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Comparing laboratory and plant data for MDEA/DEA blends

A comprehensive survey has insightful results

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GAS/SPEC Technology Group

INEOS LLC

As part of a Federal Trade Commission mandated remedy to the merger of The Dow Chemical Company and the Union Carbide Corporation, INEOS plc was able to purchase both Dow's Ethanolamines and GAS/SPEC MDEA-based specialty amine businesses. This purchase became effective on February 12, 2001.

INEOS LLC was set up as the newly acquired company, which includes the GAS/SPEC Technology Group. All the key Ethanolamines and GAS/SPEC personnel were retained by INEOS LLC. All GAS/SPEC products, technology and know-how became the exclusive property of INEOS on a global basis.

Comparing laboratory and plant data for MDEA/DEA blends

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Degradation of diethanolamine (DEA) with CO₂ to form byproducts has been demonstrated as early as 1956. Literature laboratory data has shown that DEA in methyldiethanolamine (MDEA)/DEA blends also experiences significant degradation compared to MDEA. Our lab research data confirms this effect.

However, laboratory autoclave corrosivity tests show that these solutions have less than 1 mil per year (mpy) corrosion to carbon and stainless steel coupons. Plant data is presented from many plants using MDEA/DEA blends, some operating since 1989, showing that corrosion and DEA degradation of MDEA/DEA is much less of a problem than lab degradation data indicates.

We have reviewed data from 98 operating plants that use DEA as their solvent. Of the 98 plants, only six had total DEA degradation products greater than 2 wt%. All 92 plants that had DEA levels with low degradation

had DEA concentrations of about 20 wt%. Only when the DEA concentration reached about 30 wt% did DEA degradation products become much more significant. H₂S levels do not appear to lower DEA degradation when DEA concentration is at the 30 wt% level. This can be seen by the one plant that had significant DEA degradation even with 171-psia H₂S partial pressure.

BACKGROUND

MDEA/DEA blends have been used successfully in a wide variety of gas conditioning applications for almost 20 years. Based on laboratory research in the literature dating as far back as 1956 and continuing through the present day, DEA and MDEA/DEA blends have been reported to be susceptible to degradation by CO₂, resulting in corrosion and reduced solvent performance. Do laboratory degradation and corrosion studies accurately reflect field experience with MDEA/DEA blends?

Through a combination of our own laboratory research and an extensive review of plant operating solutions and corrosion history, we have been able to determine that corrosion and DEA degradation by CO₂ in MDEA/DEA blends is much less of a problem than laboratory degradation data indicates.

Table 1. Poldermann and Steele study of 25 wt% DEA saturated with CO₂, 8-hr test

Temperature, °F	CO ₂ partial pressure, psig	Wt% DEA converted to BHEP
212	180	nil
230	195	5
248	250	22
275	325	56
302	520	92
347	600	97

Table 2. McNab and Treseder study of 20 wt% DEA and corrosion rates of carbon steel

	140°F	212°F	240°F
CO ₂ Only	3 mpy	17 mpy	27 mpy
CO ₂ :H ₂ S 3:1	3 mpy	6 mpy	11 mpy

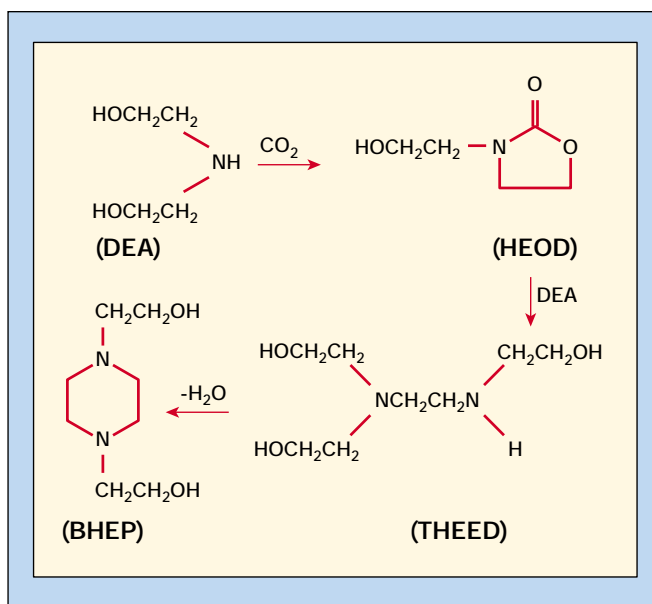


Fig. 1. Degradation of DEA to HEOD, THEED and BHEP.

