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**Preliminary Evaluation of a Concept Using Microwave
Energy to Improve an Adsorption-Based, Natural
Gas Clean-Up Process**

Topical Report

R. William Grimes

December 1992

Work Performed Under Contract No.: DE-FC21-86MC11076

For
U.S. Department of Energy
Office of Fossil Energy
Morgantown Energy Technology Center
Morgantown, West Virginia

By
Western Research Institute
Laramie, Wyoming

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SUMMARY

This report describes the results of a preliminary evaluation performed to: (1) determine if microwave energy could be used to regenerate a zeolite adsorbent and (2) to evaluate the feasibility of using microwave energy to improve the desorption phase of a pressure swing adsorption process applied to upgrading natural gas (methane) contaminated with nitrogen. Microwave regeneration was evaluated by comparing the adsorption characteristics of a zeolite preconditioned by heating under vacuum to the characteristics of the same zeolite after various lengths of exposure to microwave energy. The applicability of microwave regeneration to natural gas cleanup was evaluated by measuring the rise in adsorbent temperature resulting from the microwave exposure. Microwave energy consumed by heating the adsorbent is not productive and must therefore be minimal for a process to be economically viable.

Tests were conducted in a small pressure swing apparatus containing about 100 grams of adsorbent. The tests compared the performance of a preconditioned natural zeolite adsorbent with its performance after regeneration by 1, 2, and 3 minutes of exposure to microwave energy. A natural zeolite, chabazite, was used in the testing. In consecutive baseline pressure cycles, the preconditioned adsorbent showed a strong selectivity toward methane adsorption in the first cycle. However, as the chabazite became saturated with methane, selectivity rapidly declined in the second and third cycles and no significant separation was evident in subsequent cycles.

The methane-saturated chabazite was removed from the adsorption column and placed in a microwave oven for 1-, 2-, and 3-minute exposures. The volume of gas desorbed in the microwave oven was recorded as well as the final temperature of the chabazite for each exposure. The regenerated chabazite was then returned to the adsorption column for more pressure cycles, and its performance was compared with that of the preconditioned chabazite.

Exposure of the methane-saturated chabazite for 2 minutes to microwave energy effectively regenerated the adsorbent, but resulted in a 75°F (42°C) rise in adsorbent temperature. This temperature rise indicates that the concept is unacceptable for natural gas processing due to excessive energy consumption.

BACKGROUND

Based on discussions and negotiations between WRI and DOE, Task 4.3.2, Natural Gas Cleanup, has been defined in the Annual Project Plan for October 1, 1991 to September 30, 1992 for research on a less complex and more cost effective method for cleaning up natural gas. This report describes the investigation that has been conducted following the research approach described in the Annual Project Plan and the submitted and approved experimental plan (milestone 4.3.2A).

Increased use of natural gas can enhance U.S. energy security, environmental quality, and economic strength by decreasing our reliance on imported oil. Natural gas is plentiful in the United States, and environmentally, it is the least damaging of the fossil fuels. Though natural gas has many advantages as a fuel, demand has decreased since the early 1970's (U.S. Department of Energy 1992). One reason for this decrease is a lack of confidence in future supply. Technologies that enlarge and stabilize the domestic natural gas reserve base will help increase public confidence in the long-term availability of affordable natural gas and thus, help to encourage its use.

Technologies that clean up low quality natural gas (LQNG) will increase this reserve base without increased drilling activity. Typically, LQNG is defined as methane contaminated by other gases and/or water to the extent that it cannot be used without processing. LQNG is an extensive but as yet largely under-developed resource (U.S. Department of Energy 1992). About one-half of all domestically produced natural gas requires processing to be saleable. Dehydration technologies are currently used extensively, but many of the separation technologies that deal with other common natural gas diluents are too expensive to be practical at today's natural gas prices.

