DEMONSTRATION OF A LOW COST 2-TOWER MICRO SCALE N₂ REJECTION SYSTEM TO UPGRADE LOW-BTU GAS FROM STRIPPER WELLS

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ABSTRACT

Natural gas is marketed on the basis of its heat content (950 BTU/cu ft or higher). U.S. pipeline specifications vary but generally require nitrogen (N₂) to be less than 5% resulting in 32 tcf (17% of known reserves) to be categorized as low-BTU "sub quality". N₂ is thus a major target for removal to upgrade natural gas to pipeline quality. A significant portion of the nation's N₂-rich low-BTU gas is trapped in modest to small fields owned by stripper operators, or isolated behind pipe. These small fields are not amenable to upgrading technologies such as cryogenic separation and conventional pressure swing adsorption (PSA) because these fields cannot usually deliver the large feed volumes necessary for profitable operations of these types of technologies.

In an attempt to encourage economically viable upgrading of low-BTU gas from stripper wells, a demonstration project that encompasses the planning, design, construction, operation, and optimization of an easily built, low-cost, 2-tower micro-scale PSA (pressure swing adsorption) plant for N₂-rejection using non-patented processes and commonly available equipment was proposed as a joint project between the Kansas Geological Survey (KGS) and American Energies Corporation (AEC), Wichita, Kansas.

During the current reporting period, the N_2 rejection plant was run with a feed having a hydrocarbon (CH₄+) content of around 64%. The plant was run at different settings and results from two best settings where the feed gas could be upgraded to pipeline quality (> 950 BTU/cu ft) are presented in this report. Also, a handheld hydrocarbon detector was calibrated against gas composition measured by a (portable) gas chromatograph (GC) on samples collected from the plant. This correlation proved very useful during the plant optimization process. GC analysis also showed that the bed of activated carbon successfully adsorbed heavier hydrocarbons thus preventing them from being vented. Finally, a design flaw became evident during this testing process that was causing bed blow-out particularly during the venting process, and this required refilling of the towers with activated carbon and the placement of a filter at the top the prevent recurrence. Results obtained so far were reported in a trade journal, while necessary updates were carried out on the project web-site.

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INTRODUCTION

Natural gas is marketed on the basis of its heat content (950 BTU/cu ft or higher). U.S. pipeline specifications vary but generally require nitrogen (N_2) to be less than 5% resulting in 32 tcf (17% of known reserves) to be categorized as low-BTU "sub quality". N_2 is thus a major target for removal to upgrade natural gas to pipeline quality. A significant portion of the nation's N₂-rich low-BTU gas is trapped in modest to small fields owned by stripper operators, or isolated behind pipe. These small fields are not amenable to upgrading technologies such as cryogenic separation and conventional pressure swing adsorption (PSA) because these fields cannot usually deliver the large feed volumes necessary for profitable operations of these types of technologies.

It is the objective of this project to design, construct, operate, and optimize a micro-scale N_2 rejection plant to economically upgrading of low-BTU gas from stripper wells. Our goals were to build a low-cost, 2-tower micro-scale PSA (pressure swing adsorption) plant that would adsorb methane and heavier hydrocarbons under pressure while rejecting the N_2 followed by extraction of the hydrocarbons under vacuum.

EXECUTIVE SUMMARY

inches of Hg (vacuum), where the feed gas was upgraded to pipeline quality (> 950 BTU/cu ft) are discussed in this report. A portable hydrocarbon detector proved useful during the optimization process. Readings from the portable detector were calibrated against comparable samples measured in a gas chromatograph (GC). This correlation enabled quick estimation of gas composition and upgradation as a result of changes made to the plant operation. GC analyses of feed and sales gas showed that the activated carbon is efficient in adsorbing heavier hydrocarbons (C_2H_6+) thus preventing venting of gases with high BTU content.

The project web-site, which can be publicly accessed at <u>http://www.kgs.ku.edu/PRS/Microscale/index.html</u>, was kept updated with all results obtained from these initial tests. Results from this initial phase of study were summarized in an article and submitted for publication to the E&P journal – an industry publication that is widely circulated amongst the independent oil and gas operators particularly in Kansas.

Future plans include testing the plant with lower quality of feed, i.e. feed with a heat content ~ 620 BTU/cu ft to test the lower limits of the plant regarding its upgrading capacity.

