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Corrosion in alcanolamines for natural gas treatment – High temperature experiments in lean and rich conditions

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Summary

In the oil and gas industry, acid gas removal is required for the treatment of natural gas. New applications were also recently pushed forward for CO_2 capture from the flue gas of power plants. For both applications, the most widespread industrial processes use absorption – desorption by an alcanolamine solvent. In such amine units for acid gas removal, corrosion represents an important operational concern. Influent parameters governing corrosion are temperature and acid gas loading, amine nature, and the presence of degradation by-products.

Some of these parameters were studied with corrosion experiments in autoclave. Test temperature was between 110°C and 120°C, i.e. within the inlet and outlet temperatures of the regenerator where the risks of corrosion are the highest. CO_2 loadings of rich solvent representing absorber outlet conditions (α close to 0.5 mol of CO_2 per mol of amine) and of lean solvent similar to regenerator outlet conditions (α close to zero) were tested. Different blends of amine solvents were used.

Corrosion rates of carbon steel and of some stainless steel grades were examined through weight loss experiments and surface analysis. Concentrations of dissolved metallic species were also analysed.
The more severe corrosion risks were found in the pure DEA solutions. In rich conditions, AISI 304L could suffer depassivation and uniform corrosion. This corrosion was increased in an industrial DEA solution, containing higher levels of degradation products.
In the MDEA and MDEA / DEA solutions, the corrosion rates were considerably lowered for 304L and for C steel.

1 Introduction

1.1 Basics of acid gas removal by alkanolamines

Throughout the petroleum chain, the treatment steps are nearly always designed to purify the fluids, separate various components and eliminate undesirable species, in particular the two acid gases carbon dioxide (CO_2) and hydrogen sulfide (H_2S). Treatments to remove CO_2 and H_2S from natural gas are often implemented on the production site. At the other end of the chain, the refinery gases may also need to be stripped of these acid gases. Lastly, these separation treatments are becoming increasingly important in the fight against greenhouse gas emissions and might be used in a near future for the post-combustion capture of CO_2 in combustion fumes from industrial processes [3].

The most widely used process in all these applications uses alkanolamine-based chemical solvents (e.g. MEA, monoethanolamine, DEA, diethanolamine, MDEA, methyldiethanolamine) capable of reacting preferentially with CO_2 and H_2S. Figure 1 provides a simplified process flow description of this type of treatment unit. The main components and the operating principles are the same in the three broad domains concerned: natural gas processing, treatment of refinery gases and post-combustion CO_2 capture.