Among the contaminants common in natural gas, nitrogen is one of the more difficult to remove economically. For larger gas fields cryogenic processing in which the gas is liquefied and then distilled can be used successfully. However, due to the high capital and operating costs of cryogenic systems, their application is restricted to larger fields, typically, where production exceeds 20 MMscf/day (Minteco PSA 1991). A lot of work has been done toward developing membrane separation technologies. However, the available methods are generally too costly for practical use in low-volume natural gas cleanup (U.S. Department of Energy 1992). There is a clear need for an alternative method of separating nitrogen from natural gas, particularly for smaller fields.

Pressure Swing Adsorption (PSA) has been successfully applied to many low-volume gas separation problems, including the production of nitrogen and oxygen from air in smaller-scale commercial operations. PSA uses solid adsorbents that preferentially adsorb one component of a gas mixture, thus separating the mixture into adsorbed and non-adsorbed

components. The process also requires that the adsorbent exhibit an increased capacity for the sorbate at elevated pressure. A cycle based upon adsorption at high pressure followed by desorption as the pressure is reduced can then be used to separate a gas mixture into purified component streams.

The widespread commercial acceptance of PSA for small-volume gas separations makes it a natural choice in the search for a non-cryogenic method of separating nitrogen from methane. The application of PSA to nitrogen/methane separation has been studied extensively, and pressure/vacuum swing adsorption cycles for bulk separation of nitrogen/methane mixtures have been developed (J.S. D'Amico 1992, letter dated March 3, 1992, from Nitrotec Engineering Company to Rentec Inc. personal correspondence). However, the high capital and operating costs associated with vacuum regeneration of the adsorbent limits acceptance of the pressure/vacuum swing process.

Adsorbent regeneration has a strong influence on the efficiency of separation processes based upon cyclic adsorption. Regeneration is a major factor controlling the amount of adsorbent needed to produce a given amount of product, and thus, the size and cost of the separation system. Energy costs associated with adsorbent regeneration are a major part of operating costs in adsorption-based separation processes. Indeed, adsorbent regeneration tends to be the major factor in the overall economics of these processes (Lukchis 1973).

The unpublished results of work conducted at Western Research Institute involving microwave regeneration of adsorbent chars (Dr. C.Y. Cha 1989, University of Wyoming, personal communication) suggested the possibility that microwave energy could be used instead of vacuum pumping to increase desorption during the low pressure portion of a PSA cycle. Despite relatively high cost, microwave energy has found industrial acceptance in applications where its unique properties offer advantages that offset its cost. Direct energy transfer, homogeneous heating, selective heating, high power density, and low thermal inertia are among the properties of microwave energy that can provide a technical or economic advantage to compensate for the high capital cost of the microwave equipment (Orfeuill 1987).

Because the transfer of microwave energy to a dry solid is caused almost entirely by dipole interactions (Metaxas and Meredith 1983) and the adsorption sites in zeolites are highly polar, microwave energy could be very efficient for desorbing gases from zeolites. Thus, for this application, the ability to selectively apply energy directly and evenly to the adsorption sites could provide the technical advantage needed to offset the high cost of microwave energy.

OBJECTIVES

The objectives of this work were: (1) to determine if microwave energy could be used to regenerate a zeolite adsorbent, and (2) to evaluate the feasibility of using microwave energy to improve the desorption phase of a PSA process applied to the upgrading of natural gas (methane) contaminated with nitrogen.

EVALUATION CRITERIA

Because of the relatively low value of natural gas, upgrading processes must use energy very efficiently, particularly when the energy used is electricity. For a process which employs microwave energy in the gas desorption phase, nonproductive heating of the adsorbent is prohibitively expensive and also results in decreased adsorbent productivity by necessitating an unproductive adsorbent cooling step. The cost associated with microwave heating of the adsorbent is shown below. For any method of heating, the minimum energy consumed in heating an adsorbent bed is:

$$Q_{\min} = C_{ps} (T_2 - T_1)(W)$$

where,

$Q_{\min}$ is minimum heat requirement (Btu)
 C_{ps} is the specific heat of the adsorbent
 $(T_2 - T_1)$ is the temperature rise (degrees F)
 W is the weight of the adsorbent bed (lb)

The overall efficiency of microwave heating is typically around 50% (Orfeuill 1987), thus electric power consumed by microwave heating of the adsorbent bed is two times $Q_{\min}$ (Btu) or 0.478 times $Q_{\min}$ (watt-hours).

