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TECHNICAL PROGRESS REPORT

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Reactor Synthesis Gas

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Overview of Progress

In order to increase the production rate of purified gas stream, a new hollow fiber module (No. 4) has been fabricated with more number of hollow fibers. A few experiments have also been carried out using a CO_2 - N_2 feed gas mixture and water as an absorbent in the new module. With the new module, it was possible to increase the production rate of purified gas by a significant amount while the cycle time for vacuum desorption was considerably reduced. Preparations are now being made to study the absorption behavior of CO_2 - N_2 gas mixture with a reactive absorbent liquid such as aqueous diethanolamine (DEA) solution.

Fabrication of a Hollow Fiber Module

The fabrication procedure for the new hollow fiber module was essentially similar to that described earlier in Technical Progress Report No. 3. However, the fibers utilized in this module were tested for leakage before they were inserted in the shell.

First a mat of 1200 fibers was prepared on a polyethylene sheet over a laboratory bench top. The middle section of the fibers were kept straight and separated while the fiber ends at each side were potted in a 2 inch long hexagonal nipple (Cajon) first with a silicone rubber adhesive (RTV11, General Electric, Waterford, NY) and then using an epoxy resin mixture prepared with 80:20 ratio of C-4 resin to activator D (Beacon Chemical Co., Mt. Vernon, NY). The hollow fibers were then pressurized internally with distilled water up to a pressure level of 50 psig. Two fibers were found to be defective and were leaking during the test. They were separated and discarded. The mat of remaining 1198 fibers was then rolled to form a bundle and was inserted into the shell of the module. The fiber ends were potted with epoxy resin to form a tube sheet. The geometrical characteristics of the module is given in Table 1.

Experiment

All experiments were carried out in the following manner.

The feed was allowed to enter for a certain time through valve 1 (Figure 1) until the required pressure was achieved in the module while both valves 2 and 3 remained closed. Immediately afterwards, valve 1 was closed and simultaneously valve 3 was

opened for a short time just to take out the purified front of the mixture of gases. Then the valve 3 was closed and simultaneously valve 2 was opened for vacuum desorption. This completed one whole cycle of operation.

A constant feed gas mixture flow rate of 100 cc/min was maintained in the first six experiments (Table 2); a constant feed gas mixture flow rate of 80 cc/min was maintained during the last two experiments. In all experimental runs, the desorption time (vacuum part of the cycle) was maintained at about 24 seconds. Other sections of the time cycle were changed as required.

Results

The durations of the open and close settings of each of the solenoid valves, the corresponding feed gas pressure and the mode of operation for different experimental runs have been identified in Table 2. The purification results of each such run identified in Table 2 are provided in Table 3. All experiments were made with a feed gas mixture of 10.12% CO₂ and balance N₂, with a feed pressure of 25-35 psig in the fiber lumen. The absorbent liquid was pure water.

In our earlier studies (Technical Progress Report 4, Tables 3 and 4), the desorption time was maintained at about 40 seconds and a purified gas stream containing about 2% CO₂ was obtained. In the experiments shown in Tables 2 and 3, the desorption time was reduced by 40% to 24 seconds. Thus an increased product output rate was being achieved without sacrificing the product gas purity. In fact, purified gas containing as low as 1.5% CO₂ was obtained in these runs; this value is significantly lower than the lowest (~1.9%) obtained earlier.

Experiments are being conducted currently by reducing further the desorption time to as low as 11 seconds. If product quality remains unaffected, this should substantially enhance the production rate of purified gas. A few more experiments will be made with a different feed gas mixture (e.g., 5% CO₂ - balance N₂) before we switch to reactive absorbent solution.

Identification of Problems and Solutions

One of the electronic timers, which controlled the solenoid valve operation, failed

during experimentation. The defective timer was replaced by a new one. Further, a separate timer box has now been built and kept in reserve to prevent delays introduced by such equipment failure.

To carry out experiments with an aqueous diethanolamine (DEA) solution as an absorbent, further modifications of the experimental setup and GC configuration are necessary. It is anticipated that the purified gas as well as the vacuum exhaust line will carry some DEA vapor. Therefore, the gas streams must be discharged into a laboratory fume hood. Further, the GC column performance is adversely affected by the presence of DEA vapor. A precolumn or some other arrangement must be provided in the GC connecting lines to eliminate the DEA vapor. We are now developing the solutions of these problems.

Table 1: Characteristics of Hollow Fiber Module No. 4

Effective Length	160 cm
No. of Fibers	1198
Fiber ID	100 μm
Fiber OD	150 μm
Surface Area	6022 cm^2
Void Fraction	89.2%

Table 2: Duration of Different Settings of Solenoid Valves

Experiment No.	Module No.	Mode of Operation	Pressure in fiber lumen (psig)	Valve # 1		Valve # 3		Valve # 2	
				Open (Seconds)	Close (Seconds)	Close (Seconds)	Open (Seconds)	Close (Seconds)	Open (Seconds)
5-1	4	2	30-35	1	35	2	10.0	12	24
5-2	4	2	30-35	1	34.8	2	9.8	11.8	24
5-3	4	2	30-35	1	34.5	2	9.5	11.5	24
5-4	4	2	30-35	1	34.0	1	10.0	11.0	24
5-5	4	2	30-35	2	32.5	2	8.5	10.5	24
5-6	4	2	30-35	2	32.3	2	8.3	10.3	24
5-7	4	2	25-30	2	33.5	2	9.5	11.5	24
5-8	4	2	25-30	2	33.3	2	9.3	11.3	24

Table 3: Experimental Results

Experiment No.	Module No.	Mode of Operation	Feed Pressure (psig)	Feed Gas Composition	Purified Gas Composition
5-1	4	2	30~35	CO ₂ : 10.12% N ₂ : Balance	CO ₂ : 2.4% N ₂ : Balance
5-2	4	2	30~35	CO ₂ : 10.12% N ₂ : Balance	CO ₂ : 2.2% N ₂ : Balance
5-3	4	2	30~35	CO ₂ : 10.12% N ₂ : Balance	CO ₂ : 2.1% N ₂ : Balance
5-4	4	2	30~35	CO ₂ : 10.12% N ₂ : Balance	CO ₂ : 1.9% N ₂ : Balance
5-5	4	2	30~35	CO ₂ : 10.12% N ₂ : Balance	CO ₂ : 1.89% N ₂ : Balance
5-6	4	2	30~35	CO ₂ : 10.12% N ₂ : Balance	CO ₂ : 1.67% N ₂ : Balance
5-7	4	2	25~30	CO ₂ : 10.12% N ₂ : Balance	CO ₂ : 1.6% N ₂ : Balance
5-8	4	2	25~30	CO ₂ : 10.12% N ₂ : Balance	CO ₂ : 1.54% N ₂ : Balance

Figure 1: Solenoid Valve Locations in RAPSAB Apparatus

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