EXPERIMENTAL OBSERVATIONS & DISCUSSION

PLANT OPERATION

An example of the feed gas composition (Figure 1) analyzed by the gas chromatograph (GC) showed that the feed coming into the plant contained around 33% N_2 with a heat content of 717 BTU/cu ft (dry). The feed gas was made up of commingled production from several wells, some of which operated on pump. Thus, the feed

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composition was found to fluctuate over time. The average N_2 content in the feed measured over the duration of this test (4 days) was around 37% (Figure 2).

The plant was operated under various modes. Given the above mentioned feed composition, the plant was found to upgrade the low-BTU feed to pipeline quality (> 95 BTU/cu ft) when the plant was operated under the following mode:

i) Charge Tower 1 (from bottom) with feed gas to 34 psi while Tower 2 is desorbed to 22 inches Hg (mercury vacuum) from the bottom.

ii) Vent Tower 1 (from the top) to 2 psi while Tower 2 is held in vacuum.

iii) Desorb Tower 1 to 22 inches Hg (vacuum) while Tower 2 is connected to feed gas and charged to 34 psi as described in Step (i).

The values controlling feed gas flow into the plant are set in a way such that the time to charge the tower to the set pressure (here 34 psi) equaled that to desorb the other tower to 22 inches of mercury vacuum and vice-versa. The following sets of experiments were started after filling up each tower with new (fresh) activated carbon that was filled to the very top of each tower.

At the above mentioned settings, the sales-to-feed ratio was found to be 0.54, i.e., for 100 mcf of feed gas with 63% hydrocarbons, the plant was able to generate 54 mcf of pipeline quality gas (with an average of 953 BTU/cu ft) for the sales stream (Figure 2). The plant demonstrated an efficiency of recovering 73.2% of the hydrocarbons while rejecting 76.7% of the nitrogen entrained in the feed gas. The average N_2 concentration in the vent gas was 63.1%. At this setting, the BTU recovery (ratio of total BTU in sales to total BTU in the feed on a daily basis) was calculated as 75.7%.

The volume of gas vented is proportional to the volume of dead-space (volume that is unoccupied by the activated carbon) inside the tower and the pressure drop (ΔP) that occurs after the charged tower (at 34 psi) is vented to 2 psi. This venting process is crucial because it enables rejection of the unadsorbed N_2 -rich gas. Without this rejection (venting), the unadsorbed N₂ would end up in the sales stream during the desorption process. The percentage of tower volume occupied by dead-space adversely affects tower performance because volume of activated carbon available for adsorption would be greater if the dead-space volume were low or reduced to zero. Each of the towers, made of carbon steel, has a 48 inch diameter and is 8 feet (96 inches) tall (seam to seam). The screen that is set at the bottom of the tower to hold the bed of activated carbon is set about 18 inches from the tower bottom resulting in a dead-space volume of about 19% of the tower volume. During fill-up each of the towers was topped to the flange to minimize dead-space at the top of the tower. This dead-space at the base of the tower can not be removed without laying down the tower, removing the screen, and resetting it a few inches away from the bottom flange. This procedure would require a plant shut down followed by major repair work.

Thus without being able to reduce the dead-space immediately, the plant was operated under a different setting that resulted in the reduction of the ΔP . In this setting, each of the towers were charged alternatively to 20 psi (instead of 34 psi) and then vented to 2 psi before being desorbed to 22 inches of Hg vacuum. The chain of events controlling the plant operation remained the same as described earlier. As expected, the sales-to-feed ratio under these new settings was found to increase to 0.60 from 0.54, i.e., for 100 mcf of feed gas with 65% hydrocarbons, the plant was able to generate 60 mcf of

pipeline quality gas (with an average of 964 BTU/cu ft) for the sales stream (Figure 2). The plant demonstrated an efficiency of recovering 77.7% of the hydrocarbons while rejecting 73.8% of the nitrogen entrained in the feed gas. The average N_2 concentration in the vent gas was 63.2% (unchanged from earlier setting). At this setting, the BTU recovery (ratio of total BTU in sales to total BTU in the feed on a daily basis) was found to increase to 79.7% (from 75.7% obtained in the earlier setting).