The weight of the adsorbent needed to produce a given volume of clean gas per cycle is equal to that volume divided by the adsorbent productivity. Thus, the minimum electric energy (in watt-hours) consumed in microwave heating of the adsorbent bed with respect to product volume is:

$$Q_{\min} = C_{ps} (T_2 - T_1) (V_p) (0.478/S) \quad (2)$$

where,

$Q_{\min}$ is minimum heat requirement (watt-hours)
 V_p is the product volume (ft³)
 S is the adsorbent productivity (ft³/lb)

In work conducted independently for Williams Technologies Inc., Minteco PSA (1991) reported a methane adsorption capacity of 0.366 ft³/lb for chabazite preconditioned by heating to 750°F (399°C). Using the reported adsorption capacity and assuming 90% desorption of the adsorbed methane as product gives an adsorbent productivity of 0.33 ft³/lb/cycle. Also, as suggested by Minteco PSA, a maximum allowable processing cost of \$0.90/MMBtu was established. This cost was based upon the difference between natural gas sale prices and customary production costs.

Using equation 2, an adsorbent productivity of 0.33 ft³/lb/cycle, Q_{min} was calculated for a product gas volume of 987 scf (equivalent to 1 MMBtu of methane) and a 1°F (0.6°C) temperature rise. The electric energy requirement for microwave heating of this adsorbent would be 301 watt-hours per degree Fahrenheit adsorbent temperature rise per MMBtu of methane produced. A 20°F (11°C) temperature rise in the adsorbent bed would consume a minimum of 6.02 kilowatt-hours of electric energy. At an estimated cost of \$0.06/kilowatt-hour, this is about \$0.36 (40% of the total allowable processing cost) per MMBtu of product gas in nonproductive energy costs.

Based on the cost of nonproductive energy consumption, the criteria for this evaluation required that the adsorbent be regenerated with minimal (less than 20°F/11°C) rise in adsorbent temperature due to microwave heating.

RESEARCH STRATEGY

Williams Technologies, Inc. (WTI) has been investigating the possibility of developing an economically viable wellhead PSA system to separate nitrogen contamination from natural gas for application to a field in Kansas. They have determined that existing PSA cycles using vacuum-assisted adsorbent regeneration are unacceptable for this small field. WTI investigated the performance of a variety of solid adsorbents, including various natural and synthetic zeolites and an active carbon in PSA cycles for separation of nitrogen/methane mixtures. While they did not find a workable adsorbent, they developed process guidelines and generated data related to the performance of various solid adsorbents for the separation of nitrogen/methane mixtures.

Among the zeolites tested, WTI found that the natural zeolite, chabazite, showed a strong selectivity toward methane adsorption, but was not an acceptable adsorbent for this separation because the adsorbed methane did not desorb on pressure reduction (Minteco PSA 1991). This behavior can be predicted due to the high heat of adsorption for methane on chabazite (Barrer 1978). Because chabazite holds methane strongly and because its performance in this particular separation had been the subject of reports supplied by WTI, chabazite was chosen as the zeolite

adsorbent to test the concept of microwave-assisted desorption. If microwave energy could increase the desorption efficiency of methane held tightly on chabazite without subsequent heating of the adsorbent, then it should also work with other zeolites, such as faujasite and zeolite-L, where methane is held less tightly (Barrer 1978).

EXPERIMENTAL PROCEDURE

Pressure Swing Apparatus

Adsorbent regeneration consists of the desorption of adsorbed gas with a subsequent return of the adsorbent's adsorptive power. Thus, to evaluate the concept of using microwave energy for adsorbent regeneration, an ability to measure quantities of gas adsorbed and desorbed was needed. For this evaluation, adsorption and desorption were measured volumetrically using the apparatus shown schematically in Figure 1.

