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Desorption of CO_2 From a Three-Component Solution

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ABSTRACT

New absorbent solutions containing three components have been evaluated for efficiency in decreasing the ambient CO_2 level aboard submarines. One of the absorbent systems investigated consisted of a high-boiling organic compound, sulfolane, which has a high physical absorption capacity for CO_2, combined with monoethanolamine and water. The other absorbent system had the same components except for the amine, which was 3-aminopropanol.

At the same level of saturation of the solutions with CO_2, the absorption efficiency of the three-component solutions is about the same as, or slightly inferior to, the corresponding two-component aqueous amines. However, desorption results with the three-component solutions are so much better than for the two-component aqueous amines that the lower CO_2 loadings that would be attained in the absorber section of the present CO_2 scrubbers should result in significant reduction of the ambient CO_2 concentrations in nuclear submarines.

PROBLEM STATUS

This is the final report on this phase of the problem; work is continuing on other phases.

AUTHORIZATION

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DESORPTION OF CO_2 FROM A THREE-COMPONENT SOLUTION

BACKGROUND

This study is an extension of that reported in NRL Report 6614 (1). The ultimate objective in each case was to lower the ambient CO_2 level aboard nuclear submarines. The problem of increasing the effectiveness of the CO_2 scrubber was approached by trying to improve the desorption process. Any reduction in the CO_2 loading of the absorber solution which would decrease the effluent CO_2 percentage by 0.05% (not necessarily the best reduction attainable) would add 0.9 lb/hr of CO_2 to the removal rate (at an airflow of 250 cu ft/min). In the previous report (1) the emphasis was on redesign of the reboiler section of the stripper. In the present report, the emphasis is on the composition of the absorbent solution.

SULFINOL PROCESS

A precursor to the three-component solution for CO_2 removal is the Sulfinol Process* (2-4). This is a relatively new process for the removal of acid gases (CO_2, H_2S, carbonyl sulfide, mercaptans, etc.) from a variety of sour gas streams. The Sulfinol Process uses a mixed solvent of sulfolane (tetrahydrothiophene dioxide, also known as tetramethylene sulfone) and any one of several alkanolamines. The resultant two-component solution is nonaqueous but quite hygroscopic.

Equilibrium considerations of CO_2 absorption and desorption will be first discussed by making a comparison of the Sulfinol system with the presently used aqueous monoethanolamine (MEA) system. The rationale for the addition of the third component, H_2O, will then be somewhat evident.

ABSORPTION EQUILIBRIA

The features of CO_2 absorption are shown in Fig. 1 (from Ref. 2) in which the equilibrium solution loading in terms of volumes of CO_2 at standard conditions per unit volume of solution at room temperature, V/v, is plotted against CO_2 partial pressures. The curve for aqueous MEA is typical for the case of absorption with chemical reaction. At low CO_2 partial pressures, the solution loadings are relatively high because of the chemical reaction. As the CO_2 partial pressure increases, the stoichiometric limit of 1 mole of CO_2 per 2 moles of MEA is exceeded, and the slope of the curve increases abruptly. The solubility of CO_2 in H_2O alone at low CO_2 partial pressures is negligible and becomes significant only at CO_2 partial pressures of several hundred psi. Because sulfolane is a much better physical solvent for CO_2 than is H_2O the curve for the Sulfinol Process solvent clearly reveals a superiority in CO_2 capacity at intermediate and high pressures. Unfortunately, in the region of interest for submarine use (low CO_2 partial pressures), the Sulfinol Process solvent is not quite as effective as aqueous MEA.

*Shell Development Company service mark.