Table 3. Chakma & Meisen Tafel plot results at 77°F with no CO₂ 1020 carbon steel

Solution	Corrosion rate, mpy
30 wt% DEA	2.7
22.5 wt% DEA + 4.65 wt% HEOD + 2.49 wt% THEED + 0.37 wt% BHEP	16.1

Table 4. Chakma and Meisen autoclave weight loss test results for 30 wt% DEA at 212°F on 1020 carbon steel

CO ₂ partial pressure, psig	Corrosion rate, mpy
200	24.0
403	25.6
599	31.5

Table 5. Chakma and Meisen boiling point weight loss test results at 212°F on 1020 carbon steel

Solvent	Corrosion rate, mpy
30 wt% DEA	<1
40 wt% DEA	<1
60 wt% DEA	<1
15 wt% DEA + CO ₂	5.1
15 wt% BHEP + CO ₂	6.3
15 wt% HEOD + CO ₂	76.6
30 wt% DEA + CO ₂	63.1
30 wt% DEA + 5 wt% BHEP + CO ₂	62.0
30 wt% DEA + 5 wt% HEOD + CO ₂	75.0
40 wt% DEA + CO ₂	72.3
60 wt% DEA + CO ₂	81.6

REVIEW OF DEA DEGRADATION AND CORROSION PAPERS

In 1956, Polderman and Steele¹ discovered N,N'-bis(2-hydroxyethyl) piperazine (BHEP) in DEA CO₂ degradation studies. They also proposed a 3-(2-hydroxyethyl) oxazolidone (HEOD) to BHEP degradation route. They indicated that BHEP was basic and capable of absorbing acid gas and appeared to be noncorrosive to iron and steel, although they presented no corrosion data to support this conclusion. They also found the rate of degradation appeared to be dependent on temperature and CO₂ partial pressure. Table 1 lists a summary of their findings.

McNab and Treseder² in 1971, conducted corrosion tests of carbon steel in 20 wt% DEA solutions at 140°F, 212°F and 240°F in CO₂ only and mixed H₂S/CO₂ environments. Their results showed that corrosion increased with temperature. They also showed corrosion was lower in mixed H₂S/CO₂ acid gas service when compared to CO₂ only. Table 2 lists a summary of their work.

In 1977, Choy and Meisen³ could not produce HEOD or BHEP at 140°F with 30 wt% DEA that was CO₂ saturated at atmospheric pressure. Tests at 284°F and 600 psig CO₂ partial pressure with 30 wt% DEA also had no significant degradation. However, there was significant formation of BHEP during tests

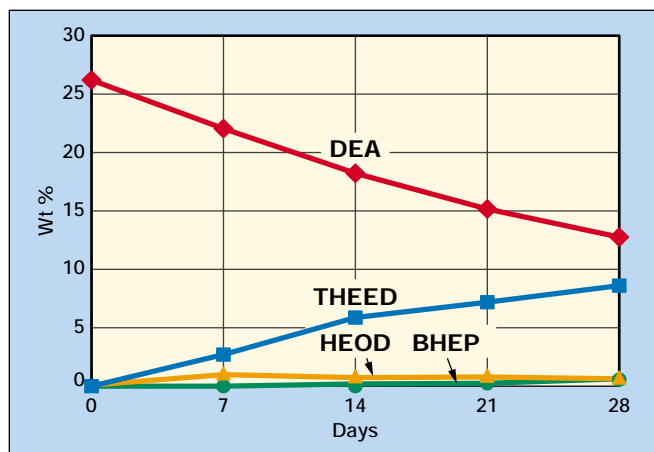


Fig. 2. Results of autoclave degradation of the MDEA/DEA solution performed at 260°F and 25 psig CO₂ partial pressure.

run at 347°F and 600 psig CO₂ partial pressure with 30 wt% DEA.