A Porter Model number 201SSVC 0.1 scfm mass flow controller was used to measure the gas feed rate. The rate multiplied by the flow time, as recorded by a Cralab Model 168 universal timer, gives the quantity of gas fed to the adsorber. Feed gas was supplied to the mass flow controller from a compressed gas cylinder. The gas mixture, containing 30% N_2 and 70% CH_4 as supplied by Scott Specialty Gases, Inc., provided a known and constant composition of the adsorber feed.

Effluent gas quantity was measured volumetrically by displacing water from six individually-selectable cylinders. The cylinders were made from 22-inch lengths of 2-inch, schedule 40, clear plastic pipe. The closed end of each cylinder was fitted with a rubber septum from which a sample of gas could be withdrawn for analysis. The cylinders were supported with their open ends in a pan of water which was fitted with an overflow to maintain a constant water level in the pan.

Gas Analysis

The effluent gas composition was determined using a Hewlett Packard 5840A gas chromatograph equipped with a thermal conductivity detector and a 0.125-inch x 20-foot column packed with Chromosorb 102. The carrier gas was helium. From each gas collection cylinder, a 0.5 cm^3 gas sample was withdrawn and injected directly onto the column of the gas chromatograph by means of a gas-tight syringe. Detector response factors were calculated using the feed gas mixture as a calibration standard. A normalization method, where area percent data are corrected using the appropriate detector response factors for the calibrated peaks and then normalized to 100%, was used to measure the concentration of nitrogen and methane in each sample.

Adsorbent

Chabazite from the Chula Claims in Arizona was supplied by WTI for use in the tests. The crushed chabazite was screened to -20/+30 mesh using ASTM Method E 11 (American Society for Testing and Materials 1990) and preconditioned by heating under partial vacuum (12 inches of Hg) for 1 hour to a final temperature of 750°F (399°C). The hot chabazite was then sealed in a clean glass jar and allowed to cool.

For each test series, approximately 100 grams of preconditioned chabazite was weighed and poured into the adsorption column. The column was tapped during filling to settle the chabazite into a packed bed. The filled column was sealed and connected to the adsorption apparatus.

Pressure Swing Cycle Procedure

The performance of the preconditioned chabazite was established using six adsorption pressure cycles. This also resulted in the production of saturated chabazite for the subsequent microwave desorption tests. The simple two-step pressure cycle consisted of feeding the gas mixture (30% N₂, 70% CH₄) at a constant rate of 1183.2 scm³/min into the column until the column pressure reached 50 psig, stopping the feed, and allowing the column to slowly depressurize from the feed end.

Before adsorption testing began, flow control valve 1 (FCV₁, Figure 1) was set to provide a small flow from the outlet end of the column. The flow rate was set so that a pressure of 50 psig was reached in 1 minute with the column filled with glass beads. This setting was then maintained throughout the adsorption tests. Gas from the outlet end of the column was collected in the first three cylinders during the feed portion of the cycle. This gas represents the waste stream from the pressure cycle.

When the column pressure reached 50 psig, the column was shut-in momentarily and then allowed to depressurize from the feed end at a preset rate controlled by FCV₂ (Figure 1). The gas from the depressurization portion of the cycle was collected in the remaining three cylinders. This gas represents the product from the pressure swing cycle. Depressurization took about 7 minutes in a typical cycle, but a slow evolution of gas continued and was collected for an additional 15 minutes after the column reached 0 psig. Figure 2 shows a typical pressure swing cycle. Table 1 lists the valve operation sequence used for the pressurization and depressurization of the column.

