Canadian Fusion Fuel Technology Project

Microwave Regeneration
Of
Molecular Sieves

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1.0 SUMMARY

Molecular sieve driers are being used for removing tritiated water from the atmosphere in CANDU (Canada Deuterium Uranium) generating stations and have also been included in the design of tritium handling systems for fusion reactors. In these fusion reactor systems, there is a need to maintain extremely low exit dew points from the driers as well as a capability to rapidly reduce tritium concentrations following an accident. Thus the required capacity of the driers is very high resulting in high capital and operating costs. The conventional method of regenerating these sieves after a water adsorption cycle is with hot air at about 260°C (thermal regeneration). However, because water is rapidly heated by microwave energy, this technology may be suitable for decreasing the bed regeneration time and hence may allow reduced capital and operating costs associated with a smaller bed. The present study was very limited in scope and was conducted to obtain preliminary information on the technical feasibility of regenerating molecular sieves with microwave energy. The study concentrated on Type 4A molecular sieve with a few tests on Type 13X sieve and also a silica gel adsorbent.

A parallel study carried out under this contract at the University of New Brunswick indicated that a temperature of about 350°C was necessary for efficient drying to the low dew points required. Such temperatures and even up to 500°C were easily produced with microwave heating. Very limited aging tests (six cycles) indicated no detectable reduction in the adsorption capacity of sieves regenerated at 350°C. However, temperature measurement in a microwave field and temperature non-uniformity along the length of the bed were major problem areas.

The temperature non-uniformity resulted in undesirably high moisture contents (~4 g H_2O/100 g sieve) at the low temperature regions of the bed but acceptably low values (~0.2 g H_2O/100 g sieve) at locations where the temperature was about 350°C. The molecular sieves appeared to absorb a significant amount of microwave radiation at the frequency investigated, 2450 MHz. This resulted in continued heating of the sieves which is likely not attributable to the residual moisture content which was very low (<0.1 gH_2O/100 g sieve).

The temperature non-uniformity in the bed was also dependent on the regeneration airflow rate. Relatively high airflow rates (~0.15 m/s used in thermal regeneration) resulted in sub-cooling and high residual moisture content at the inlet of the bed. Lower airflow rates resulted in higher inlet temperatures. However, high residual moisture contents were obtained at the bottom of the bed even though the temperature there was in the 300 - 350°C range. This was probably due to the high water concentration in the flowing gas stream during the approximately twenty minute regeneration period.

The tests on silica gel showed significantly less microwave absorption because of its non-crystalline structure and the temperature did not exceed 170°C. However, a preliminary comparison shows that for a typical fusion air processing system, a silica gel bed must be about four to eight times larger than a bed of type 4A molecular sieve to achieve the same performance.

It is recommended that a preliminary estimate be made to compare the costs of a conceptual microwave regenerated system with that of a conventional thermal regeneration system. Additional testing at other microwave frequencies and at adequate power levels should also be carried out together with an investigation on the measurement of temperature in a microwave field.

2.0 INTRODUCTION

Molecular sieve driers will be required for first generation fusion reactors to reduce tritium emissions to the environment during normal operations as well as following an accident. Such driers are presently being used in CANDU stations to limit in-station tritium exposure, heavy water losses and tritium emissions. Because of the need to maintain extremely low exit dew points from driers operating in a fusion air processing system as well as the capability to rapidly reduce tritium concentrations following an accident, the installed capacity of the driers is very high resulting in high capital and operating costs.

A large part of these costs is a result of the time required to regenerate the sieves off-line. The sieves are usually regenerated after an in-service time

period (or if the exit dew point increases beyond a set value) by passing air, heated to about 260°C through the bed. If the time for the thermal regeneration could be reduced, the drier beds could be made smaller for a given duty resulting in possible capital cost savings.

Since water is rapidly heated by microwave energy, this technology may be suitable for decreasing the bed regeneration time. The objective of this study was to obtain preliminary information on the technical feasibility of regenerating molecular sieves with microwave energy. Although molecular sieve driers are used in several different parts of the tritium handling systems for fusion reactors, each having its own performance requirements, the emphasis in this study was placed on the air processing system.

Additional background material on this project is presented in Section 3 and details of the experimental equipment and the results are given in Section 4 and 5. A discussion of the results follows in Section 6.

3.0 BACKGROUND

Design criteria for an air processing facility to remove tritiated water vapour from a fusion reactor containment system have been proposed in a McDonnell Douglas draft report (1). These criteria call for the use of deep bed (0.75 m) driers, containment dew points of -18°C and recirculation dryer exhaust dew point of -60°C under chronic leakage conditions. Adsorption of moisture on molecular sieve adsorbents has been widely studied at higher humidity levels but at the levels being considered above the available information is fragmentary and often contradictory. Hence, as part of this contract, a study on the low moisture level performance of molecular sieves was carried out at the University of New Brunswick (UNB) concurrently with the present investigation. The UNB results are reported elsewhere (2), but the results have been incorporated where appropriate in this report.

