Corrosion in post-combustion CO2 capture plants - comparisons between MEA 30% and new processes

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CO_2 capture and storage (CCS) is one of the main option for CO_2 mitigation. Post-combustion capture processes using alkanolamines are considered as one of the preferred options for CCS, since their industrial viability was already demonstrated. Absorption – desorption by 30% monoethanolamine (MEA) represents the reference technology for post-combustion CO_2 capture. However, the cost of CO_2 removal with this MEA 30% process remains very large, and needs to be reduced.

In CCS field, a lot of research program aim at developing new solvents, with a particular focus on the energy consumption needed at regeneration step. Minimizing solvent degradation is also often considered as the second immediate priority. Solvent degradation is usually caused by chemical reaction with oxygen, which is always present in post-combustion fumes. Other impurities, such as NO_2 or SO_2, may also react with the solvent and form undesirable by-products.

This paper is divided in two sections. In the first part, we propose a review of CO_2 capture with monoethanolamine, which represents the reference situation. Focus is given to corrosion aspects, including laboratory results and pilot plant studies.

In the second part of the paper, we present two ways of improving CO_2 capture processes that are currently studied at IFPEN. The first one is an evolution of the reference 30% MEA process, using a higher MEA concentration in order to reduce the energy consumption. The second one uses a newly developed solvent, with improved performances. For both cases, specific corrosions tests will be presented, both in laboratory conditions and in pilot plant operations.

Introduction – post-combustion CO_2 capture with alkanolamines

Post-combustion CO_2 capture with 30 % mass monoethanolamine represents the most widely accepted reference process. It is based on the high chemical affinity of the alkaline amine with CO_2, following the global reaction:

$$2HOC_2H_4NH_2 + CO_2 \rightleftarrows HOC_2H_4NHCO_2^- + HOC_2H_4NH_3^+ \qquad \text{(Reaction 1)}$$
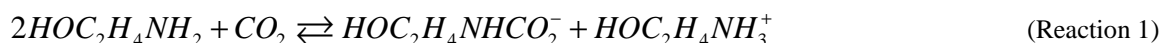
The main reaction products are the amine carbamate and the protonated amine.
This chemical equilibrium is greatly influenced by temperature. It is shifted to the left at high temperature and high pressure. This property is put into advantage in the industrial process, which consists of successive absorption – desorption in a loop system.
Figure *1* provides a simplified process flow description of this type of treatment unit.
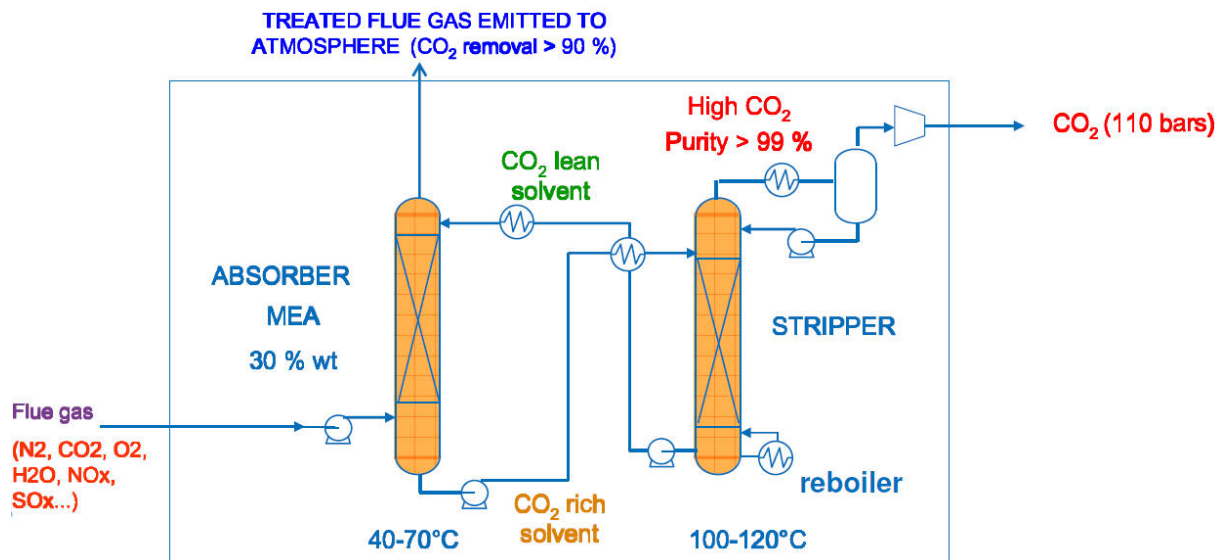
Figure 1: Simplified diagram of a MEA CO_2 capture unit

The flue gas which contains high concentration of CO_2 enters in the treatment plant at the bottom of the absorber. Lean MEA is introduced at the top of the column, and chemical reaction between the amine and CO_2 takes place.