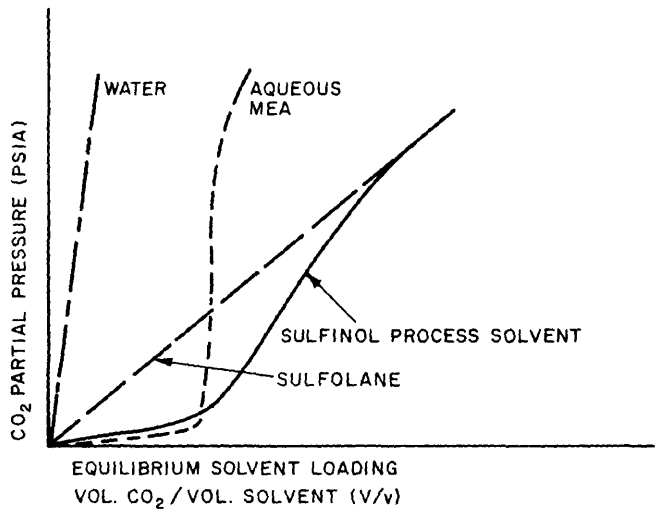

Fig. 1 - CO_2 absorption isotherms

DESORPTION EQUILIBRIA

The features of CO_2 desorption are shown in Fig. 2 (from Ref. 2), which compares equilibrium CO_2 loadings of the Sulfinol Process solvent and aqueous MEA at absorption and desorption temperatures. The regeneration of the carbonated solvents is clearly much easier for the Sulfinol solvent than for aqueous MEA at the same temperature, since the difference in solution loading or the net CO_2 removal is greater for the Sulfinol solvent. A notable observation of Deal, Dunn, et al. (2), is: "The difference is over and above that expected from the fact that in the Sulfinol Process solvent only part of the acid gas is chemically held, whereas in aqueous alkanolamine almost all of the acid gas is so bound."

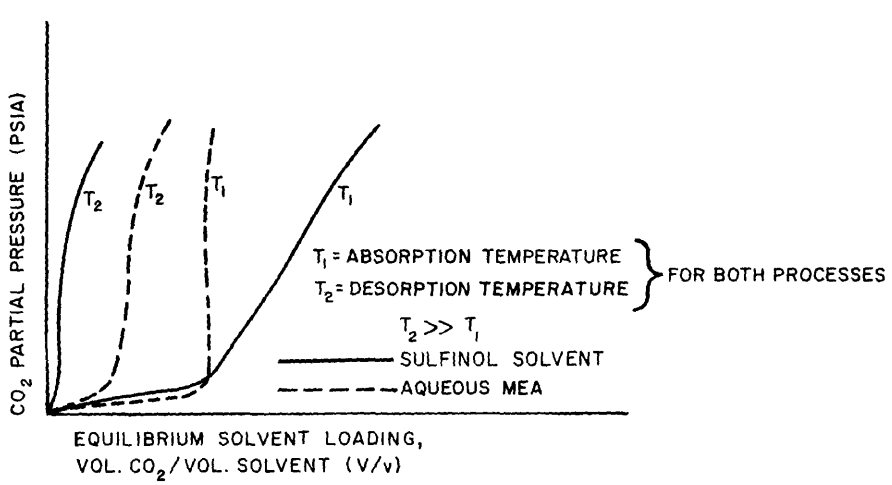

Fig. 2 - CO_2 absorption-desorption isotherms

THE THIRD COMPONENT

Although the Sulfinol Process has some outstanding features for CO_2 removal, it is most efficient in high-pressure operation. Therefore, some solvent modification was necessary for treating atmospheric air with low CO_2 partial pressures as found on nuclear submarines. One obvious change was to make the solvent a three-component system by adding H_2O. Under the more restrictive low pressure conditions, H_2O should help in the absorption part of the cycle by tending to diminish the difference in performance between the Sulfinol solvent and aqueous MEA. This improvement could come from better wetting of the absorber packing and from faster CO_2 diffusion as a result of a decrease in solvent viscosity.

Under the same CO_2 solution loading, the three-component system should absorb CO_2 about as well as aqueous MEA. If improved regeneration allowed the CO_2 loading of the new solution to be reduced to a level lower than is currently possible for aqueous MEA, the absorption capability of the three-component solution would be superior and the ambient CO_2 could be maintained at a more desirable level.