4.0 EXPERIMENTAL DETAILS

Experimental investigations of the amount of water removed from the adsorbent as heating time was increased were carried out using both microwave and conventional thermal regeneration. The latter thermal regeneration tests were carried out for preliminary comparative purposes and the results are not representative of the full scale performance because of almost isothermal operation for small beds while full scale beds operate almost adiabatically. Adsorbent materials investigated included Types 4A and 13X molecular sieve extrudates (3.1 mm) and a silica gel. Limited aging tests were performed after it was determined from the UNB studies that high regeneration temperatures were required to reduce the residual moisture contents in the sieves to very low levels for efficient drying to -60°C dew point and to obtain reasonable bed capacities. These were done by repeatedly heating the sieves to high temperatures in the microwave oven and then determining the capacity over the adsorption phase. The tests reported in this work were carried out on small adsorbent samples (80 g) and on larger samples (1000 g) which occupied a significant space in the microwave oven.

A schematic of the experimental equipment is shown on Figure 1. The microwave oven was a domestic type (Amana Model RRL-8TD) operating at a frequency of 2450 MHz with a total power consumption of 1450 W. Maximum available microwave power estimated by monitoring the time for a known quantity of water to boil in the oven was 550 W which is comparable to the 700 W given on the manufacturer's literature. The oven had a variable power input from 10 to 100% of maximum power level and a rotating antenna to minimize microwave field non-uniformity. It was placed on its side to utilize the maximum head room available and holes (1.25 cm dia) were drilled into the cavity wall to allow inlet and outlet gas lines to be fitted to the column. Suitable wire mesh was used at the penetrations to prevent microwave leakage.

The experimental equipment in the oven was limited to glass or rubber since metal in the microwave field (except at the boundary walls) absorbs microwave energy. Hence, temperature measurement with metal thermocouples was not possible during the time period the oven was in operation. Because of the preliminary nature of the work, temperatures were measured simply by inserting

metal thermocouples at appropriate times into the molecular sieve bed after disabling power to the microwave oven.

The absorbent beds were housed in vertical cylinders and humidification was carried out with upflow while regeneration was carried out with downflow. The thermal regeneration tests were carried out with the column well insulated with fibreglass wool to attempt to simulate more closely the adiabatic conditions in full scale drier beds.

5.0 RESULTS

5.1 Bench Scale Tests - Water Removal Efficiency and Adsorbent Temperature Rise

Microwave regeneration tests were carried out on 80 g samples of 4A and 13X sieves and a silica gel that had been saturated in a 20°C dew point airstream. The regeneration was carried out with the oven operating at full power using dry (10 $\mu L H_2 0/L$) air and at a flow rate of 0.14 m/s. As shown in Figure 2, about ninety-two per cent of the moisture in these small sieve beds was removed after about 6 min (residual moisture ~1.8 g/100 g sieve assuming regeneration at 350°C, for two hours using air containing 10 $\mu L H_2 0/L$). Similarly, Figure 3 shows that almost all of the moisture was evaporated from the silica gel with the temperature rising to about 170°C in ten minutes. However, as shown on Figure 4, the temperature of the molecular sieves rose to higher values in the range 400 to 500°C. These temperatures are much higher than those achieved during conventional regeneration of molecular sieves dryers (~260°C) and could cause significant sintering of the sieve. The 4A grade sieve was heated to somewhat lower temperatures than the 13X sieve. Previous studies (3) showed that the 4A sieve appeared to be more resistant to aging, ie degradation of its drying performance after repeated adsorption/regeneration cycles and testing was therefore continued on the 4A grade sieve.

5.2 Bench Scale Tests - 4A Sieve Aging Characteristics

University of New Brunswick studies (?) indicated that for the required design exit dew point of -60°C (≡ 10 μL H_2O/L), the water content of the 4A sieve is only about 4 g H_2O/100 g sieve. Higher than conventional regeneration temperatures, such as obtained with microwave regeneration, may be desirable to utilize most of the capacity of the sieve. However, because of the possibility of increased aging rates at the higher temperatures obtained with microwave regeneration, limited aging tests were carried out by first loading the sieves using a humidified airstream and then regenerating in the microwave oven. The procedure was repeated for a total of six adsorption/regeneration cycles. The test conditions were similar to those used in our stations with adsorption at 0.4 m/s with humidified air (0°C dew point) until the air exiting the bed exhibited a -31°C dew point. (Under chronic leakage conditions, the design criteria for fusion system containment dew point and dryer exit dew point are -18°C and -60°C, respectively; however, the above values approximate conditions in our nuclear generating stations and were used to reduce the time required for loading moisture on the sieves and to facilitate measurement of dryer exit humidities).

Regeneration was carried out for 5 min using dry air (-60°C dew point) at a flow rate of 0.14 m/s. Under these conditions, the temperature of the molecular sieve (measured at one point in the middle) rose to 300-350°C. As shown in Table 1, Column 2, the weight of the 4A sieve after microwave regeneration and reloading with water was almost constant showing no great loss in adsorption capacity over these limited cycling tests. The small weight increase is believed to be due to excessive oil present during these tests only in the service air line following maintenance of the compressed air supply system.

5.3 Bench Scale Tests - Thermal Regeneration

In order to demonstrate how the time for regeneration was dependent on the amount of regeneration power available, thermal regeneration tests

carried out on an 80 g sample of 4A molecular sieve at an entering air temperature of 320°C and flow rate of 0.15 m/s. The results are presented in Table 2 which shows that for the low residual moisture contents required, the present bench scale microwave regenerated system had a greater capacity than the thermally regenerated one. This is largely due to the short microwave heating time resulting from all of the microwave power (550 W) being concentrated on a relatively small bed (cf thermal regeneration power input with the hot air is about 100 W). Also apparent from Table 2 is that the rapid attainment of 320°C in the microwave regeneration results in residual moisture contents of about 0.2 g H_2O/100 g sieve. These results show that diffusion of water vapour from the interior of the molecular sieve pellet and its removal at the 0.15 m/s purge gas rate used are sufficiently fast for rapid attainment of low moisture contents and that a large microwave power input to bed size ratio is desirable.

