat source rise $T_D(=T'-T_{MW})$ calculated using adsorbed equilibrium relation of Figure 1 of the various zeolite is shown in Table 1 together with the result of microwave irradiation time 15min in OXY SIV-5 of previous study [1]. Moreover, temperature rise rate ($\Delta T/\Delta \theta$) and desorption rate ($\Delta q_{MW}/\Delta \theta$) of adsorbent layer desorption ratio ($1 - q_{MW}/q_E$) is shown in Figure 4 and Figure 5 respectively. Here, q' and T' are defined as follows like the previous study. In this experiment, adsorbent temperature rises by microwave irradiation. By contrast, adsorbent layer inlet temperature are always constant in 30°C and $RH=40\%$ (absolute humidity: $H=0.0106\text{kg/kg-air}$). Therefore, relative humidity in the layer decreases because adsorption layer temperature rises. q' is adsorbed amount corresponding to this temperature rise on isotherms. Equally, desorbed amount by microwave irradiation to inverse operation was carried out, the temperature required for equilibrium theory equivalent to q_{MW} was decided T' . In this calculation, Clausius-Clapeyron Equation was used in consideration of temperature, dependency of adsorption isotherm based on adsorption isotherm of two temperatures of various zeolites.

The following was observed from Table 1, Figures 4 and 5. 1) Largeness of q_{MW} is DF-9 > OXY SIV-5 > 4A, and so 4A, OXY SIV-5 are respectively 0.36 times and 0.73 times DF-9. 2) Largeness of q_{MW}/q_E is also DF-9 > OXY SIV-5 > 4A. This value was slightly smaller than DF-9 in OXY SIV-5. In contrast, 4A was 0.54 times of DF-9. 3) T_{MAX} is DF-9 > 4A > OXY SIV-5. 4) R_d shows more than 1, and 4A, DF-9 was about 1.4 times and 1.6 times of OXY SIV-5. T_D of 4A and DF-9 is about 0.9 times and 3.0 times of OXY SIV-5. 5) The ascent rate of temperature ($\Delta T/\Delta \theta$) is a little different by the type of the zeolites in desorption early time, but there is no big difference in change. On the other hand, desorption rate decreases ($1 - q_{MW}/q_E$) showing the maximum by 0.98-1.0 in DF-9 and 4A, and decreasing in ($1 - q_{MW}/q_E$) afterwards. Moreover, maximum of desorption rate and desorption rate after maximum desorption rate attainment are the following order, DF-9 > OXY SIV-5 > 4A.

The above mentioned results 1) supports equilibrium adsorbed amount under the conditions given, in this experiment in range, the adsorbent with a little equilibrium adsorbed amount shows that q_{MW} becomes small. By the way, equilibrium adsorbed amount in OXY SIV-5 is 0.76 times when based on 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 DF-9, q_{MW} decreases greatly with 0.36 times. If it takes that into consideration this will not make a little difference in the rate of temperature rise of adsorbent (Figure 4.) and achieving temperature of 15 minute later by microwave heating, it is thought that it is influenced by pore size which is one of dominant factor of water vapor adsorption property in range of this experiment. This is shown by a result