Kennard and Meisen⁴ in 1980 found HEOD, N,N'-tris(2-hydroxyethyl)ethylene-diamine (THEED) and BHEP in 25 wt% DEA solutions that were tested from 212°F to 356°F in solutions that were CO₂ saturated. They found no degradation of DEA solutions that were tested up to 401°F under a nitrogen pad. They also showed that degradation increased with DEA concentration. Degradation also appeared to be limited once BHEP equilibrium was established, and that the presence of H₂S also appeared to inhibit degradation. They also claimed BHEP was corrosive, but did not have any corrosion data to support this claim.

In 1982, Blanc, Grall and Demarais⁵ conducted degradation studies of 30 wt% DEA from 194°F to 230°F at 0.45 M/M of CO₂. They also studied 30 wt% DEA at 266°F and 87-psia CO₂ partial pressure. They proposed a HEOD → THEED → BHEP degradation route (Fig. 1). They also concluded that degraded solutions containing BHEP and H₂S (0.8 mpy) have lower corrosion rates than fresh DEA (2.0 mpy). These tests were run at 176°F and 290-psia H₂S partial pressure.

Chakma and Meisen⁶ in 1984, found that degradation of DEA increased with higher temperature, DEA concentration and CO₂ partial pressure. In dynamic tests, they noted that the rate of degradation fell when amine flowrate was increased. They also observed fouling along with degradation. A mathematical model for predicting DEA degradation was also presented. Degradation studies were conducted with 20 to 40 wt% DEA, 140°F to 392°F, 200 to 606 psia CO₂ partial pressure and 0.17 to 0.27 gal/min amine flowrate.

Also in 1984, Kim and Sartori⁷ conducted degradation studies with 34 wt% DEA at 212°F and 248°F using various CO₂, H₂S and CO₂ / H₂S loadings. They confirmed the HEOD → THEED → BHEP degradation route. They also indicated that DEA degradation by CO₂ is dependent on temperature and CO₂ partial pressure. They also presented their own kinetic model for predicting DEA degradation.

Kennard and Meisen⁸ in 1985 performed degradation testing of 30 wt% DEA at 194°F to 482°F and CO₂ partial pressures of 218 to 1,000 psia. They confirmed that HEOD, THEED and BHEP are major

Table 6. Autoclave data for MDEA / DEA blend after 28 days

	29.79 wt% MDEA + 26.25 wt% DEA
Metals by ICP, ppmw	
Cr	85
Fe	202
Ni	57
Coupon Corrosion, mpy	
Carbon Steel (liquid)	0.3
304SS (liquid)	0.2
316SS (liquid)	0.9
410SS (liquid)	0.1
Carbon Steel (vapor)	1.4
304SS (vapor)	0.1
316SS (vapor)	0.2
410SS (vapor)	0.2
Calculated ppmw metals from data	
Cr	226
Fe	1,336
Ni	160

degradation products; that DEA degradation is dependent on temperature and CO₂ partial pressure; and that an equilibrium level of BHEP reduces the DEA degradation rate. They also developed a kinetic model for predicting DEA degradation. They noted that as the pH of the solution becomes more acidic (by addition of CO₂), the degradation rate is enhanced. In further testing of various DEA strengths, they observed very low degradation in 0 to 10 wt% solutions and increased degradation in the range of 11 to 30 wt% DEA solutions.

Also in 1985, Hsu and Kim⁹ discovered triamines in addition to HEOD, THEED and BHEP during testing of 33 wt% DEA solutions at 248°F to 284°F and either 0.44 M/M CO₂ loading or 0.43 M/M CO₂ + 0.10 M/M H₂S. They also suggested that degradation leads to reduced performance of DEA solutions.