5.4 Semi-Pilot Scale Tests - Continuous
Full Power Microwave Regeneration

Microwave regeneration tests were repeated on a larger (1 kg) sample of 4A sieve which was humidified by passing air (0°C dew point) at 0.4 m/s until the exiting air dew point increased to -31°C. As shown in Figure 5, at the normal thermal regeneration design velocity of 0.14 m/s, the bed moisture was reduced from 13.2 g H_2O/100 g sieve to about 4 g H_2O/100 g sieve in about 20 min with continuous full power input. Unlike thermal regeneration, the regeneration air with microwave was not preheated before introduction to the bed.

5.5 Semi-Pilot Scale Tests -
Effect of Regeneration Airflow Rate

As shown in Table 3 overall average residual moisture was high (about 4 g H_2O/100 g sieve) at the end of the twenty minutes microwave heating period at the normal regeneration flow of 0.14 m/s. Limited temperature measurements in the bed indicated similar temperatures at the

vessel wall and at the centre of the bed. However, while the temperature at the middle and lower portion of the bed attained acceptable values, the upper portion of the bed reached only 110°C. High residual water content of about 4 g/100 g was measured in material from the top of the bed while the corresponding value measured at the bottom of the bed was ~0.3 g/100 g. A heat balance based on the microwave energy input (550 W) indicates that if all the energy goes into heating the regeneration air, the exit temperature will be about 300°C which is similar to the temperature measured at the lower portion of the bed. It appears therefore, that high regeneration flows maintain low temperature at the bed inlet and also limit the temperature at the lower portion resulting in high residual moisture in the bed. This should be considered in the sizing of the microwave power supply.

As shown in Table 3, at lower regeneration flows the temperature tended to be more uniform while at very low flows the temperature at the upper portion of the bed increased to unacceptably high values. In all cases, residual moisture contents were reduced with decreasing flow rate. It is possible that at the very low flow rates where little heat is removed by the regeneration air, the upper portion of the bed is heating up rapidly since the amount of water there in the length of unused bed, is small. Also at the very low flow rates not enough heat is removed by the regeneration air from the upper portion to heat up the lower portion.

As also shown in Table 3, residual moisture content was reduced with decreasing flow rate. However, while high temperatures were achieved, the residual moisture did not fall below 1 g/100 g sieve and this is discussed further below.

5.6 Semi-Pilot Scale Tests - Extended Heating Times and Temperature Control

As indicated above, the University of New Brunswick studies showed that for the dryer design exiting dew point of -60°C (≡10 μL H_2O/L) the capacity of the 4A sieve was only about 4 g H_2O/100 g sieve and that a significant amount of time was required for removal of the last traces of moisture from the sieve. In order to utilize most of the capacity of the sieve, it is desirable to regenerate to very low residual water contents. Attempts were therefore made to maintain the temperature of the molecular sieve in the range 300 to 400°C by adjusting the power input level of the microwave oven over two extended regeneration time periods. Since temperature measurement during the microwave on-time was not possible, the power supply (and airflow) were switched off intermittently for thermocouple insertion and temperature measurement after which the power level was adjusted to obtain the desired temperature. As shown in Table 4, only limited success was achieved in maintaining 300-400°C and the temperature was lower than the desired value at the top, and higher at the bottom of the bed. Although high temperatures were realized (cf thermal regeneration results, below), the moisture content of the bed did not fall below 1.5 g H_2O/100 g sieve and this is discussed below.

5.7 Semi-Pilot Scale Tests - Thermal Regeneration

Thermal regeneration tests were also carried out on the 1 kg bed, for comparison with the microwave regeneration tests and the results are given in Table 5. The airflow rate (0.14 m/s) was similar to the normal design value but the regeneration air temperature was increased from 260 to 325°C (equivalent to 670 W input to heat the regeneration air from 25 to 325°C). The thermal regeneration data of Table 4 indicate a very low residual moisture content of 0.3 g H_2O/100 g sieve was possible after 40 min. Also a residual moisture of about 1.5 g H_2O/100 g was obtained at 25 min (interpolation of 20 and 30 min data)

with the bed temperature in the range 110 to 325°C at the end of the heating period. (Note that in the full scale system, these times could be longer due to the lag in system heat up).

Comparison with the microwave regeneration data of Figure 5, Table 3 and Table 4 indicates that although much higher temperatures, in the range 200-450°C, were achieved for similar time periods during the microwave regeneration of the bed, the residual moisture was not appreciably lower. This may be due to two factors. First, the lower flow velocities used during microwave regeneration reduced the rate of moisture removal from the external film by increasing its thickness and in particular the concentration of water vapour there (even at the normal regeneration flow rate of 0.14 m/s, if the upper part of the bed is drying at a relatively slow rate of 0.1 g H_2O/100 g sieve/min or only 1.5 g H_2O/100 g sieve over fifteen minutes, the water vapour concentration in the regeneration airstream will be about 1300 µL/L; at this water vapour level, the lower portion of the bed even though its temperature is as high as 350°C will contain as much as 1.5 g H_2O/100 g sieve). Second, the power input during the microwave regeneration was less than 550 W because of the 400°C temperature limitation while power during thermal regeneration was about 670 W.