These results indicate that reduction in the tower charge pressure improves both the sales-to-feed ratio and the BTU recovery. However, an exact comparison between the above described plant settings is difficult because between the two settings the average feed composition changed from 63% to 65% hydrocarbons, i.e., from 687 BTU/cu ft to 722 BTU/cu ft.

BED BLOWOUT

The dead-space volume at the base of each tower contains feed gas at 2 psi after completion of the vent stage, and upon desorption (tower evacuation to vacuum from the bottom) this nitrogen-rich feed gas (35 to 37%) ends up in the surge tank, which connects to the sales stream. In order to better vent the feed gas that has never contacted the bed of activated carbon and that which accumulates at the base of each tower, the plant was run at two different settings by venting simultaneously from both the top and bottom of each tower. It was assumed that such dual venting might improve the BTU or hydrocarbon content of the gas desorbed from the bed and stored in the surge tank.

By the time the plant was operated under top and bottom venting, the tower beds had been recharged for 7 days. In the 1st setting, the towers were alternatively charged to 20 psi with feed gas, and then vented from top and bottom to 2 psi before desorbed to 22

inches of Hg vacuum. In the 2nd setting, the towers were charged to 30 psi followed by similar venting and desorption. At both these settings, the feed composition showed minor fluctuations over a 12-day period. However, the sales-to-feed ratio dropped to 0.51 for the 1st setting and then to 0.48 for the 2nd setting, which are lower than results obtained under similar settings (0.60 and 0.54 respectively) with venting from top only. This sudden reduction in the sales-to-feed ratio under minimal variations that typify the feed suggested that the beds in the towers were being blown out during the venting stage with no filter having been placed in the flange at the tower top. The venting stage results in a sudden and rapid decrease in tower pressure to 2 psi and this resulted in entrainment of fine bed particles to the flare tower where carbon blow-out was visible from the surface. With bed material blown out, the dead-space increased inside each tower and this resulted in lower volumes of feed gas being adsorbed and higher volumes of gas being vented.

The extent of the bed blow-out was appreciated when the towers were opened and it was found that about 14 inches of bed was missing from the top of each tower. The towers needed to be refilled with fresh carbon which was then topped by a filter set below the top flange to prevent future bed blowouts.

HEAVY HYDROCARBON ADSORPTION

Samples from the vent stream were also collected and analyzed with GC and the hand-held meter. Figure 3 displays the gas analysis of a sample taken from the vent stream. It is to be noted that the gas composition of the vent stream changes continually over the venting period. However, all samples taken from the vent stream at different period have a common characteristics, i.e., the ratio of heavy hydrocarbons to total hydrocarbons (C₂H₆+/CH₄+) is significantly lower (≈ 0.03) compared to that (≈ 0.07) in feed stream (Figure 1). This reduction in heavy hydrocarbons (C₂H₆+/CH₄+) signifies that the bed of activated carbon is effective in adsorbing the heavy hydrocarbons that have high BTU content. On the other hand, this effective removal of heavy hydrocarbons from the vent stream reduces the heat content of the vent stream where the entrained CH₄ (at 1010 BTU/cu ft) provides the only major source of BTU. Thus, this puts into question the economics of attempting to recover additional BTUs from the vent gas that has mostly been stripped of the heavy hydrocarbon content.

The heavier hydrocarbons effectively adsorbed by the bed of activated carbon are recovered for sales during the desorption process. Figure 4 shows an example GC analysis of the sales stream where the C_2H_6+/CH_4+ ratio has increased to 0.112 (from 0.075 in the feed). This higher C_2H_6+/CH_4+ ratio is one of the reasons why the desorbed gas attains pipeline quality (>950 BTU/cu ft). This indicates that our plant will perform better by operating with a higher sales-to-feed ratio if the C_2H_6+/CH_4+ ratio in the feed is higher than that observed in the current feed.

CORELLATION – PORTABLE DETECTOR & GC

Gas samples were collected from the feed and sales stream and analyzed using a portable GC. The hydrocarbon content (CH₄+ %) for each sample was also measured using a handheld gas meter. Figure 5 plots the CH₄+ % from the handheld meter against the total hydrocarbon content (GC CH₄+ %) from GC analysis for respective samples (plotted as red filled squares). The plotted points show a good correlation (R-square = 0.96). The equation that corrects the hydrocarbon content (CH4+ %) read using the handheld meter to that based on GC analysis is also shown on Figure 4. The handheld

meter is easy to use and quick to read in a field setting particularly when trying to understand the effects caused by changes to the plant operation settings. Thus, this equation proved handy to instantly correct hydrocarbons percentages read using the handheld meter. Such a correlation between handheld meter readings and more accurate GC-based analyses should be established for optimizing plant operations of this nature.