Chakma and Meisen¹⁰ in 1986 proposed a mechanism for corrosion in DEA—that CO₂ absorption, degradation products and amine heat stable salts lower solution pH, which increases solution corrosivity. They also claim degradation products contain metals and act as chelating agents, further contributing to corrosivity. The authors ran Tafel plots, autoclave weight loss tests and boiling point weight loss tests. Tables 3, 4 and 5 list the results of their corrosion testing. They observed that boiling point weight loss corrosion rates were higher than expected and surmised that boiling disturbed formation of a protective passive layer.

In 1996, Dawodu and Meisen¹¹ tested 50 wt% amine solutions ranging from 100% MDEA to 100% DEA with various blends at 248°F to 356°F and 374 psia CO₂ partial pressure. They concluded that MDEA/DEA blends require more maintenance to keep the MDEA/DEA ratio from changing, but had very little data to support this claim. They also looked at the difference in degradation by CO₂ of MDEA, DEA and monoethanolamine (MEA) and concluded that MDEA was the most resistant, followed by MEA and then DEA. While noting MDEA does degrade in the pres-

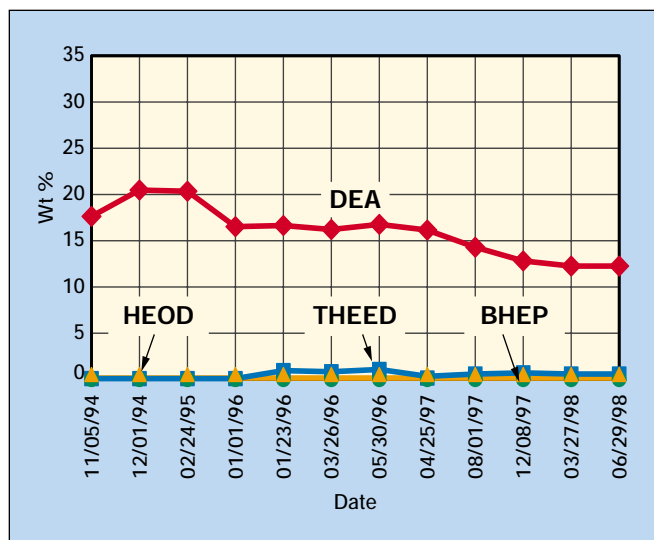


Fig. 3. Plant A MDEA/DEA blend over time showing DEA degradation (61 psia CO₂ partial pressure).

ence of CO₂, they acknowledged that the degradation rate was low.

Holub, Critchfield and Su¹² in 1998 reviewed amine degradation chemistry in CO₂ service. They concluded, after accelerated laboratory autoclave degradation tests at 250°F and 300 psig CO₂ partial pressure for 28 days,¹³ that DEA (alone) and DEA in MDEA/DEA blends degrade at unacceptable rates, adversely affecting solvent performance. However, these tests may overestimate plant degradation since few plants operate at such a high CO₂ partial pressure.¹³ Holub, et al., also linked CO₂ degradation products of DEA to corrosion by characterizing these products as metal chelating agents. Finally, they suggested that metals level in a DEA solution or MDEA/DEA blend is indicative of active corrosion. However, no corrosion data was presented to verify that these solutions were corrosive.

EXPERIMENTAL DETAILS

General. MDEA and DEA were tested to be >99% pure by gas chromatography (GC). Distilled water from a local storage facility was used as the water source for all experiments.

Autoclave and corrosion studies. Corrosion rates of different metals with the alkanolamine mixtures were measured using weight loss coupons in 2-L, 316SS pressure reactors (rated at 2,000 psi) with magnetic stirrer drives connected to temperature controllers. Each reactor head was custom fitted with a 316SS rack. Carbon steel, 304SS, 316SS and 410SS coupons were totally immersed in the liquid and additional coupons were fully placed in the vapor space above the liquid. Each coupon was mounted to the rack using Teflon spacers to fully insulate the coupons from the rack.