Also shown in Table 5 are the times for the mid-point bed temperature and the exit air temperature to fall to 60°C. These values may be used to estimate the total regeneration cycle time and hence the bed capacity. The temperature to which the bed should be cooled before return to service was given at about 60°C in a McDonnell Douglas draft report (1). However, this figure may be revised after further consideration of the position at which the temperature is measured and the low residual water content required when the bed is returned to service.

6.0 DISCUSSION

6.1 Molecular Sieve Regeneration

The major problems with microwave regeneration of molecular sieves in the configuration and at the microwave frequency tested were temperature measurement and temperature non-uniformity. While the temperature measurement method used in this study was adequate for preliminary purposes, it is not suitable for a practical system. An investigation on the availability of suitable devices for temperature measurement (up to 450°C) in a microwave field was beyond the scope of this work. Additional work on the availability and, if necessary development of these devices is required if this technology is to be used in a practical drier system.

At the initiation of the project it was hoped that the water would rapidly absorb the microwave energy without appreciable absorption of the microwave energy by the sieve. However, it was found that the sieves rapidly absorbed the radiation. This resulted in continued heating of portions of the bed which were already essentially dried. Investigation of other microwave frequencies is recommended.

Adjustment of the regeneration flow rate can reduce the temperature non-uniformity as shown in Table 3. However, too low a flow results in undesirably high inlet temperature and slow moisture removal from the bottom of the bed because of the high water concentrations in the gas there. On the other hand, too high a flow results in sub-cooling at the inlet of the bed and therefore, high residual moisture there. Specifically, the normal thermal regeneration purge rate of 0.14 m/s produced sub-cooling at the inlet portion of the bed at a microwave power input similar to the power input during thermal regeneration. This may be reduced to an acceptable level if the microwave power supply is adequately large. This was shown by the bench scale tests (550 W input to 80 g bed rather than 1 kg bed) in which acceptable residual water contents were obtained but confirmation on a larger scale is necessary.

From the bench and semi-pilot scale studies, it would appear that scale-up may be readily accomplished with the usual mass and heat balances. Other methods of operation are also possible. One possible method is to dry the upper part of the bed for five minutes at a low flow rate while limiting the temperature particularly at the top of the bed to less than 325°C. Following this, the flow rate is increased for another five minutes to dry the lower half. Another method is to supply enough microwave power to rapidly heat the bed to exceed 325°C at all positions at a sufficiently high purge rate and accept the increased aging rate at the high temperature areas. Further experimental testing is recommended particularly on bed lengths that approximate the full scale values, with a microwave power supply which can deliver the required energy in a short time period, and possibly, with a preheat inert bed to reduce sub-cooling at the inlet. Temperature control through local microwave power input, purge air additions at intervals along the column or steadily increasing purge rates may also be necessary.

Apart from the temperature measurement and control problems, at 2450 MHz and similar thermal and microwave (as opposed to the total electrical) power input capabilities, a microwave molecular sieve regeneration system does not appear to have significantly increased capacity as compared to a thermally regenerated one. Although, as discussed above, faster microwave drying is possible if the power source is large, a larger condenser may be required.

It is recommended that a preliminary analysis be carried out to compare the costs of a conventional thermal regeneration system operating at high temperatures with that of a microwave regenerated system of higher power capability and larger condenser size assuming suitable temperature measurement and flow control subsystems.

6.2 Silica Gel Performance

The bench scale tests showed that the silica gel absorbed considerably less energy than the molecular sieves because of its noncrystalline structure and the temperature did not exceed 170°C. Under these

conditions no sintering of the silica gel micropores with concomitant loss of adsorption capacity will occur. A comparison may therefore be made on the use of silica gel and 4A sieve in the recirculation dryers (as opposed to exhaust dryers) under chronic leakage conditions and neglecting the length of unused bed. Assuming static isotherm data, no swamping (humidification of the entering gas stream) of the silica gel bed and microwave regeneration times of 40 min for the silica gel and 45 min for the molecular sieve, it is estimated that the silica gel bed will be about four times larger than the molecular sieve bed. This is due mainly to its much lower assumed capacity for the -18°C entering and the -60°C exit dew points. Similarly, if the entering gas stream is swamped to a 15°C dew point, the size of the silica gel bed will be about eight times larger than the molecular sieve bed.

7.0 CONCLUSIONS

1. The required high temperatures for efficient regeneration of molecular sieves are easily acheived with microwave heating. However, temperature measurement in a microwave field and temperature non-uniformity were the major problem areas associated with microwave regeneration of molecular sieves in the configuration studied.

2. Limited cycling tests showed no significant loss of adsorption capacity at the high regeneration temperatures required for low humidity operation.

3. The molecular sieves themselves absorbed microwave radiation at the 2450 MHz frequency used and this contributed to temperature non-uniformity and high residual moisture at the relatively cool portions of the bed. Type 4A sieves were heated to slightly lower temperatures as compared to 13X.

4. Silica gel adsorbent was heated to significantly lower temperatures than the molecular sieves but the required bed size for the same duty is about four to eight times greater than for type 4A molecular sieve.

5. Low moisture purge rates during reasonably short (20 min) regeneration time periods resulted in high concentrations of water vapour near the lower portion of the bed and retarded the removal of moisture from adsorbent material there, even though the temperature was acceptably high. High moisture purge rates resulted in unacceptably low temperatures and high moisture contents at the bed inlet.