TEST PROCEDURE

Data for the three-component system were taken with the laboratory tube reboiler, and the experimental procedure was the same as previously reported (1). A change in the apparatus, however, was required with the appearance of two liquid phases. To obtain the required data, a circulating pump was added to the feed storage tank so that a homogeneous solution could be fed to the reboiler tube. The only other difference was the introduction of the feed solution into the vapor space of the reboiler instead of under the liquid level. The new feed location eliminated rotameter disturbances caused by the boiling liquid.

RESULTS, DISCUSSION, AND CONCLUSIONS

Comparisons between laboratory and shipboard data are somewhat difficult, because shipboard conditions are variable and complete information is usually not available. Studies relating CO_2 solution loadings to ambient CO_2 concentrations aboard submarines, for example, are complicated by events such as simultaneous operation of two scrubbers, replacement of absorbent solutions, addition of makeup solution, and atmospheric changes due to snorkeling or surface ventilation.

The desorption rates for CO_2 from aqueous MEA in a land-based full-size scrubber are given for various ambient percentages of CO_2 in Table 1 (5). Unfortunately, the CO_2 solution loadings under the test conditions were not available, and a comparison of these data with laboratory results can be made only by assuming values of V/v which experience has shown to be representative. The relationship between the ambient CO_2 level and CO_2 content of the rich scrubber solution aboard submarines was determined for the February 1961 cruise of the USS SKIPJACK (6). On this cruise an ambient CO_2 level of 1% corresponded to a V/v of 23 for the rich solution which had a normality of 3.8. In the same report, the plot of the relation between CO_2 content of the air, MEA normality, and refractive index clearly reveals that solutions of higher normality contain more CO_2 at a given ambient CO_2 level. Therefore, for 4.5N MEA, the V/v corresponding to the ambient level of 1% CO_2 should be higher than 23.

Table 1
Desorption Results from a Land-Based Scrubber, Mark II
(Aqueous MEA)

Given Data			Derived Information		
Ambient CO_2 (%)	CO_2 Removal Rate (lb/hr)	Condenser Duty (Btu/hr)	Change* in V/v	lb Steam† / lb CO_2	Condenser Duty (Btu/hr) at Stripping Rate of 1/8 gal/min
1.5	16	13,900	16.3	0.9	1740
1.0	12	16,300	12.2	1.5	2040
0.5	6	19,500	6.1	3.5	2440

*Decrease in CO_2 solution loading at a stripping rate of 1 gal/min.
†The steam rate is equal to the condenser duty divided by the heat of vaporization of H_2O at 45 psia.

In consideration of the previous paragraph, it is not unreasonable to assign a rich V/v value of about 30 for the 1.0% CO_2 level of a 4.5N MEA solution. This indicates a rich V/v value of about 40 at 1.5% and perhaps 20 at 0.5% CO_2. Confirmation can be found in laboratory experience with desorption and in data to be presented later that the low steam-to-CO_2 ratio of 0.9 in Table 1 at an ambient level of 1.5% is indicative of the ease of stripping associated with solutions highly loaded with CO_2 and thus provides justification for the estimate of a rich V/v of 40. Since it is difficult to obtain lean V/v values close to 10 (particularly in solutions of higher normality), the estimated rich V/v of 20 at 0.5% CO_2 also appears to be reasonable because the lean V/v, obtained by subtracting the change in V/v of 6.1, would be about 14. These estimates are based on the assumption that sufficient time has been allowed for the solution in a single scrubber operation to reach these values.

A comparison between Tables 1 and 2 reveals that the changes in V/v are much higher in the tube reboiler at lower steam-to-CO_2 ratios, although at mostly higher energy inputs as indicated by the condenser duties. The output of CO_2 ranges from about 1/3 to over 1/4 of the full-size scrubber at 1/8 the stripping feed rate. The stripped solutions had very low CO_2 loading with all values of V/v being less than eight. Run 5B, however, is exceptional in its excellent performance at a deliberately maintained low energy input. The temperatures of Table 2 are higher than the recommended operating range of 280 to 285°F for aqueous MEA. The lowest reboiler pressure, 21 psig, resulted in the lowest temperature, 287°F.