The solution to be studied—1,200 g total solution was used in each test—was placed in the reactor (glass liners were not used in any of these tests since silicates are known corrosion inhibitors in industrial cool-

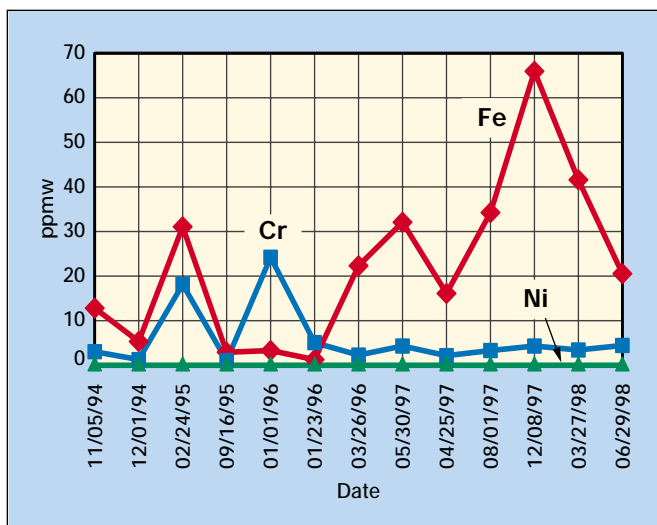


Fig. 4. Plant A metal levels vs. time.

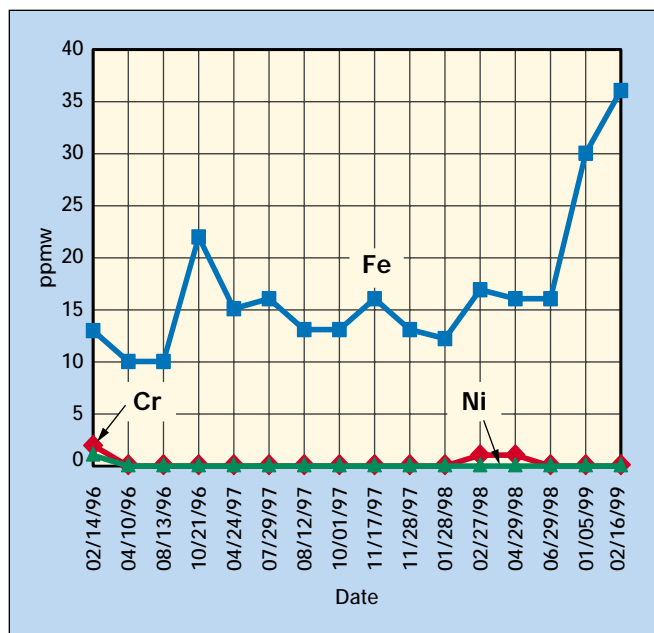


Fig. 6. Plant B metal levels vs. time.

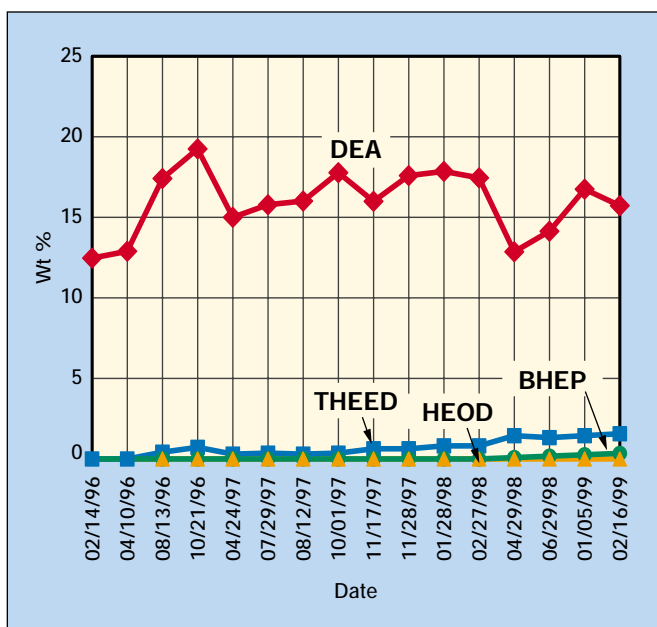


Fig. 5. Plant B MDEA/DEA blend over time showing DEA degradation (125 psia CO₂ partial pressure).

ing water systems and automotive cooling systems) and the reactor head was then bolted to the reactor to seal it. Nitrogen was passed through each solution before heating to remove any trace oxygen. CO₂ was then loaded at room temperature into the reactor via a 0.5-L sample bomb. When the proper CO₂ loading was obtained, enough sample was removed so that 1,100 g of solution remained in the autoclave.