6. At similar thermal and microwave power inputs, a microwave molecular sieve regeneration system operating at 2450 MHz does not appear to have a significantly increased capacity over a thermally regenerated one.

8.0 RECOMMENDATIONS

It is recommended that (i) additional work be performed to determine molecular sieve energy adsorption at other microwave frequencies (ii) a preliminary cost comparison be made between microwave regenerated and thermally regenerated systems and (iii) if microwave regeneration is found to show potential for significant cost benefits, further testing at adequate power levels and with sufficiently large beds be carried out together with an investigation of devices for temperature measurement in a microwave field and extensive laboratory testing of microwave regenerated dessicant crush strengths and attrition .

9.0 REFERENCES

(1) Scott, F.R., Defreece, D.A., Waganer, L.M., Zuckerman, D.S., "Assessment of Technical Risks and R&D Requirements for a Magnetic Confinement Fusion System". Study in progress for the Electric Power Research Institute, Project RP1969.

(2) Ruthven, D.M., "Adsorption of Moisture on Molecular Sieve Adsorbents at Low Humidity", Report Prepared for Ontario Hydro Chemical Research Department, File 836.3332, June (1983).

(3) Ruthven, D.M., "Technical Evaluation of Molecular Sieve Adsorbents", Report Prepared for Ontario Hydro Chemical Research Dept, File 836.3332, May (1981).

APPENDIX A

Experimental Results

TABLE 1

Effect of High Temperature on Bed Capacity

CYCLE NO	SIEVE WEIGHT AFTER H_2O ADSORPTION** (g)	TEMPERATURE AFTER FIVE MIN MICROWAVE HEATING (°C)
1	89.0*	350
2	89.3	350
3	89.2	305
4	89.3	320
5	89.4	310
6	89.6	340

* Initial wt of 4A Sieve = 82.2 g.
** To Exit Dew Point of -31°C.

TABLE 2

Capacity of Microwave and Thermally Regenerated 4A Sieve Beds (80 g)

	MICROWAVE REGENERATION	THERMAL REGENERATION (320°C inlet air temp)
Heating time (min)	5	15
Cooling time (min)*	10	9.5
Sieve water content before regeneration (g H_2O/100 g sieve)	8.0**	7.7**
Water removed per cycle (g H_2O/100 g sieve)	7.8	7.4
Capacity (g H_2O/100 g sieve/h)	31.3	18.1

* To 60°C bed temperature at mid-point.

** Values are lower than equilibrium capacity because of the length of unused bed.

Regeneration Flow 0.15 m/s.

TABLE 3

Effect of Regeneration Flow Rate on Bed Temperature Distribution

REGENERATION FLOW RATE (m/s)	RESIDUAL WATER CONTENT (g H_2O/100 g sieve)	BED TEMPERATURE AT END OF HEATING PERIOD (°C) DISTANCE FROM BED INLET (% OF BED LENGTH)		
		25	50	75
0.14	4.2	110	275	305
0.066	2.0	400	400	400
0.044	1.9	410	400	300
0.022	1.0	450	320	320

Conditions: Power setting 100%; microwave heating time ~19.5 min; initial moisture ~13.2 g/100 g. 4A sieve; 12 cm dia bed x 12.5 cm long. Type 4A Union Carbide sieves activated at 350°C with dry (10 µL H_2O/L) air for 2 h.

TABLE 4

Effect of Extended Heating Period on Residual Water Content

MICROWAVE POWER ON TIME (min) AT POWER LEVEL (%)	REGENERATION FLOW RATE (m/s)	BED TEMPERATURE RANGE DURING HEATING PERIOD (°C) DISTANCE FROM BED INLET (%)			RESIDUAL WATER CONTENT g/100 g sieve
		25%	50%	75%	
16 min/100% 5/70 4/50 25 min total*	0.066	25-375 375-320 320-370	25-335 335-415 415-440	25-315 315-375 375-450	1.7
15/100 15/50 9/15 39 min total**	0.044	25-200 200-300 300-170	25-290 290-420 420-325	25-230 230-450 450-380	1.5

Conditions: Initial water content ~13.2 g H_2O/100 g sieve; Type 4A Union Carbide sieves; 12 cm dia bed x 12.5 cm long

* Time to measure temperatures which is not included ~ 6 min

** Time to measure temperatures which is not included ~ 18 min

TABLE 5

Temperature Profiles and Residual Water Content in a Thermally Regenerated Bed

HOT REGENERATION AIR PASSAGE TIME (min)	RESIDUAL WATER CONTENT IN SIEVE (g H_2O/100g sieve)	ENTERING AIR TEMPERATURE TO BED TOP (°C)	TEMPERATURE AT END OF HEATING PERIOD		COOLING OF BED AFTER REGENERATION		
			MID-POINT OF BED (°C)	EXIT OF BED (°C)	TIME FOR 60°C MID-POINT BED TEMPERATURE (min)	*EXIT AIR TEMP* WHEN MID-POINT BED TEMP IS 60°C (°C)	TIME FOR 60°C EXIT AIR TEMP (min)
20	2.2	325	305	98	8	140	19
30	0.94	325	305	120	11	145	16
40	0.34	325	310	243	9	140	15

Conditions: Initial water content ~13.2 g/100 g 4A sieve; regeneration/cooling air flow = 0.14 m/s; 12 cm dia bed x 12.5 cm long; regeneration air humidity ~-60°C dew point; Type 4A Union Carbide sieves activated at 350°C, 10 µL H_2O/L air for 2 h.