At the liquid output at the bottom of the absorber, the solvent is enriched in acid gas: we speak of rich amine. At the top of the absorber, the gas has been stripped of its CO_2.

The rich amine is then pre-heated by a heat exchanger then fed into the top of a regeneration column. In this part of the unit, the solvent is raised to high temperature by steam, which releases the dissolved CO_2. At the liquid output of the regenerator, the solvent is hot and contains less acid gas: we speak of lean amine. The solvent is then cooled by the heat exchanger and sent back to the top of the absorber to start a new cycle. Pure CO_2 is collected at the top of the regenerator. Before sequestration, it has to be compressed to 100 bar for transportation. This compression step also represents an important penalty in terms of energy consumption.

Section 1 – Reference process with 30% MEA

Performance indicators for the 30% MEA process

30% MEA represents the reference case for CO_2 capture process. In most recent pilot plant studies, this solvent was tested for comparison with newly developed solutions. An excellent review of research programs ongoing worldwide on CO_2 absorption was proposed last year by Wang et al. [1].

A complete description of the performances of CO_2 capture process with MEA far exceeds the goals of this paper. However, a few key features must be given, in order to explain the research roads described in the second section of the paper.

The first major parameters for efficient CO_2 post-combustion capture are chemical reactivity and reaction kinetic between the solvent and CO_2. Indeed, Post-combustion fumes are

produced at a high flow rate and with a low CO_2 partial pressure. The solvent needs to be extremely reactive to allow a high level of CO_2 transfer from the gas phase to the liquid phase. High reaction kinetics is also an important feature: the lower the kinetics, the larger the contact time between the gas and liquid phase to achieve the target CO_2 removal. Thus, decreasing the solvent kinetics can only be mitigated by increasing the height of the absorber tower, or by using more efficient contactors, which a huge impact on capital costs. Regarding this specific aspect, MEA represents an excellent solvent. CO_2 loading, α, in the solvent at equilibrium with 100 mbar CO_2 is close to 0.5 mol_{CO2}/mol_{MEA}. The reaction kinetics is also extremely fast, allowing reasonable dimensions of industrial equipments for the absorption.

The second performance indicator for solvent ranking is the regeneration energy, i.e. the energy needed to release the absorbed CO_2. This energy is usually provided by steam generated in the reboiler, and constitutes a direct penalty for the power plant performance. MEA 30% is known to be very demanding in terms of energy consumption. Indeed, about 3.7 GJ/t_{CO2} are required for solvent regeneration [2,3], representing the most important part of the operating costs.

The last two important parameters regarding industrial feasibility of chemical absorption is the solvent stability and corrosivity to construction materials.
MEA is known to react quite rapidly with O_2 contained in the flue gas, with two major consequences: i/ degraded solvent becomes less reactive, and needs to be periodically cleaned and replaced; ii/ some degradation products are corrosive to steel. Pilot plant studies conducted during the EU funded Castor project have shown that the degradation of the 30% MEA process corresponded to 1.4 kg MEA degraded / t_{CO2} captured [4]. At the industrial scale, this degradation corresponds to the consumption of approximately two batches of solvent per year, representing a cost of 7 M€/year for a CO_2 capture plant in a 600 MWe power plant.
Corrosion is also a major drawback of most gas treating units using amines. Corrosion can be caused either by the carboxylic acids (i.e. acetic acid, formic acid and oxalic acid formed by oxidative degradation), or by the amine itself.
A brief summary of actual knowledge of corrosion reactions in 30% MEA is given below.

Focus on corrosion

Several papers presenting laboratory corrosion measurements in MEA solutions have been published in the last years [5-9]. It appears that carbon steel corrosion rate is mainly influenced by temperature and CO_2 loading in the solvent, as illustrated in Figure 2.