The solutions were heated to the desired temperature (260°F), maintaining a stir speed of 190 rpm throughout the test. Each week, 20 g of sample was removed (the first 10 g was discarded) for sample analysis. After the test, the coupons were cleaned and the weight loss was measured. Coupon weight loss was converted to a corrosion rate in mpy using:¹⁴

$$\text{Corrosion rate} = \frac{\text{Weight loss} \times 534}{(\text{Area}) (\text{Time}) (\text{Metal density})}$$

where weight loss is in milligrams, surface of each

coupon is in square inches metal surface area exposed (each about, 1.5-in. long x 0.6-in. wide x 0.1-in. thick), test duration time is in hours exposed and density of each metal is in grams per cubic centimeter (carbon steel = 7.86 g/cm³, 304SS = 7.94 g/cm³, 316SS = 7.98 g/cm³ and 410SS = 7.70 g/cm³).

Before the tests, the C1018 carbon steel and 410SS coupons were cleaned with a 1:1 mixture of inhibited HCl (HCl containing 1,000 ppmw corrosion inhibitor) and water for 2 to 3 min., scrubbed with warm water and then dried with acetone. The 304SS and 316SS coupons were cleaned with a mixture of 1:1 nitric acid for 2 to 3 min., scrubbed with warm water and then dried with acetone. After the test, all coupons were cleaned with a 1:1 mixture of inhibited HCl, scrubbed with warm water and then dried with acetone.

RESULTS

Autoclave degradation and corrosivity experiments were performed on solutions of MDEA containing DEA. The solution used was 2.35M MDEA (29.79 wt%) and 2.35M DEA (26.25 wt%). Each solution was loaded with 1.1 wt% CO₂ (approximately 25 psig CO₂ partial pressure) and heated for 28 days at 260°F.

Fig. 2 shows the results of the MDEA/DEA solution degradation experiment. DEA decreases from 26.25 wt% to 12.66 wt% after 28 days. The THEED concentration increases to 8.47 GC wt%, whereas the HEOD and BHEP remained below about 0.75 GC wt% after 28 days.

Table 6 shows the amount of Fe, Cr and Ni detected by inductively coupled plasma (ICP) that is soluble in solution. The MDEA/DEA solution shows a high level of soluble metals. According to work done by other researchers,¹² this indicates that the solution is corrosive due to the chelating nature of the DEA degradation compounds, particularly THEED. However, the solution has low corrosivity to carbon steel and stainless steel weight loss liquid and vapor coupons, indicating that metal levels in the solution is not a good

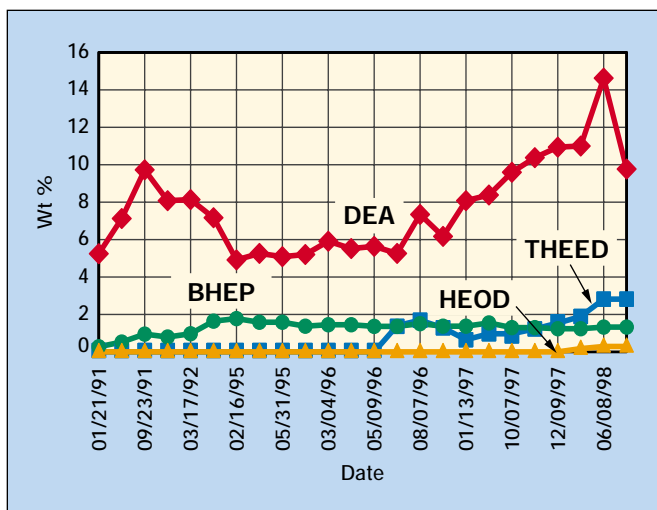


Fig. 7. Plant C MDEA/DEA blend over time showing DEA degradation (93 psia CO₂ partial pressure).