Figure 6 displays a good correlation (R-square = 0.9982) between the corrected hydrocarbon content (GC CH4+ %) and the BTU per cubic foot. This equation was widely used to estimate BTU content of gas samples analyzed with the hand-held meter.

OTHER OBSERVATIONS

The compressor power is critical in desorbing each tower to 22 inches of Hg (vacuum), and therefore controls the volume throughput of the plant. If the feeder wells can supply enough gas volumes at sufficient pressure, then the tower charging time (to 30 or 20 psi) can be controlled by adjusting the inlet valve. In this case, the charge cycle time was set to be equal to the desorption cycle time due to compressor limitations resulting in the throughput volume to be controlled by the time taken to evacuate each tower from 2 psi to vacuum. It is therefore recommended that required investments be made to install a powerful compressor in order to maximize sales volume from the plant.

TECHNOLOGY TRANSFER

A web site dedicated to this project has been updated with pictures, results, crosssections, log analyses etc. All reports and presentations have been posted on this web site. Results obtained till the end of this reporting period has been summarized in an article that has been submitted to the E&P Journal. This manuscript is currently under review by the journal's editors and is expected to be published in the August 2008 issue.

FUTURE TASKS

It is expected that during the next reporting period, the following tasks will be undertaken:

a) rectify bed blow-out by topping each tower with new activated carbon and holding it in place by a filter set in the tower top exit flange,

b) operate plant using poorer quality of feed with higher N_2 concentration which is available in areas of the neighboring Elmdale field (Chase County, Kansas),

c) define plant operation mode that will ungrade this poor quality feed to pipeline quality,

d) present results at the SWC Technology Transfer Conference at Erie, Pennsylvania, and

e) provide a link from the project website to the article summarizing project results and expected to be published in the August 2008 issue of E&P Journal.

CONCLUSIONS

The following conclusions were arrived at during this period of study:

1. The plant is able to upgrade low-BTU feed gas (37% N2) to pipeline quality (>950 BTU/cu ft) under two different settings.

2. The best results were obtained when each tower was charged with feed to 20 psi, then vented to 2 psi, and desorbed to 22 inches of mercury (vacuum). At these settings, the plant was able to deliver 60 mcf/d of pipeline quality sales gas using 100 mcf/d feed with 77.4% efficiency in hydrocarbon recovery, 73.8% efficiency in nitrogen rejection, and 79.7% efficiency in BTU recovery.

3. Reduction of dead-space volume resulted in improved plant performance, particularly in higher sales-to-feed ratios.

4. The venting process blows out part of the bed and this bed degradation results in poor plant performance (by unintentional increase in dead-space volume) and reduction in sales-to-feed ratios with minimal variation (changes) in the feed composition.

5. The activated carbon bed is effective in adsorbing a significant portion of the heavy hydrocarbons from the feed stream. The vent stream has minimal amounts of heavy hydrocarbons, and therefore may not be an attractive target for secondary upgradation particularly at low feed volumes.

6. It is anticipated that the plant will perform better, i.e., operate with higher sales-to-feed ratios if the feed is enriched with heavy hydrocarbons as these are effectively adsorbed by the bed and recovered in the sales stream.

7. A portable hydrocarbon meter is very effective during field operations for plant optimization. Good correlations developed with gas chromatographic (GC) analyses enable quick correction of portable meter readings to estimate hydrocarbon concentration and BTU value at different sampling points in the plant.

8. Compressor strength to evacuate each tower quickly from 2 psi to 22 inches of Hg (vacuum) plays a critical role in determining the plant throughput volumes.