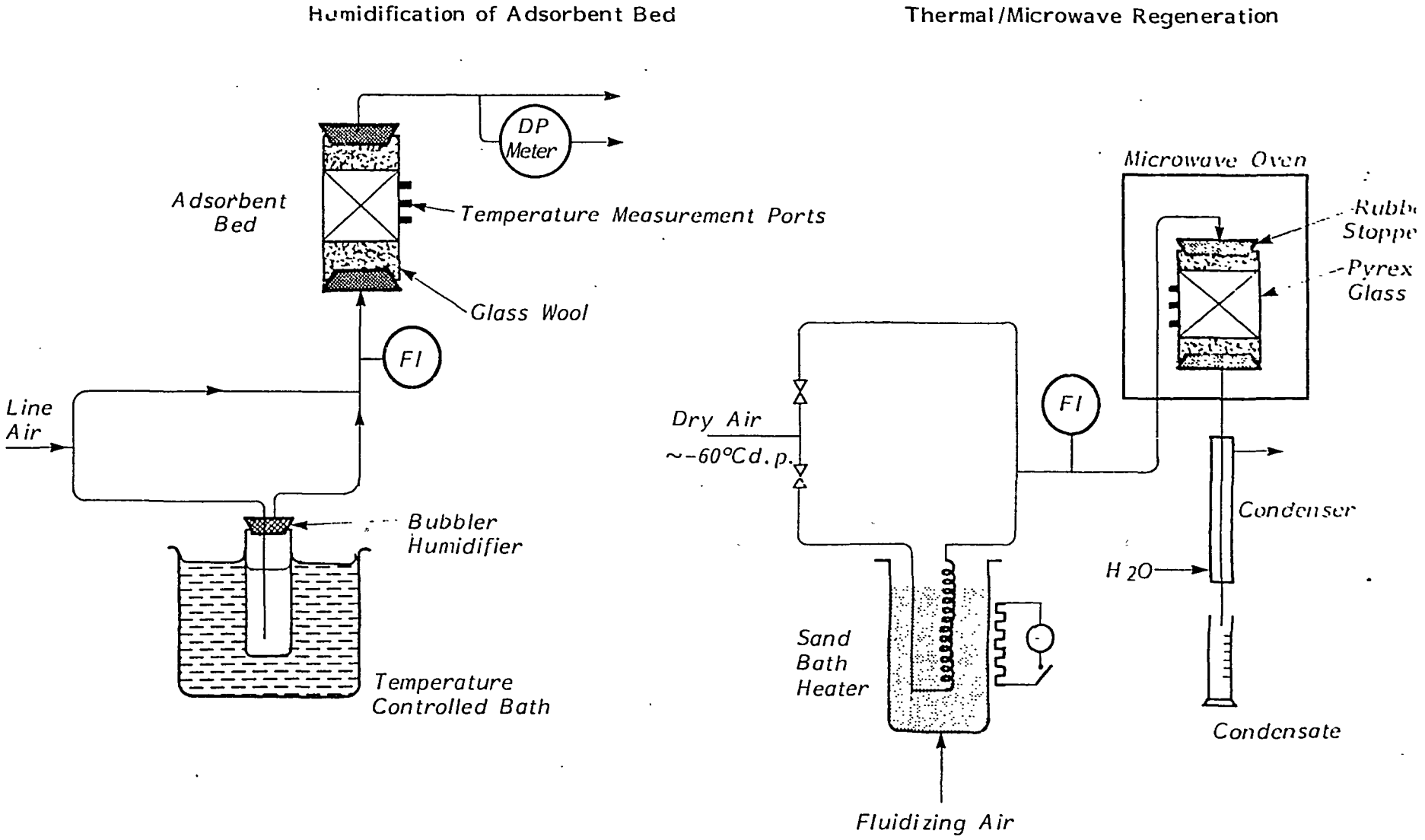

FIGURE 1

EXPERIMENTAL EQUIPMENT

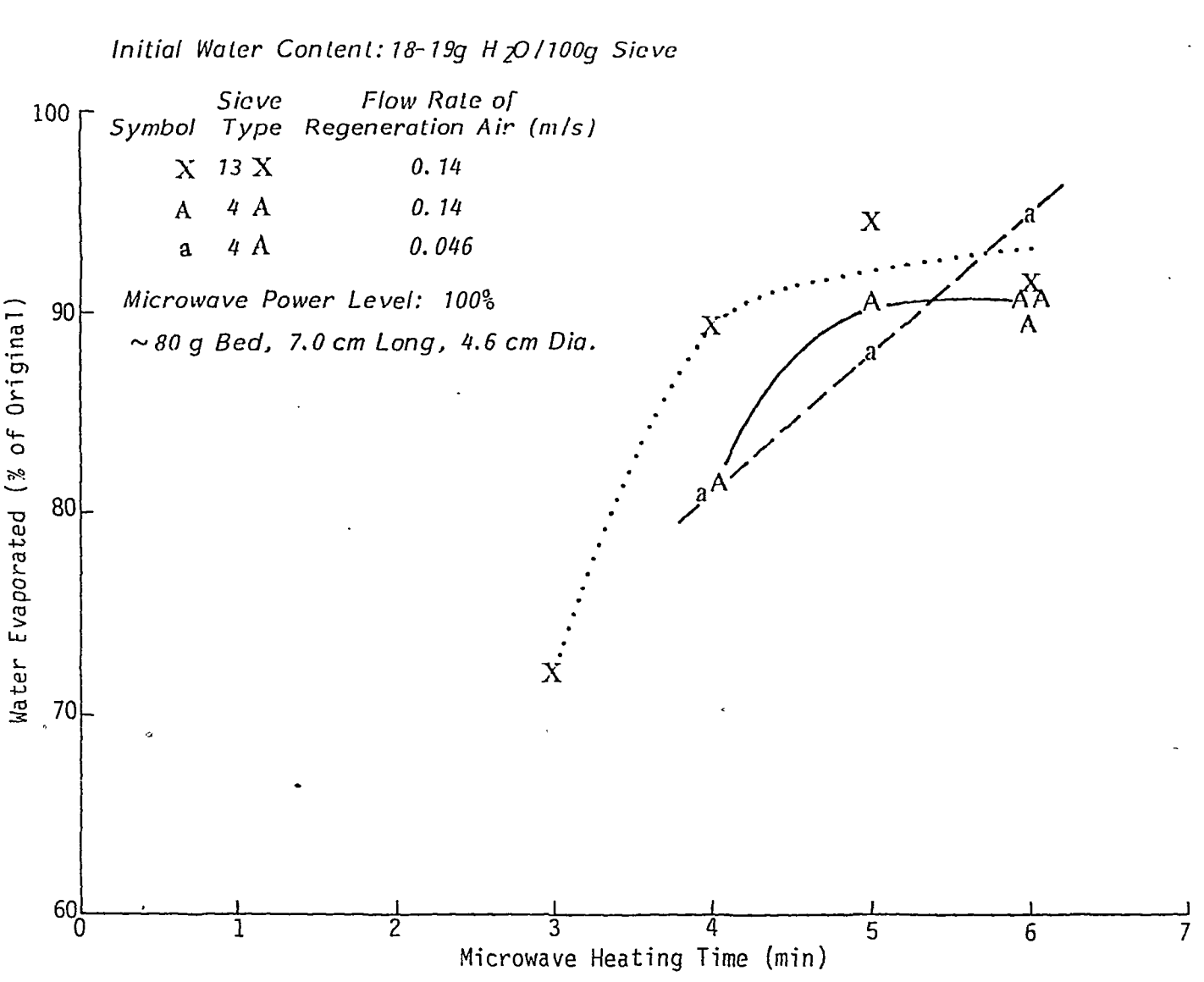

FIGURE 2

EFFECT OF HEATING TIME ON THE AMOUNT OF