Because desorber pressures less than 30 psig may not be practical in the operation of the existing CO_2 compressors on shipboard scrubbers, further investigations were made at and above 30 psig. To decrease the operating temperatures and reduce the degradation of MEA, the three-component solution was diluted with H_2O and MEA was added to maintain normality. The results are shown in Table 3, which also includes data for another three-component system containing propanolamine instead of MEA and for the corresponding two-component aqueous solutions of propanolamine and MEA.

Table 2
Initial Results of Desorbing CO_2 from a Three-Component Solution* in the Tube Reboiler (stripping feed rate of 1/8 gal/min)

Run No.	V/v Rich	V/v Lean	Change in V/v	CO_2 (lb/hr)	Condenser Duty (Btu/hr)	Lb Steam / Lb CO_2	Reboiler† Pressure (psig)	Temperature‡ (°F)
4	41.8	4.7	37.1	4.6	2500	0.6	27.2	302
5A	33.1	4.5	28.6	3.9	2800	0.8	26.5	297
5B	33.1	7.7	25.4	3.2	1400	0.5	26.1	295
6A	21.8	7.4	14.4	1.9	3700	2.2	21.2	287
6B	21.9	6.9	15.0	1.9	4000	2.2	21.0	287

*52% Sulfolane, 27.5% MEA, and 20.5% H_2O (all percentages on volume basis).
†Minimum pressure found.
‡Temperature at reboiler exit.

Table 3
Results of Desorbing CO_2 from Amines in the Tube Reboiler*

Solution†	V/v Rich	V/v Lean	Change in V/v	CO_2 (lb/hr)	Temperature (°F)	Condenser Duty (Btu/hr)	Lb Steam / Lb CO_2
4.3N MEA	45.4	17.8	27.7	3.3	282	6200	2.0
	43.5	18.6	24.9	3.0	278	6200	2.2
	43.4	25.1	18.3	2.3	272	1400	0.7
	34.3	15.3	19.0	2.2	283	3600	1.8
4.5N MEA, sulfolane, and 30% H_2O	49.6	8.6	41.0	4.9	296	3400	0.8
	31.4	7.1	24.3	2.9	293	5100	1.9
4.5N MEA, sulfolane, and 40% H_2O	43.8	11.3	32.5	4.4	287	7900	1.9
4.6N 3-amino-propanol, sulfolane, and 30% H_2O	44.1	13.2	30.9	3.7	293	4200	1.2
	31.8	11.8	20.0	2.7	291	4500	1.8
4.3N 3-amino-propanol	44.6	21.1	23.5	2.6	280	6200	2.6

*The stripping rate was 1/8 gal/min, and the reboiler pressure was 30 to 36 psig.
†All H_2O percentages are volume percents.

In Table 3 the results in terms of changes in V/v are better for all solutions than in Table 1. The condenser duties are mostly higher in the tube reboiler than in a full-size scrubber — probably higher than necessary, since it was easier to insure steadier operation and to collect more reliable data at higher energy inputs. The steam-to-CO_2 ratio, however, is not unreasonable in any of the runs. (The aqueous MEA runs with steam-to-CO_2 ratios of 0.7 and 1.8 at V/v values of 43 and 34, respectively, are notable in providing support for the estimates of V/v made in Table 1.) Some allowance should be made in the rough comparisons for variations of ±5% in the 1/8-gal/min stripping rate. The feed rate was also subject to small variations during a run because of minor fluctuations in reboiler pressure.