Experiments at various MEA concentrations between 20 and 40% showed that the increase of amine concentration had a detrimental impact on corrosion rate, but in a much lower magnitude than temperature and CO_2 loading [6,8].
The impact of heat stable salts on MEA corrosivity was also reported [5,10]. Among the most common heat-stable salts, oxalic acid seems to have the largest impact on corrosion. However, the mechanism is more related to decreasing the protectivity of corrosion scale by a chelating effect, rather than to an electrochemical activity.

From all laboratory studies, it appears that carbon steel grades cannot be used safely in all operating conditions encountered in a MEA capture unit, with predicted corrosion rates

exceeding 100 µm/year [6]. Thus, many studies are dedicated to corrosion inhibitors [11,12]. Unfortunately, most efficient inhibitors, such as salts of heavy metals, often adversely affect amine degradation. It also appears from most laboratory studies that austenitic stainless steel grade 316L seems to perform well [6,10].
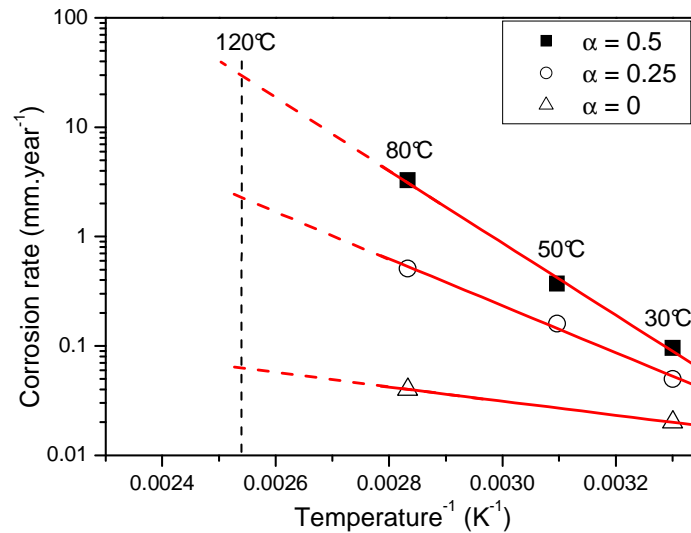
Figure 2: Impact of temperature and CO_2 loading on the corrosion rate of carbon steel in 30% MEA [6]

All these corrosion results have been compared, at various levels, with field data from pilot plants.

Excellent correlation between laboratory evaluation and pilot plant data were found for carbon steel. Experiments in the Castor pilot plant operated by Dong Energy in Denmark or in the pilot unit of the International Test Centre for CO_2 Capture (ITC) in Canada showed corrosion rates exceeding several hundreds of µm/year in the hot rich amine sections [13].

Good performance of stainless steel grade AISI 316L was also demonstrated by specific corrosion experiments in the Castor pilot plant [13], as well as in the RWE pilot plant in Niederaussem, Germany [14].

Discussion – roadmap for lowering the cost of 30% MEA process

Considering MEA 30% as the benchmark, new generation processes for post-combustion CO_2 capture must provide significant improvements for the key performances.

With that aim, CO_2 capture cost reduction is the main driving force for all R&D activities in the domain. Two main types of costs must be considered with equal importance.

Operating costs (OPEX) include power needs of ancillary equipments (pumps, blowers, compressors,...), reboiler heat for solvent regeneration, solvent reclaiming for the removal of degradation products, and solvent make-up to replace degraded amine. Recent multi-scale simulations of the costs of CO_2 capture showed that the OPEX represents approximately from half to 2/3 of the total cost of avoided CO_2 depending if one considers an integrated plant or not [15,16]. According to the latter studies, the energy consumption directly used for the regeneration of 30% MEA, estimated at 3.7 GJ/t_{CO2}, is by far the first item in terms of cost

since it represents from 30 to 60% of the total CO_2 capture cost. This explains why a lot of research programs tend to develop new solvents requiring less energy consumption for the regeneration.

On the other hand, capital costs must not be forgotten. CAPEX is mainly influenced by the size of the equipments, and more precisely the absorber and the regenerator. Thus, decreasing the reaction kinetics has a direct impact on column height and cost. Material selection is also an important parameter defining the final cost.

In the next section of this paper, two different alternatives currently investigated at IFPEN are described.

The first one consists in an optimization of the MEA process, by increasing the solvent concentration, resulting in improvements of energy needs.

The second one is a more radical evolution, and uses a new generation demixing solvent, allowing a significant reduction of regeneration energy, as well as a high temperature regeneration at high pressure.