Figure 2. Typical Pressure Swing Cycle

Table 1. Valve Operation Sequence

	SV ₁	SV ₂	SV ₃
Step 1	Open	Open	Closed
Shut-In	Closed	Closed	Closed
Step 2	Closed	Closed	Open

For each cycle, all effluent gas was collected, and its volume and composition was determined and recorded. The feed time was also recorded and, when multiplied by the feed rate, gave the volume of gas fed in each cycle. Atmospheric pressure and the temperature of the water in the gas measuring cylinders was recorded for each test.

Microwave Desorption

After six pressure swing cycles, the chabazite was removed from the adsorption column and placed in a 500 mL round bottom flask. The flask was connected by a length of plastic tubing to a gas collection cylinder and placed in a Kenmore Model 564,8773680 microwave oven. The gas collection cylinder was located outside the microwave oven. The oven

provided about 700 W of power as measured by the two-liter method used for such appliances (Van Zante 1973). The oven was turned on for 0, 1, 2, or 3 minutes, and the effluent gas was collected, and its volume measured. The flask was removed from the oven, and the temperature in the center of the chabazite was measured with a thermocouple probe.

The regenerated chabazite from the microwave oven was then reloaded into the adsorption column, and additional pressure swing cycles were run. Effluent volume and composition of the gas streams were determined and recorded and subsequently compared to the baseline values obtained for the preconditioned chabazite.

RESULTS

Preliminary Testing

A series of tests was conducted to determine the repeatability of volumetric measurements made with the laboratory apparatus. Tests were conducted with the adsorption column by-passed, with an empty column, and with the column filled with glass beads. In each of over 20 tests, volumetric repeatability was within $\pm 1\%$.

The gas chromatograph was calibrated using feed gas from the compressed gas cylinder as a standard. The composition of gas collected by water displacement was compared with the original feed gas composition to determine whether the solubility of nitrogen and methane in water would affect the results. No detectable difference was found between the compositions of the original feed gas and the gas collected by water displacement.

Pressure Swing Cycles

Table 2 lists the volume of methane and nitrogen in the feed and effluent from consecutive pressure swing adsorption cycles using preconditioned chabazite and chabazite regenerated by 1 and 2 minutes in the microwave oven. The effluent volume from the pressurization and depressurization portions of the cycle are listed separately. Figure 3 shows methane and nitrogen adsorption per gram of adsorbent for the cycles.

Table 2. Feed and Effluent Volumes for Selected Cycles, scm³

Cycle	Feed		Effluent			
	CH ₄	N ₂	Pressurize		Depressurize	
	CH ₄	N ₂	CH ₄	N ₂	CH ₄	N ₂
Baseline 1	2518.9	1087.3	18.7	919.5	1390.0	501.8
Baseline 2	2064.7	891.2	475.1	597.1	1399.3	479.7
Baseline 3	2050.9	885.3	537.9	462.7	1347.2	521.1
Baseline 4	2064.7	891.2	680.6	395.8	1351.3	523.9
Baseline 5	2050.9	885.3	666.8	402.7	1352.8	537.6
Baseline 6	2064.7	891.2	684.0	402.7	1327.1	522.1
One Minute 1	1584.1	683.8	11.0	52.8	1090.0	539.6
One Minute 2	1418.8	612.4	4.5	57.4	1260.5	609.6
Two Minutes 1	2424.3	1046.5	39.6	697.7	1318.5	476.6
Two Minutes 2	2024.9	874.0	455.1	480.5	1335.0	502.5
Two Minutes 3	1928.5	832.4	588.7	357.0	1340.7	500.4

Baseline Pressure Cycles

In the pressurization portion of the first cycle (Baseline 1), the preconditioned chabazite adsorbed 24 scm³ of methane per gram of adsorbent (see Figure 3). Nitrogen (adsorbed from the air while the column was being loaded with adsorbent) was replaced by methane for a negative net adsorption of nitrogen in the first cycle. Very little methane was found in the waste gas stream during the pressurization portion of the cycle (2.0%), and based upon the volume and composition of this waste stream effluent from the first cycle, it can be concluded that methane was selectively adsorbed on chabazite. The depressurization effluent from the first cycle does not show significant methane enrichment (73.5 versus 70.0%). This is probably due to the fact that the column void volume and volume in the connecting tubing that is part of the apparatus are all filled with nearly pure nitrogen at the end of the first pressurization cycle. The rapid blow-down step used in more complex cycles (Yang 1987) would increase product purity as would desorption of more of the residual methane held by the chabazite.