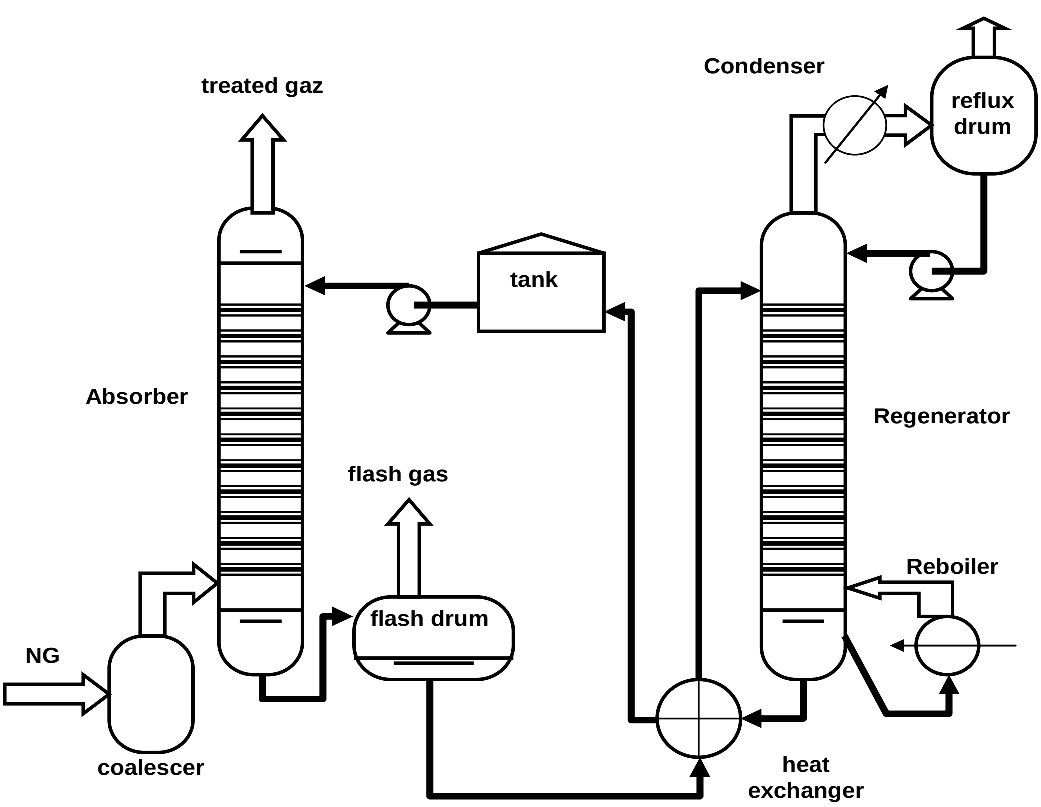

Figure 1: Simplified diagram of an alkanolamine acid gas removal unit

The gas to be treated is introduced in the unit at the bottom of the absorber where it comes into contact with the solvent. The acid gases (CO_2 and/or H_2S) react and are absorbed by the solvent according to reactions 1 to 6 below.

The reaction with H_2S, which is the same irrespective of the type of amine considered, consists of direct proton transfer (Reaction 1), where R_1, R_2 and R_3 represent alkyl groups or a hydrogen atom.

$$R_1R_2R_3N + H_2S \Leftrightarrow R_1R_2R_3NH^+ + HS^- \qquad \text{(Reaction 1)}$$

The reaction with CO_2 is slightly more complex. Two different mechanisms may be involved. A first reaction may occur, involving the successive formation of carbonic acid and bicarbonate (Reaction 2 and Reaction 3), followed by direct proton transfer identical to that observed for H_2S (Reaction 4).

$$CO_2 + H_2O \Leftrightarrow H_2CO_3 \qquad \text{(Reaction 2)}$$

$$H_2CO_3 \Leftrightarrow HCO_3^- + H^+ \qquad \text{(Reaction 3)}$$

$$H^+ + R_1R_2R_3N \Leftrightarrow R_1R_2R_3NH^+ \qquad \text{(Reaction 4)}$$

This mechanism may occur for all types of amine. The global kinetics is slow, however, since it is limited by dissociation of carbonic acid into bicarbonate (Reaction 3).

For the primary and secondary amines, which have hydrogen bond to the nitrogen, a second mechanism is possible.

$$CO_2 + R_1R_2NH \Leftrightarrow R_1R_2N^+ + HCOO^- \qquad \text{(Reaction 5)}$$

$$R_1R_2N^+ + HCOO^- + R_1R_2NH \Leftrightarrow R_1R_2NCOO^- + R_1R_2NH_2^+ \qquad \text{(Reaction 6)}$$

This second reaction mechanism is generally much faster than the CO_2 hydrolysis reaction (Reactions 2 to 4). Reaction 5 and 6 forms the well-known amine carbamate. For the primary and secondary amines, there is little difference in the reaction kinetics between CO_2 and H_2S. For the tertiary amines, however, unable to form the carbamate, the reaction kinetics with H_2S is much faster than with CO_2. This property is put to good advantage to give some solvents selectivity between CO_2 and H_2S.

At the liquid output at the bottom of the absorber, the solvent is therefore enriched in acid gas according to one or more of reactions 1 to 6: loaded solvent is rich amine. At the top of the absorber, the gas has been stripped of its undesirable components CO_2 and/or H_2S.

The rich amine is then pre-heated by a heat exchanger then fed into the top of a regeneration column. In column, the solvent is heated to high temperature by the reboiler, up to about 130℃. The heat transferred to the solvent at high temperature releases the dissolved acid gases. This is due to the fact that the equilibria of reactions 1 to 6 are shifted to the left at high temperature and low pressure. At the liquid output of the regenerator, the solvent is hot (about 130℃) and contains less acid gas: the off-loaded solvent is lean amine. The solvent is then cooled by the feed/bottom heat exchanger, trim cooled and sent back to the top of the absorber to start a new cycle. The acid gases are collected at the top of the regenerator, they are cooled down for their suitable processing.

1.2 Corrosion mechanisms and influent parameters

In such complex units, numerous pieces of equipment are exposed to equally numerous types of corrosion. We can nevertheless adopt the classification proposed by Nielsen [4], who identifies:

- wet acid gas corrosion,
- amine solution corrosion.