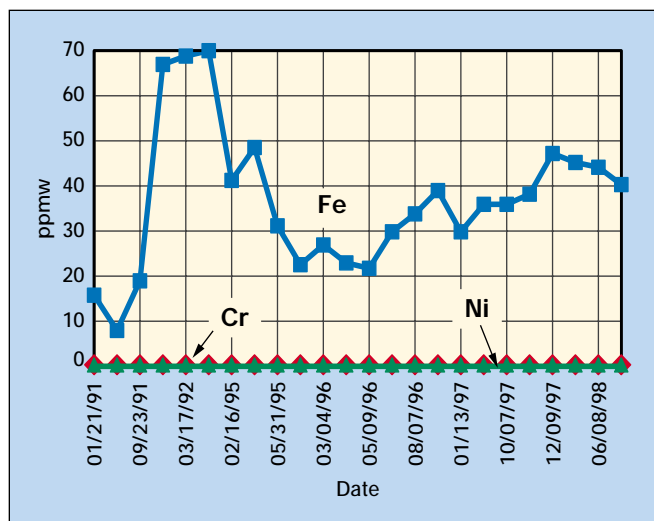


Fig. 8. Plant C metal levels vs. time.

indicator of solution corrosivity.

When the metals dissolved in solution is compared to the theoretical amount of metals that should be present, given the coupon weight losses measured and the surface area of the autoclave and coupons, the solution should have had much higher solubility of metals dissolved in solution. For example, 1,336 ppm of Fe should have been measured by ICP based on the calculation, but only 202 ppm Fe was dissolved in solution.

What this shows is that the MDEA/DEA solution has a solubility for metals that is much lower than the amount of metals that must be removed from the autoclave and coupons given the corrosion rate results. The remaining insoluble metals not detected are most likely precipitated in the 2-L autoclave or on the coupons and rack used in these experiments. It is expected that the same will happen in plant; that is, any excess metals that are not soluble in the solution will precipitate out of solution into low spots of the plant. For a more detailed discussion regarding why metals in solution may not be a good indicator of corrosion and how to calculate theoretical metal levels based on the corrosion rate and plant surface area, see reference 15.

We are aware of over 100 gas treating plants that use MDEA/DEA blends. Most of these plants operate well and do not show the amount of degradation and corrosion that various researchers have reported using laboratory degradation studies. In a review of some 25 applications over 8 years, for plants requiring to meet only a 2 mol% CO₂ outlet specification, degradation products accounted for less than 0.3 wt% of the operating solution. In many cases, there were no degradation compounds identified. Solvent formulations were very stable with no significant change in DEA or MDEA concentration requiring formulation adjustment. Although solvent make-up is unknown, and tech-

Table 7. Plant data using DEA only

DEA ^a	HEOD ^a	THEED ^a	BHEP ^a	CO ₂ pp ^b	H ₂ S pp ^b	Comments
30.4	0	2.86	0.17	64	0	Liquid/liquid treater
29.5	0	5.7	0	26	0.25	Liquid/liquid treater, hot oil reboiler, 245°F stripper bottom temp.
31.2	0	2.97	0.74	50	171	Gas/liquid treater
32.5	0	7.94	0.99	61	1.5	Gas/liquid treater
29.2	0	6.1	0.69	73	3.0	Gas/liquid treater, ethylene glycol as heat medium for reboiler, 244°F stripper bottom temp.
31.7	6.68	4.65	0.22	Unknown	Unknown	—

^aWt%

^bPartial pressure, psia

nically would affect the level of degradation components, the low vapor pressure of this type of specialty solvent formulation limits amine make-up requirements to that necessitated by normal operational upsets. An average number would be about 0.25 to 0.50 lb of amine / MMscf of gas treated.