Comparison between Tables 1 and 3 will indicate the extent of the contribution of each of the two factors — reboiler redesign and the presence of sulfolane — to desorption improvement. Considering the aqueous MEA run in Table 3 with a condenser duty of 1400 Btu/hr, there is a gain of at least 12% in the change in V/v over the corresponding run in Table 1. This indicated that improvement effected by the improved reboiler design could be slightly better than 12% at 1700 Btu/hr and at a temperature which is usually higher than 272°F. Comparing the three-component and two-component solutions of Table 3, it is evident that the addition of sulfolane has improved the desorption capabilities of both the aqueous MEA and aqueous propanolamine solutions. If the results of Table 2 are ignored, the most effective solution, with very low CO_2 loadings of the lean solution, is the three-component solution containing sulfolane, MEA, and 30 vol-% H_2O. The second-best solution, containing sulfolane, MEA, and 40 vol-% H_2O, however, may be the most practical choice for overall performance when absorption ability, reduction of viscosity, and self-regulation of lower desorption temperatures are all considered. The remaining solutions, ranked in order of their facility in desorption, are the three-component solution with sulfolane, propanolamine, and 30 vol-% H_2O; aqueous MEA; and aqueous propanolamine.

An increase in the concentration of H_2O, therefore, reduces the desorption capability of the three-component solution but not enough to offset a still impressive gain over aqueous MEA. Sulfolane's contribution, for example, is evident even with a three-component solution containing 40 vol-% H_2O as revealed by a comparison in Table 3 with the second run of aqueous MEA. Even if allowance is made for the difference in temperature between 287°F and 278°F, and between condenser duties of 7900 and 6200 Btu/hr, the increase in desorption is still nearly 30%.

FAVORABLE SULFOLANE PROPERTIES

Characteristics of sulfolane are mostly favorable for its proposed use aboard submarines. Sulfolane is a highly polar compound which is generally unreactive and has good thermal stability up to temperatures of 428°F (4). It is a high boiling compound, 545°F, with low volatility.

Pilot plant data (3,7) of the two-component Sulfinol Process system reveal the following advantages over aqueous MEA:

1. Lower solvent entrainment and volatility losses.

2. Lower energy input due to ease of stripping and lower solvent heat capacity.

3. Reduced foaming tendencies.

4. Apparent inhibition of MEA degradation.

While most reports on the Sulfinol Process are ambiguous in their reference to the alkanolamine component, the Person plant in Texas definitely used diisopropanolamine (3) (probably because DIPA is relatively insensitive to degradation by carbonyl sulfide). Over a 10-month period, the degradation rate for aqueous MEA was 8 times greater than for DIPA in identical plant service.

Degradation of amines used as absorbents for CO_2 in the presence of O_2 results in serious losses of alkalinity and the formation of corrosive products. Consider now the data obtained by an accelerated oxidation test in which the aqueous amines at 176°F were exposed to a gas mixture of 50% CO_2 and 50% O_2, as shown in Table 4 (8). The results for diethanolamine (DEA) are included to aid in the comparison.

Table 4
The Relative Effect of Oxidation on 2.5N Aqueous Solutions of Various Amines (Free Amine)*

Amine	Before Exposure (eq/l)†	After Exposure (eq/l)	Weight Loss of Amine (%)
MEA	2.47	1.34	45.7
DEA	2.54	0.98	61.4
DIPA	2.45	0.18	92.7

*Free Amine refers to an amine which is available to react with CO_2. It is that amine which is titratable with a strong acid using methyl red as an indicator.
†Equivalents per liter.

The stability of DIPA in the Sulfinol Process at the Person plant versus the degradation shown in Table 4 illustrates the protection provided by sulfolane. From this effect, it may be assumed that MEA might be protected by sulfolane even in a three-component system.

The advantages mentioned in the pilot plant data for the Sulfinol Process are expected to remain favorable in a three-component system. It has been shown in the laboratory that sulfolane reduces foaming in a three-component system (9). The same study also indicated that the three-component system can reduce the effluent CO_2 to a lower level than does aqueous MEA. Air containing 1.0% CO_2 was passed through various amines, and a relative measure of absorption efficiency was obtained by monitoring the percentage of CO_2 in the effluent air with time. The data are pertinent to this discussion and are repeated here in Table 5.