Wet acid gas corrosion may be encountered in all parts of the unit in contact with an aqueous phase with a high concentration of dissolved acid gases CO_2, H_2S, as well as NH_3 and HCN for refinery units. This type of corrosion is found primarily in zones where the gaseous phases have high concentrations of acid gases and where water may condense, mainly at the bottom of the absorber and the top of the regenerator.
For gas containing mostly CO_2, parts of the installation made from carbon steel may suffer fast CO_2 corrosion, up to several mm/year. In the presence of H_2S, this uniform corrosion is generally lowered by the formation of a protective iron sulfide layer. A minimum H_2S/CO_2 ratio of 1/20 is often considered as sufficient to avoid risks of CO_2 corrosion [5,6]. In the presence of H_2S however, specific cracking phenomena may also be encountered (hydrogen embrittlement, HIC, SSC, etc.). In the presence of HCN and/or NH_3, the risks of cracking are also increased.

The second type of corrosive media found in acid gas removal units is the amine solution itself. Generally, amines are not intrinsically corrosive, since they associate both high pH and low conductivity. They may nevertheless become corrosive when they absorb CO_2 or H_2S or when exposed to degradation. Furthermore, since the treatment units operate in semi-closed circuit, the solvent may become enriched with possibly corrosive degradation products.
No consensus has yet been reached concerning the mechanisms of corrosion by amine solutions. The models proposed vary depending on the type of amine (in particular, primary, secondary and tertiary), the H_2S/CO_2 ratio in the gas to be treated, the possible presence of oxygen either as contaminant in the circuit or as component of the input gas (e.g. CO_2 capture in fumes). For more information on specific corrosion models, the reader may refer to the relatively extensive bibliography on this subject [4,7-10].
We may nevertheless identify some systematic trends governing the corrosiveness of acid gas chemical solvents. Acid gas loading and temperature are usually considered as the most important factors. The acid gas loading (α) is defined as the quantity of acid gas absorbed by a defined quantity of solvent and is often expressed in moles of acid gas per mole of amine. Increasing the acid gas loading increases the corrosiveness of amine solutions [9-12]. Temperature generally has an extremely important effect on corrosion phenomena since most electrochemical reactions involved are thermally activated. It is common practice in industry to consider that the corrosion rate is doubled when the operating temperature increases by 10℃ to 20℃.
For gas treatment units, the effect of temperature is relatively difficult to asses on an

individual basis. Temperatures vary widely in the installation, with extreme values ranging from 40℃ in the absorber up to 130℃ in th e reboiler. However, these temperature variations have a significant effect on the chemistry of the solution, in particular the acid gas loading. Taking into account both the loading and the temperature, we may consider that the main corrosion risks are encountered in areas with high loading and high temperatures [10,13]. These conditions are generally found in the rich amine line after the heat exchanger and up to the regenerator input.

The type of amine is also one important factor. Usually, primary amines (e.g. MEA) are the most corrosive, secondary amines (e.g. DEA) slightly less and tertiary amines (e.g. MDEA) exhibit the lowest risks of corrosion [9,10,12,14-17]. Amine concentration also has an influence on corrosion. Excessively high amine concentrations should generally be avoided with C steel facilities. Nevertheless, the results obtained from the few laboratory studies conducted on the effect of amine concentration on corrosiveness vary widely, between a marked effect [10,12] and a moderate or null effect [18,19].

The concentration in degradation products and contaminants can significantly influence corrosion reactions. A distinction must be made between basic and acidic degradation products. Basic amine degradation products mainly result from chain reactions between amine and CO_2, for example the following compounds: HEOD (3-(2-hydroxyethyl)-2-oxazolidone), BHEP (N,N'-bis(2-hydroxyethyl)piperazine), THEED (N,N,N'-tris(2-hydroxyethyl)ethylenediamine). The studies on corrosiveness by these degradation products date back a number of years, the general conclusion being an absence of specific corrosiveness [15,16]. Most acidic degradation products result from reactions with oxygen. The main products include salts of oxalic, glycolic, formic and acetic acids, which are stronger than carbonic acid. As a result these salts are not thermally regenerated in the process, hence their name: Heat Stable Salts (HSS). The effect of these products on corrosion has been well documented through laboratory tests; they increase corrosion of carbon steel [20-22].