Another application that is often well suited for MDEA/DEA blends is when a 1,000 ppmv treated gas CO₂ specification must be met. Generally, the solvent will contain more DEA in the blend to meet the lower CO₂ specification. In a review of the analytical history of plants using higher-strength DEA blends, it was found that degradation was minimal, although slightly higher than blends with a 2 mol% CO₂ outlet requirement.

Figs. 3 through 8 show metal levels (Fe, Cr and Ni) and solution degradation (DEA, HEOD, THEED and BHEP) vs. time for the three worst-case plants (A, B and C). These plants represent typical analytical trends that were observed in other higher strength DEA blend plant solutions. Each of these plants have fairly high CO₂ partial pressures in the absorber (61 to 100 psia). The inlet gas the plants treat does not contain any H₂S and the plants have several years experience using

MDEA/DEA blends. Even these three worst-case plants have low levels of DEA degradation products.

Plant experience with MDEA/DEA blends also confirms our findings that high metal levels do not always indicate corrosion. For the most part, corrosion has been a non-issue in a majority of the studied plants, with plant corrosion inspections bearing this out. Although corrosion testing is offered as part of our analytical package, none of our customers using MDEA/DEA blends require this service. What is important to monitor are things such as CO₂ loadings, solvent strength and heat stable salt levels. Most plants are adequately equipped to determine acid gas loadings, alkalinity and heat stable salt level. However, monitoring of MDEA/DEA levels, degradation products and individual heat stable salt levels (i.e., formates, acetates and chlorides) requires equipment and personnel that are not normally available at most plants.

We have reviewed data from 98 operating plants that use DEA as their solvent. Of the 98 plants, only six plants had total DEA degradation products greater than 2 wt% (Table 7). All 92 plants that had low degradation had a DEA concentration of about 20 wt%. Only when the DEA concentration reached about 30 wt% did DEA degradation products become much more significant.

It appears that DEA degradation increases when a higher concentration of DEA is coupled with the higher CO₂ loadings for DEA (~0.01 M/M) compared to MDEA/DEA blends (~0.005 to 0.01 M/M). H₂S levels do not appear to lower DEA degradation when DEA concentration is at the 30 wt% level as is seen by the one plant that had significant DEA degradation even with 171 psia H₂S partial pressure.

RECOMMENDATIONS

Based on a review of the literature, our own research, and a review of operating and analytical history of plants using MDEA/DEA blends, the following conclusions can be drawn:

- DEA, and DEA in MDEA/DEA blends, degrades in the presence of CO₂ to HEOD, THEED and BHEP.
- Degradation is a function of temperature, CO₂ partial pressure and DEA strength.
- The presence of H₂S does not appear to affect DEA degradation rates.
- Degradation of actual plant solutions comprised of MDEA/DEA blends by CO₂ is not as severe as the literature and our own lab research indicates. Laboratory studies tend to use high CO₂ partial pressures and temperatures to accelerate degradation and corrosion. While plants do operate at high partial pressures of CO₂ in the absorber (60 to 100 psia is not uncommon), the absorber temperature rarely exceeds about 150°F. In the high-temperature areas of the plant (i.e., bottom of the stripper where temperatures of 250°F are common), the CO₂ partial pressure is generally on the order of only 1 to 3 psia of CO₂.
- MDEA/DEA blends can be used successfully with low degradation and corrosivity if proper CO₂ loadings, solvent strength and heat stable salt guidelines are maintained.

- The presence of metals such as Fe, Cr and Ni in an operating amine solution does not necessarily indicate the solution is corrosive.

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Tom Bacon is currently an engineering consultant in the gas processing industry with emphasis on gas treating, project engineering and technical training. During his 33-year career with The Dow Chemical Co., which he left in November 1998, he served in a number of engineering and management roles including process research, plant operations, management of process technology, design, construction and startup of newly developed technologies, and development of and technical service for gas treating processes for the natural gas, geothermal power, oil refining and coal fired power industries. Earlier experience includes three years with Texaco in gas production and processing. Mr. Bacon received his BS degree in chemical engineering from Texas A&M in 1961.

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