The first factor to consider in this table is the effect of foaming. Aqueous MEA foamed to a greater extent than the three-component system as seen in the height-of-foam column. While the most effective CO_2 absorption was obtained with a foam height of 3 to 10 in., this is a misleading efficiency because foaming is detrimental to scrubber operation for several reasons. The main objection to foam is the reduced overall capacity resulting from a higher pressure drop and decreased airflow. The three-component system would, therefore, appear better than shown in the table if it were to be compared with equivalent acceptable foam heights for aqueous MEA.

Table 5*
Percent CO_2 in the Effluent Versus CO_2 Loadings†

Sample	Foam Column Height (in.)	Effluent CO_2 (%) at CO_2 Loadings (V_g/V_s)‡ Indicated								V_g/V_s at Saturation
		0	10	20	30	35	37	40	50	
4N MEA-H_2O	3-10	0.20	0.10	0.20	0.30	0.40	0.50	0.80	—	42.4
4N MEA-H_2O	3-10	0.15	0.10	0.10	0.30	0.40	0.45	0.70	—	41.9
4N MEA-H_2O	1-10	0.25	0.25	0.25	0.30	0.45	0.55	0.80	—	41.9
4N MEA-TMS-20%§ H_2O	1/2-1	0.10	0.15	0.25	0.50	0.65	0.75	1.00	—	39.5
4N MPA-TMS-20%¶ H_2O	1/2-1	0.10	0.10	0.25	0.45	0.65	0.75	1.00	—	39.7
5N MPA-TMS-25% H_2O	1/2-1	0.10	0.15	0.20	0.30	0.35	0.40	0.45	1.00	50.1

*From Ref. 9.
†The conditions were 3.3 l/min of 1.0% CO_2 in air at 88°C and 100% RH, using a 50 ml sample.
‡Same as V/v.
§TMS = Sulfolane; all H_2O percentages are volume percent.
¶MPA = 3-aminopropanol.

The next factor of interest is the comparison of absorption efficiency of the three-component solution at low CO_2 loadings versus aqueous MEA at higher CO_2 loading. Such a comparison is valid, since the three-component solution has a much better desorption capability and should operate with significantly leaner solutions. Table 5 shows that the MEA-sulfolane-H_2O solution has an effluent CO_2 percentage of 0.25 at a V/v value of 20, whereas aqueous MEA with the closest equivalent foam height (1 to 10 in.) has effluent CO_2 percentages from 0.30 to 0.80% over a V/v range of 30 to 40. While the particular MEA-sulfolane-H_2O solution investigated shows a decrease in absorption efficiency at V/v values of 30 and above, a remedy is suggested by comparing the results for the amino-propanol solutions where increased efficiency is shown for the higher normality. The three-component solution containing MEA would similarly be expected to be a better absorbent if it were 5N MEA.

POSSIBLE DISADVANTAGES

Toxicity

Sulfolane is classified as a slightly toxic compound. Its LD_{50} oral toxicity on rats is in the 500 to 5000-mg/kg range, while its LD_{50} percutaneous toxicity on rabbits is higher than 2820 mg/kg, the highest dose applied to the skin. Twenty-four hour exposure to the skin of rabbits produced no irritation (4). In the proposed use, the admixture with a caustic substance, MEA, would provide a warning for immediate removal from the skin.

Impurities

The main impurity in commercial sulfolane (Shell, Phillips) is 3-sulfolene (up to 2 wt-%), which decomposes thermally at temperatures well below the boiling point of sulfolane into butadiene and SO_2 (10). Purification of sulfolane was accomplished in the laboratory (10) with no difficulty, and presumably commercial sulfolane could be purified economically.

Absorption of Hydrocarbons

Since the ambient concentration of hydrocarbons is low on submarines, the absorption capacity for C_5^+ and higher components would probably be negligible. While the higher absorption of aromatics might be significant, it is conceivable that the CO_2 compressor would be able to handle the small quantities involved without additional purification equipment or modification.