Finally, the solvent flow rate and conditions favourable to turbulence (gas flash, gas injection zones, etc.) may cause risks of erosion-corrosion. This type of corrosion is specific to carbon steels, since stainless steel grades are far more resistant. This type of corrosion is probably aggravated when the content of degradation products becomes too high: as indicated above, some of these products have a chelating effect on iron and may favour more efficient and faster dissolution of the protective deposits exposed to erosion [4,5,11,23].

2 Experimental study

2.1 Objectives

This experimental work follows some return of experience in sweet DEA treatment plants, where corrosion of some stainless steel grades had been observed [13,24].

Thus, the objective of the study is to compare the corrosiveness of different amines, and for different steel grades. Laboratory experiments were performed in hot rich and lean solutions of secondary and tertiary amines. Degraded DEA solution sampled in a treatment plant was also available, and used for tests in the same conditions.

2.2 Experimental methods

The first series of experiments was carried out in an industrial DEA solution (4.5 M), sampled in a sweet gas treatment plant. A complete analysis of this solution was performed before it was used in the laboratory for corrosion tests. The results are presented in Table 1. Even though this DEA solution had a dark brown colour, indicating a strong degradation, the concentration in degradation products remained just below usual limits given in the literature (e.g. ref. [25]), even though the total HSS content was close to maximum acceptable values (e.g. ref. [24]), .

Table 1: Analysis of the industrial DEA solution used for corrosion tests.

Family	product	concentration
Amine	DEA	477 g/L
acid gas	CO2	8 mg/L
anions	chloride	4 mg/L
	thiosulfate	< 10 mg/L
	sulfate	42 mg/L
cations	iron	667 mg/L
	chromium	65 mg/L
	nickel	7 mg/L
	molybdenum	13 mg/L
	sodium	15 mg/L
	manganese	1 mg/L

Family	product	concentration
HSS	formiates	43 mg/L
	acetates	496 mg/L
	propionates	22 mg/L
	oxalates	< 10 mg/L
	glycolates	< 10 mg/L
basic degradation products	MEA	0.02 wt. %
	MAE	0.002 wt. %
	BAE, BEA, DEP	< 0.01 wt. %
	HEED	< 0.2 wt. %
	HEP	< 0.01 wt. %
	others	1.1 wt. %

Other test solutions were prepared with analytical grade DEA and MDEA and deionised water.

For each test solution, two corrosion tests were carried out successively, using the same batch of solution. The first experiment was carried out in a rich solvent ($\alpha = 0.45$) at 110℃. The second experiment was carried out in a lean solvent ($\alpha < 0.1$) at 120℃. This lean solvent was obtained by N_2 purging during at least 24 hours. Therefore, the lean solutions already contained dissolved metals and some degradation products.

In order to obtain rich solutions at the desired CO_2 loading, saturated solutions were first prepared by purging CO_2 for approximately 12 hours at ambient temperature. This saturated solution was then mixed with a lean solution. CO_2 titration was checked after mixing, to ensure that the target loading was reached. The method used to determine the quantity of absorbed CO_2 consisted in a volumetric measurement after adding an excess amount of hydrochloric acid to a sample of the solution [26]. Acid gas loading was then expressed as the ratio between the number of moles of CO_2 and the number of moles of amine.

All experiments were carried out in a 1L autoclave made of Hastelloy C276, and were performed for four weeks.

The autoclave was first filled with the test solution at the set CO_2 loading. It was then closed and heated to the desired temperature. During this heating, a new equilibrium between the gaseous and dissolved CO_2 was established. Nevertheless, the gaseous volume in the autoclave was sufficiently low to consider that the loading remained unchanged. Furthermore, after all corrosion tests CO_2 titration was systematically performed to ensure that the loading did not significantly evolve during the experiment.

Corrosion coupons were made of AISI 1020, AISI 304L and AISI 316L. Those grades are representative of C steel and of austenitic stainless steel used in gas treatment units. The chemical composition is given in Table 2. The specimens were cut from thin plates into 26 mm x 26 mm squares. Before each experiment, all specimens were ground with 600 grit SiC paper, degreased with ethanol and rinsed with deionised water. The specimens were mounted to the rotating axle with polymer spacers to prevent galvanic coupling effects. Stir speed was maintained constant, and the peripheral speed of the specimen was close to 0.4 m/s (< 1.3 feet / s) .
Before final weighing, each specimen was rinsed with distilled water and dried. When needed, corrosion scales were removed using a plastic brush and/or by a chemical cleaning method, as proposed in ASTM G1 standard [27]. Analysis of the test solutions were performed in order to determine the concentration of dissolved metals.