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| Sample date | 30-May-08 | |
|--------------------|-----------|---------|
| Sample No. | KGS 1 | |
| Sample description | Feed gas | |
| | | |
| Component | Mole % | BTU/scf |
| L hadro ero e | 0.0000 | 0.00 |
| Hydrogen | 0.0000 | 0.00 |
| Helium | 0.6495 | 0.00 |
| CO2 | 0.2135 | |
| Neopentane | 0.0014 | 0.00 |
| Nitrogen | 33.7049 | |
| Argon | 0.1748 | 0.00 |
| Methane | 60.3800 | |
| Ethane | 2.8948 | |
| Propane | 1.3320 | |
| i-Butane | 0.1826 | 5.94 |
| n-Butane | 0.3161 | 10.31 |
| i-Pentane | 0.0664 | 2.66 |
| n-Pentane | 0.0665 | 2.67 |
| n-Hexane | 0.0135 | 0.64 |
| n-Heptane | 0.0040 | 0.22 |
| | 0 | |
| CH4+ | 65.2559 | |
| C2H6+ | 4.8759 | |
| C2H6+/CH4+ | 0.075 | |

Figure 1. Example gas chromatographic analysis of feed gas to plant.

| | | Corrected | Corrected | | | | | | | |
|-----------|---------|-----------|-----------|------------|--------------|------------|----------|----------|-----------|-----------|
| Tower | Vent to | Avg Feed | Avg Sales | | Efficiency | Efficiency | N2 % in | | | |
| Charge Pr | psi | CH4+, fr | CH4+, fr | Sales/Feed | N2 stripping | CH4+ Rec | Vent Gas | BTU feed | BTU sales | BTU rec % |
| 34 | 2 | 0.63 | 0.84 | 0.54 | 76.7 | 73.2 | 63.1 | 687 | 953 | 75.7 |
| 20 | 2 | 0.65 | 0.85 | 0.60 | 73.8 | 77.4 | 63.2 | 722 | 964 | 79.7 |

Figure 2. Comparison of plant performance at two different settings.

| Sample date | 3-Jun-08 | |
|--------------------|----------|---------|
| Sample No. | KGS 2 | |
| Sample description | Vent gas | |
| | | |
| Component | Mole % | BTU/scf |
| | 0.0000 | |
| Hydrogen | 0.0000 | 0.00 |
| Helium | 1.0348 | 0.00 |
| CO2 | 0.1317 | 0.00 |
| Neopentane | 0.0000 | 0.00 |
| Nitrogen | 60.7047 | 0.00 |
| Argon | 0.0000 | |
| Methane | 37.1535 | |
| Ethane | 0.6415 | 11.35 |
| Propane | 0.3337 | 8.40 |
| i-Butane | 0.0000 | 0.00 |
| n-Butane | 0.0000 | 0.00 |
| i-Pentane | 0.0000 | 0.00 |
| n-Pentane | 0.0000 | 0.00 |
| n-Hexane | 0.0000 | 0.00 |
| n-Heptane | 0.0000 | 0.00 |
| CH4+ | 00 4007 | |
| | 38.1287 | |
| C2H6+ | 0.9752 | |
| C2H6+/CH4+ | 0.026 | |

Figure 3. Example gas chromatographic analysis of vent gas from the plant.

| Sample date | 6-Jun-08 | |
|--------------------|-----------|---------|
| Sample No. | KGS 5 | |
| Sample description | Sales gas | |
| | | |
| Component | Mole % | BTU/scf |
| Hydrogon | 0.0000 | 0.00 |
| Hydrogen Helium | 0.0000 | 0.00 |
| CO2 | 0.1225 | 0.00 |
| | | 0.00 |
| Neopentane | 0.0000 | |
| Nitrogen | 14.5400 | 0.00 |
| Argon | 0.3692 | |
| Methane | 75.3267 | |
| Ethane | 5.2381 | 92.70 |
| Propane | 2.7426 | 69.01 |
| i-Butane | 0.3890 | 12.65 |
| n-Butane | 0.7116 | 23.22 |
| i-Pentane | 0.1574 | 6.30 |
| n-Pentane | 0.1640 | 6.58 |
| n-Hexane | 0.0363 | 1.73 |
| n-Heptane | 0.0205 | 1.13 |
| | | l |
| CH4+ | 84.7862 | |
| C2H6+ | 9.4595 | |
| C2H6+/CH4+ | 0.112 | |

Figure 4. Example gas chromatographic analysis of sales gas from the plant.



Figure 5. Correlation between hydrocarbon percent read from handheld portable gas meter with that calculated from gas chromatographic analyses.



Figure 6. Correlation to estimate BTU/cu ft from total hydrocarbon content.