WATER EVAPORATED FOR MOLECULAR SIEVES

FIGURE 3

EFFECT OF HEATING TIME ON THE AMOUNT OF WATER EVAPORATED FOR SILICA GEL

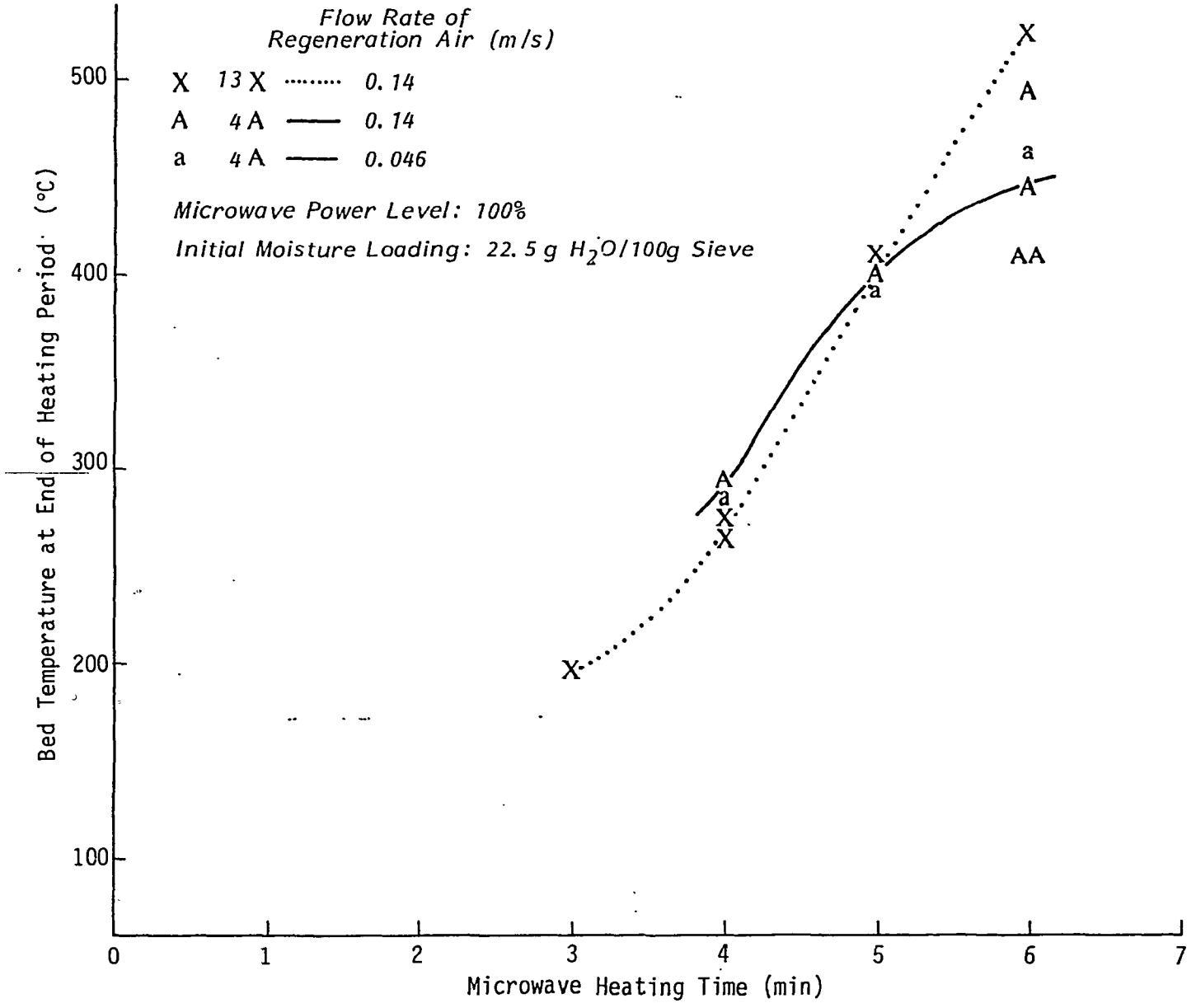