Figure 3. Methane and Nitrogen Adsorption for Selected Cycles

Effluent from the pressurization portion of the second cycle (Baseline 2) contains a considerable amount of methane (44.3%). Table 3 lists the percentage of the methane feed reporting to the waste stream during pressurization for each cycle listed above. Significant methane in the waste stream is indicative of low product recovery, a condition that is not acceptable for natural gas processing. Methane adsorption in the second cycle is reduced to about 12.5 scm³ per gram of adsorbent, one-half of the amount found for the first cycle. Effluent from the depressurization portion of the second cycle is very similar to that of the first cycle, with no significant methane enrichment of the product detected (74.5 versus 70.0%).

Table 3. Methane Loss During Pressurization for Selected Cycles

Cycle	Methane in Feed, scm ³	Methane in Waste, scm ³	Percent of Feed
Baseline 1	2518.9	18.7	0.7
Baseline 2	2064.7	475.1	23.0
Baseline 3	2050.9	537.9	26.2
Baseline 4	2064.7	680.6	33.0
Baseline 5	2050.9	666.8	32.5
Baseline 6	2064.7	684.0	33.1
One Minute 1	1584.1	11.0	0.7
One Minute 2	1418.8	4.5	0.3
Two Minutes 1	2424.3	39.6	1.6
Two Minutes 2	2024.9	455.1	22.5
Two Minutes 3	1928.5	588.7	30.5

By the end of the third cycle (Baseline 3), the chabazite shows essentially no selective adsorption of methane from the gas mixture, and the composition of the effluent gas during pressurization and depressurization are nearly equal for each of the remaining three pressure swing cycles. Thus, the preconditioned chabazite shows a strong selectivity toward methane adsorption, and a methane capacity of 24 scm³ per gram of adsorbent for the first pressure swing cycle. Selective adsorption and capacity for methane decline rapidly; after three pressure swing cycles, there is essentially no selective adsorption of methane and capacity is down to 10 scm³ per gram of adsorbent.

Microwave Cycles

Figure 4 shows the volume of gas desorbed and the corresponding temperature rise of the chabazite after 0, 1, 2, and 3 minutes in the microwave oven. Desorption volume was proportional to microwave exposure time for the first 3 minutes in the microwave oven. Heating of the adsorbent was evident for all three microwave exposure times.

Figure 4. Desorption and Adsorbent Heating in a Microwave Oven

Regeneration of the adsorbent was not accomplished in 1 minute in the microwave oven, even though a rise of over 50°F (28°C) in adsorbent temperature was noted for this exposure. Note that the methane content of the products resulting from the depressurization portion of the first and second cycles (One Minute 1 and 2) is less than that in the feed (67 versus 70%). Two minutes in the microwave oven were needed to regenerate the adsorbent to the activity observed for the preconditioned adsorbent. However, this exposure resulted in a 75°F (42°C) rise in adsorbent temperature. The methane content of the products resulting from the depressurization portion of the three cycles (Two Minutes 1, 2, and 3) averages about 73% versus 70% in feed, an enrichment comparable to that observed for the preconditioned chabazite.

CONCLUSIONS

Microwave energy can be used successfully to regenerate a zeolite adsorbent, but excessive heating of the zeolite makes the concept impractical for application in natural gas cleanup. Microwave-assisted desorption may prove useful in adsorptive purification processes where recovery of the sorbate with minimal dilution is required.

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DISCLAIMER

Mention of specific brand names or models of equipment is for information only and does not imply endorsement of any particular brand.

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