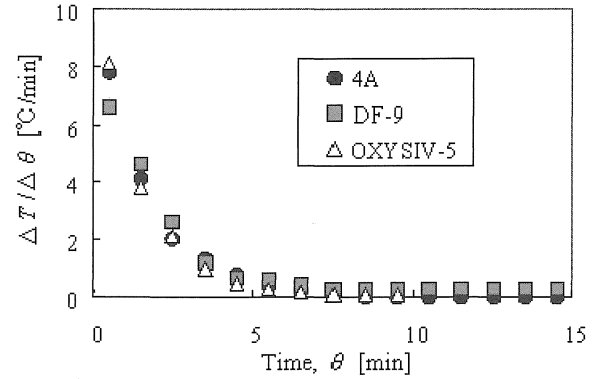


Fig. 4 Rate of temperature rise for zeolite beds

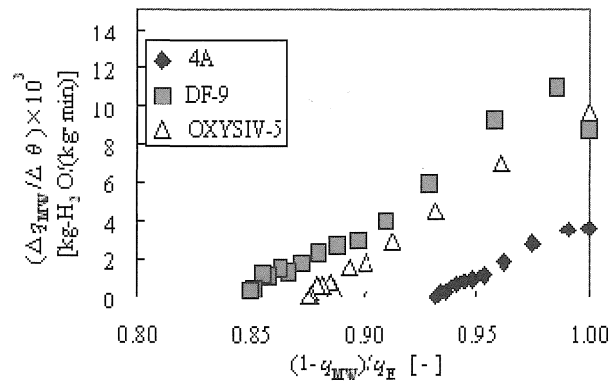


Fig. 5 Relationship between desorption rate and adsorbed ratio

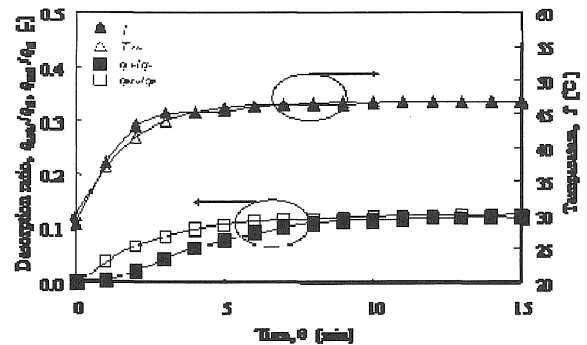


Fig. 6 Desorption ratio and temperature during MW irradiation and heating for zeolite DF-9 (MW: 800W, flow rate: 3.18 m/min)

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

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

of 5), compared with DF-9 and OXY SIV-5 of pore size 0.8nm and 1.0nm, maximum desorption rate of 4A of pore size 0.4nm is confirmed, respectively because they are about 0.33 times and 0.38 times. On the other hand, pore size of DF-9 is slightly small in comparison with OXY SIV-5, desorption rate depends on adsorbed amount in the range of pore size of this level, and it is thought that equilibrium adsorbed amount grew large in large adsorbent.

As for the result of R_d of 4), excess desorption effect arises in the same way as OXY SIV-5 with 4A and DF-9 that we used by this experiment, and the effect shows a different thing by the type of the zeolites. Concretely, R_d is 1.4 times while beginning adsorbed amount of 4A when the experiment

condition is 0.88 times OXYIVE-5 comparing it with OXYIVE-5 and 4A. This shows that desorption by microwave heating is more advantageous than warm air heating in zeolite with small pore size. On the other hand, by DF-9, the beginning adsorbed amount is 1.3 times compared with OXYIVE-5, R_d is 1.6 times. This shows that microwave heating is effective in desorption of zeolite which is adsorbing water vapor in large quantity.

Next to confirm excess desorption effect by the microwave more clearly, another outcome of an experiment by OXYIVE-5 is shown in Figure 6. This figure shows adsorbent layer temperature and the desorbed amount change in desorption of microwave power 800W and warm air heating desorption at adsorption tube inlet temperature 30°C, $RH=40\%$. T_{MW} in figure is adsorbent layer temperature by microwave heating. In addition, T_{HE} is adsorbent layer temperature under the warm air supply condition that performed warm air heating to show increased temperature as same as T_{MW} . T_{HE} and T_{MW} almost draw the same curve according to figure. On the other hand, desorption ratio of microwave heating (q_{MW}/q_E) is larger than desorption ratio of warm air heating (q_{HE}/q_E) at all time. It is especially remarkable in beginning desorption. As for this, microwave heating causes excess desorption more than warm air heating desorption. It is shown that it is an effective under a condition with much adsorption water and bear out a result of Figure 3. T_D of 3) shows effect of decreasing of temperature of heat source 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 desorbed amount when desorption of warm air temperature 93.7°C uses warm air temperature 50.1°C together with microwave heating, and the decrease of 43.6°C of temperature of heat source becomes possible.