Table 2: Chemical composition (wt. %) of steels used for the corrosion tests.

	Fe	Cr	Ni	Mo	C	Mn	Si	S
AISI 1020	bal.	0.01	0.01	<0.005	0.17	0.75	0.01	0.003
AISI 304L	bal.	19.14	9.27	022	0.02	1.62	0.04	0.001
AISI 316L	bal.	16.86	10.30	2.18	0.02	1.34	0.45	0.002

2.3 Results

Corrosion rates and dissolved metals measured after the experiments are gathered in Table 3 to Table 6.

Table 3: Corrosion rates (µm/year) for different steel grades in rich (α = 0,45 at 110℃) solutions of different amines.

	316L	**304L**	**1020**	**Observation**
Industrial DEA 45%, rich	65	130	160	$FeCO_3$ scale
DEA 40%, rich	15	80	150	$FeCO_3$ scale
MDEA 40% + DEA 5%, rich	< 5	10	30	
MDEA 40%, rich	< 5	< 5	20	

Table 4: Corrosion rates (µm/year) for different steel grades in lean (α < 0,1 at 120℃) solutions of different amines.

	316L	**304L**	**1020**	**Observation**
Industrial DEA 45%, lean	< 5	< 5	485	
DEA 40%, lean	< 5	< 5	1790	
MDEA 40% + DEA 5%, lean	< 5	< 5	25	
MDEA 40%, lean	< 5	< 5	11	

Table 5: Solubility of different ions (mg/L) measured after four weeks of corrosion testing in rich (α = 0,45 at 110℃) solutions of different amines.

	Fe	**Cr**	**Ni**
Industrial DEA 45%, rich	244	175	95
DEA 40%, rich	175	100	82
MDEA 40% + DEA 5%, rich	20	30	20
MDEA 40%, rich	15	50	30

Table 6: Solubility of different ions (mg/L) measured after four weeks of corrosion testing in lean (α < 0,1 at 120℃) solutions of different amines.

	Fe	**Cr**	**Ni**
Industrial DEA 45%, lean	1800	82	50
DEA 40%, lean	830	94	80
MDEA 40% + DEA 5%, lean	75	50	36
MDEA 40%, lean	49	62	49

Obviously, active corrosion of AISI 304L can be encountered in rich DEA solutions. The effect is more pronounced in the industrial DEA solution, indicating some probable effect of degradation products. For this solution, some uniform corrosion of 316L is observed, even if the corrosion rate remains moderate (65 µm/year). This tendency is confirmed by the high level of soluble metals in the rich DEA solutions after the experiments. According to work done by other researchers [15], this indicates that the solution contains chelating agents, capable of complexing chromium and nickel constituting the protective passive film of AISI 304L. This complexing power is increased in the industrial DEA solution compared to the laboratory DEA solution, which indicates that degradation products should play an important role.

Carbon steel corrosion follows a slightly different trend in the rich DEA solutions. Even if the content of soluble iron is also quite high, the corrosion rate remains moderate, in the same range as that of AISI 304L. However, a thick and dense iron carbonate layer was observed at the surface of the carbon steel specimens after the experiments (Figure 2). This layer might have had some protective action.

Corrosion in rich MDEA and in rich MDEA / DEA is radically different than in rich DEA. In the former solutions, corrosion rates of both stainless steel and carbon steel remained extremely low. Nevertheless, surprisingly high levels of soluble chromium and nickel were measured, in a comparable range than in DEA solution. Some chelating compounds should then also be expected in MDEA and in MDEA + DEA blend. This also indicates that metal solubility might not always be a good indicator of solution corrosiveness, as already mentioned by Rooney and co-workers [28].

In lean DEA solutions, both stainless steel grades exhibited perfect corrosion resistance. During these experiments, soluble chromium and nickel content also decreased (the initial concentration corresponding to the concentration measured at the end of the experiment in rich condition), indicating some precipitation. It is therefore probable that the complexing tendency is not only related to degradation products, but also to some carbonic species. On the other hand, in MDEA and MDEA / DEA solutions, soluble chromium and nickel levels remained constant or increased slightly. The difference might then be associated with amine carbamate, which forms only by reaction between CO_2 and primary and secondary amine, but not with tertiary amine like MDEA.