FIGURE 4

EFFECT OF HEATING TIME ON FINAL BED TEMPERATURE

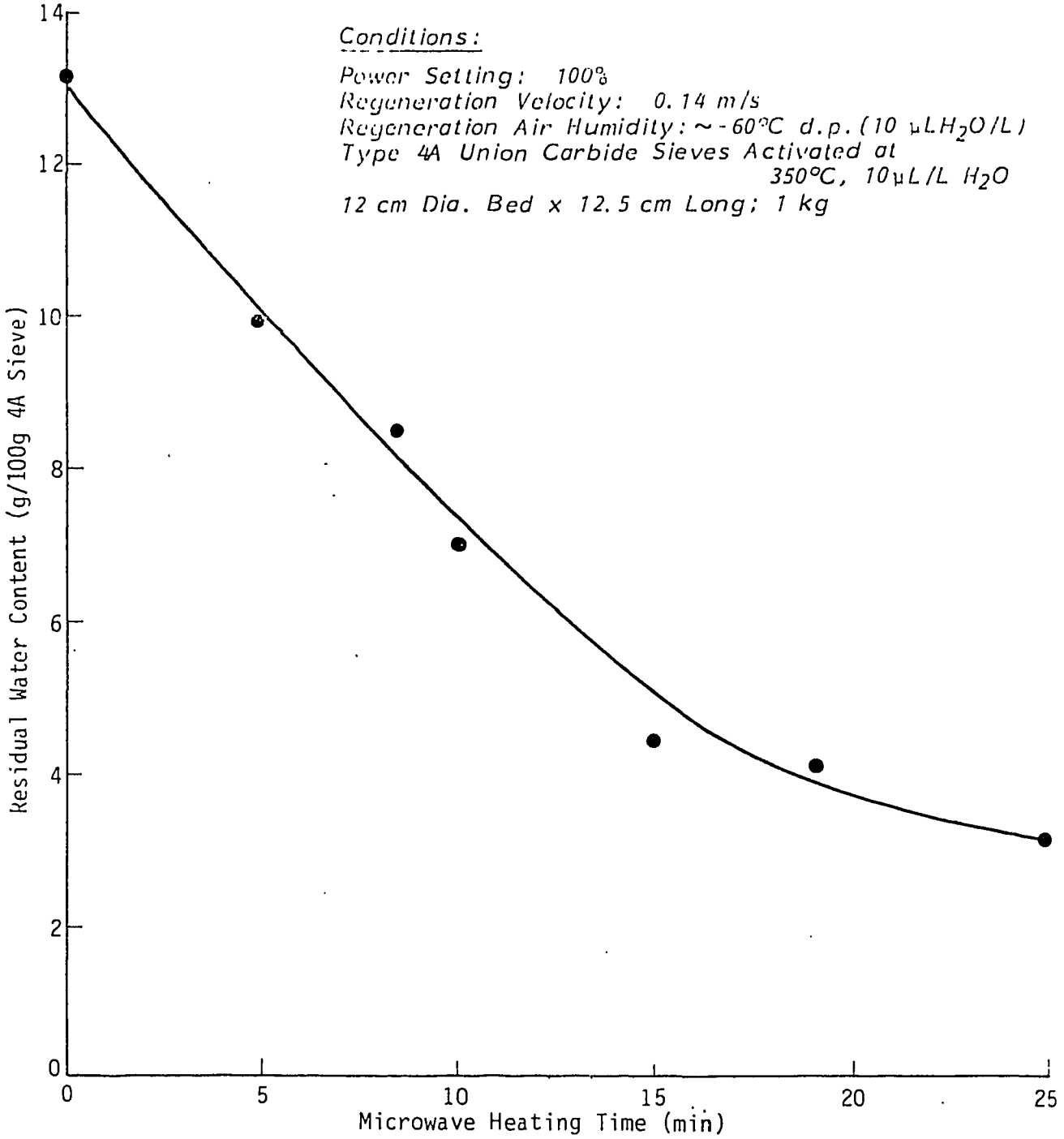

FIGURE 5

EFFECT OF MICROWAVE HEATING TIME ON RESIDUAL WATER CONTENT