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

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

In order to clarify influence of warm air temperature exerted on microwave heating desorption and warm air desorption according to warm air heating (preset achieving temperature; 45°C) about DF-9, adsorbent layer temperature alteration and desorbed amount alteration of desorption experiment by warm air and microwave combined are shown in Figure 7. The following are observed from this figure. 1) Adsorbent layer center temperature by warm air heating is almost adjusted to preset achieving temperature. Moreover, adsorbent layer temperature T_{MW} of microwave irradiation heating shows more than T_{HE} . 2) The maximum amount change of warm air desorption shows up in about three 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 three minutes, and the value is maintained for about two minutes longer, and appears larger than the initial value in 17 minutes. This tendency was similarly observed through changed warm air temperature. Then, in search of q_{MW} and q_{HE} for adsorbent layer temperature T_{MW} and T_{HE} which is based on 17 minutes after the desorption starts, the relation is shown in Figure 8. The standard of Temperature of the equal desorbed amount is $T_{MW} < T_{HE}$ without depending on warm air temperature. In addition, the difference of the temperature $\Delta T (= T_{HE} - T_{MW})$

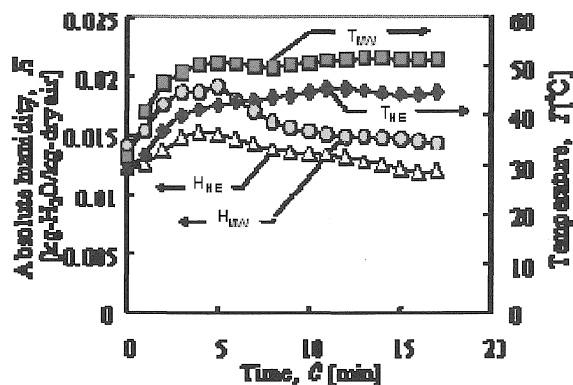


Fig. 7 Changes of absolute humidity and temperature for zeolite DF-9

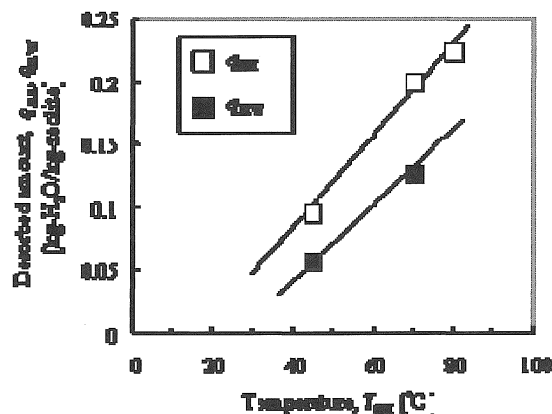


Fig. 8 Relationship between desorbed amount and air temperature for zeolite DF-9

is equivalent to the temperature of heat source effect of decreasing, T_{MW} shows that effect of decreasing of 16.5°C, 13.2°C and 8.6°C is obtained respectively at 45°C, 60°C and 70°C. It depends on loss coefficient for the water of the microwave shrinking with increased temperature that temperature of heat source effect of decreasing shrinks with increasing of T_{MW} [3]. 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 appraised, 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 zeolites that has small pore size (4A) or large adsorbed amount (13X (DF-9)). The amount of water desorbed from zeolite by microwave irradiation was 2.22 (4A) and 2.59 (13X (DF-9)) times larger than by hot air heating. This amount corresponded to that obtained by hot-air heating at 13–43°C higher than the zeolite bed temperature.
- 2) The desorption rate increased along with the increasing of the particle size of the zeolites. Moreover, the desorption rate of same diameter zeolites increased along with the increasing of the adsorbed amount.
- 3) The desorbed effect of microwave irradiation according to the results of the temperature rise experiments during hot air heating was as same as the effect of microwave irradiation.

4) The minimum value of desorption ratio appeared with the air flow rate change.

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.

NOMENCLATURE

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

REFERENCES

- [1] M. Saitake, M. Kubota, F. Watanabe and H. Matsuda: "Enhancement of Water Desorption from Zeolite by Microwave Irradiation" (in Japanese), Kagakukougaku ronbunshu, 33, 53-57 (2007).
- [2] H. Yoshida, "Handbook on Porous Adsorbents" (in Japanese), pp.190-193, Fuji Technosystem, Tokyo, Japan (2005).
- [3] T. Koshijima, C. Shibata, T. Toishi, K. Norimoto and S. Yamada: Microwave Heating Technology Collection (Maikuroha Kanetsu gijutsu Shusei), p.9, NTS Inc., Tokyo, Japan (2004).