The most marked difference between experiments in lean solvents was observed for the carbon steel coupons. In DEA solutions, the corrosion rate of carbon steel appeared more severe than in rich solution. In that case, no protective scale was observed at the steel surface. In the lean solvent, the lower concentration in carbonic species induces a higher solubility of iron, thus less scaling tendency. This assumption is confirmed by the chemical analysis, revealing extremely high levels of soluble iron. Unexpectedly, we also observed faster corrosion of carbon steel in the laboratory lean DEA solution than in the industrial solution. This result might be the consequence of a poor regeneration of the test solutions before the experiment. Indeed, the procedure only ensured that the lean solvent contained less than 0.1 mol of CO_2 / mol of amine. At such levels, a sufficiently high residual oxidizing power might be found. This result also shows that the higher HSS level in the industrial solution is not sufficient to generate a higher corrosiveness.

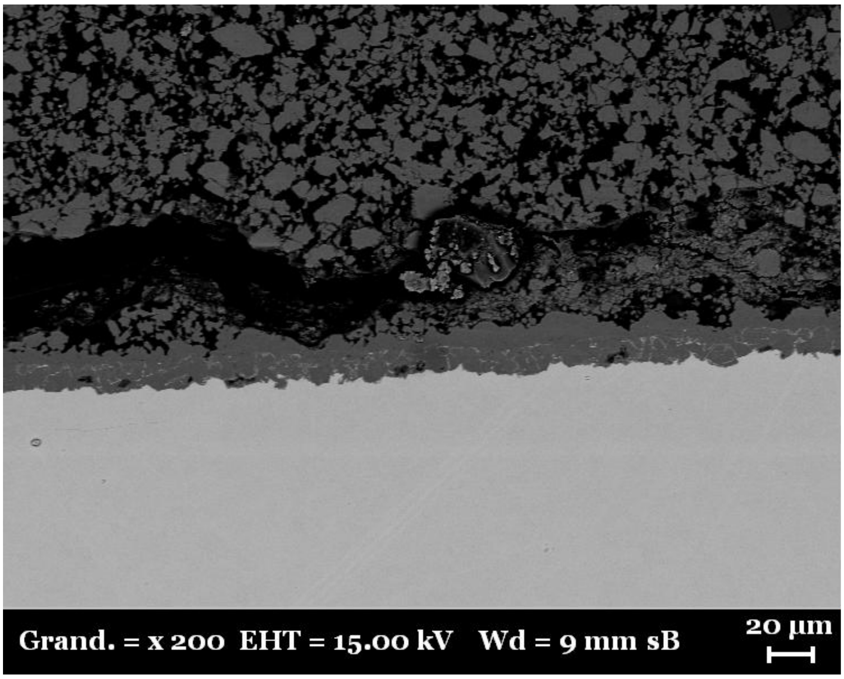

Figure 2: iron carbonate layer formed at the surface of AISI 1020 coupon after 4 weeks exposure in rich DEA at 110℃.

3 Conclusions

These experiments confirm that DEA solutions used for acid gas treatment present severe risks of corrosion.

In hot rich conditions, austenitic stainless steel grade 304L might suffer depassivation and severe uniform corrosion. The mechanism probably involves some chelating

agents, capable of complexing nickel and chromium. These specific risks are increased with acid gas loading and with concentration in degradation products.
Carbon steel also suffers corrosion in DEA solutions. In our experimental conditions, corrosion was more severe in lean than in rich solvent. In the latter case, precipitation of iron carbonate was observed at the surface of the specimens, and provided some protection. This phenomenon is however highly dependent on the hydrodynamics, and it would be extremely hazardous to rely on it in a gas treatment plant. In lean DEA solutions, the concentration in carbonate species was too low to allow for iron carbonate scale formation, and severe uniform corrosion was observed.

MDEA and MDEA / DEA solutions showed much less risks of corrosion for stainless steel grades and also for carbon steel. Contrary to tertiary amine, DEA has the faculty to form amine carbamate by reaction with CO_2. This molecule has probably some role in the increased corrosiveness and solubility of metals. However, the levels of dissolved metals after corrosion tests in MDEA and MDEA / DEA remained high as compared with the observed corrosion rates. In that sense, metals solubility should not be regarded as a universal indicator of the solution corrosiveness.

The last important result of this work concerns the variability of metals solubility between rich and lean solutions. In a closed loop system of the process, one could then expect to dissolve iron in the lean sections, and precipitate iron carbonate in the rich sections. This also highlights the importance to select with care the sampling points, when an analytic survey of the solvent composition is made.

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