Flammability

Tests for flammability of the three-component system with 15 wt-% H_2O were made by passing a fine spray of the solvent through a Bunsen burner flame. There was no ignition. A portion of the solvent on a heated metal plate did not catch fire until the H_2O boiled off, and then the combustion was not particularly vigorous.

Viscosity

A 4.5N MEA-H_2O solution at 86°F has an absolute viscosity of 2.4, which increases to 3.3 when fully carbonated, whereas the uncarbonated 4N MEA-sulfolane-20% H_2O solution has a viscosity of 6.0 centipoise (9). An increase in the H_2O content of 40 or 45 vol-% would help, but, in any event, the three-component solution will be more difficult to pump.

Freezing Point

The freezing point of 82°F is a handicap in handling the undiluted solvent. The addition of 3 wt-% H_2O alleviates this problem considerably by lowering the freezing point to about 48°F. This property would present no problem in scrubber operation.

Two-Phase System

Sulfolane, MEA, and H_2O are miscible with each other in all proportions. However, the introduction of CO_2 results in two liquid phases at V/v values above 10. The bulk of the MEA carbonate is found in the upper lighter phase, while most of the lower phase is sulfolane. (Density of sulfolane is 1.261 g/cc at 86°F.) The lower phase (about 94 wt-% sulfolane) was usually about 30 to 40% of the total volume. Sulfolane has appreciable solubility (26 wt-% in one sample) in the upper phase. On the other hand, the solubility of MEA carbonate is very low (3 wt-% or less) in the lower phase. The separation of the phases by gravity is quite rapid, which presents analytical problems. Phase separation should not, however, be a problem in the CO_2 scrubber. A recycle rate of 20 to 27

gal/min should keep about 40 gal of absorber solution rather well mixed. In the desorber, attempts might be made to use the phase separation to an advantage, since only part of the stream, the upper phase (still three components), would need to be processed. (The three-component system with propanolamine also formed two liquid phases after being carbonated.)

Logistics

The logistics of the new system would be simplified if MEA and sulfolane were stored together in the proper ratio. Common storage would presumably lessen the rate of degradation of MEA. In scrubber operation, H_2O could be added, as at present, to maintain solution level in the absorber, while the mixed solution would be added as needed to adjust solution normality. It is believed some tolerance in solution composition would be permissible. The logistic problem could also be partially diminished by a longer service life than for aqueous MEA.

RECOMMENDATIONS

The excellent desorption results obtained with sulfolane emphasize the desirability of testing the three-component solution in a full-size land-based scrubber of the present design. The initial solution should contain about 40% H_2O, 27.5% or 4.5N MEA, and about 32.5% sulfolane, all percentages being volume percent. The composition of 40% H_2O, 30% or 5N MEA, and about 30% sulfolane, while not tested here, should also be effective. The other three-component solution containing propanolamine should also be tested, if a slightly less effective performance can be tolerated in order to eliminate MEA from submarines.

This work has reemphasized the potential gain in overall efficiency that could be attained with a reboiler incorporating the features suggested in Ref. 1. The combination of a more effective absorbent and a redesigned stripper should achieve lower ambient CO_2 levels than presently possible aboard submarines.

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13. ABSTRACT

New absorbent solutions containing three components have been evaluated for efficiency in decreasing the ambient CO_2 level aboard submarines. One of the absorbent systems investigated consisted of a high-boiling organic compound, sulfolane, which has a high physical absorption capacity for CO_2, combined with monoethanolamine and water. The other absorbent system had the same components except for the amine, which was 3-aminopropanol.

At the same level of saturation of the solutions with CO_2, the absorption efficiency of the three-component solutions is about the same as, or slightly inferior to, the corresponding two-component aqueous amines. However, desorption results with the three-component solutions are so much better than for the two-component aqueous amines that the lower CO_2 loadings that would be attained in the absorber section of the present CO_2 scrubbers should result in significant reduction of the ambient CO_2 concentrations in nuclear submarines.

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