Section 2 – Research in progress for high performance CO_2 capture

Optimization of energy consumption with MEA – Hicapt$^{+(TM)}$ process

As discussed in the previous part of this paper, CO_2 post-combustion capture with MEA presents many drawbacks. The two more important are the energy consumption for the regeneration and the oxidative degradation. Corrosion is also an important operational problem, but there exists efficient metallurgical solutions.

In spite of these difficulties, MEA remains one of the most widely investigated solvent for CO2 post-combustion capture. Indeed, it is cheap, easily available, non toxic and highly effective due to its high capacity and fast kinetics. Several large scale projects, e.g. Castor pilot plant or the Enel pilot plant in Brindisi [17] have demonstrated the good operability, flexibility, stability and reliability of this process during long test runs.

Based on all the knowledge acquired during pilot tests with 30% MEA, it was found that increasing the amine concentration to approximately 40% could result in significant reduction of energy demand for the regeneration. This feature was verified in the pilot plant operated by ENEL in the Brindisi coal fired power plant [17]. Two successive test runs were conducted with 30% and 40% MEA, resulting respectively in a regeneration energy of 3.4 GJ/t_{CO2} and 3.0 GJ/t_{CO2}.

Unfortunately, it is known also that increasing MEA concentration usually results in a strong aggravation of the degradation. This was verified in laboratory experiments, showing that MEA consumption was doubled when the concentration was raised from 30% to 40% At this level, operation of the process would not be economically viable: it would require extremely frequent reclaiming of the solvent, and MEA consumption would represent up to four batches per year, which represents an important cost even for the cheap MEA. At the same time, the associated cost of degraded solvent disposal could become a problem.

An important part of our research on MEA based process was therefore dedicated to minimizing the oxidative degradation [18-21]. More than 200 candidate oxidation inhibitors belonging to more than fifteen different chemical families were tested. Some molecules with

excellent properties were found, as illustrated in Figure 3. These oxidation tests were performed at 80°C under continuous air flow. Under these conditions and without inhibitor, the degradation of MEA proceeds extremely fast, forming NH_3 as typical ultimate product. This gaseous component can easily be monitored in the gas stream by FTIR analysis, giving a good indication of degradation process. It is quite obvious from these results that several chemical species present excellent oxidation inhibition performances.
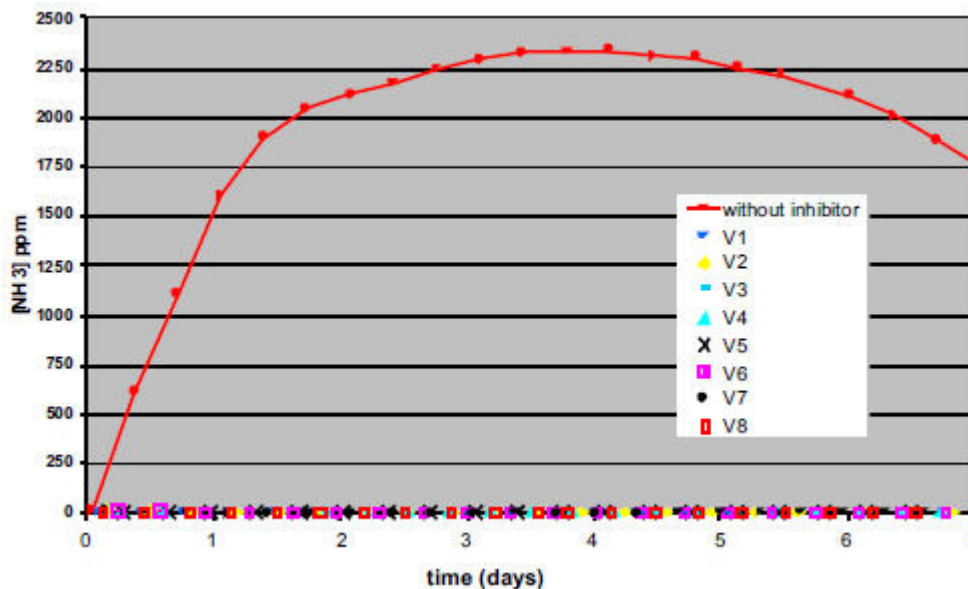

Figure 3: Effect of oxidation inhibitors on NH_3 evolution from MEA 40% [19]

The direct results of this research is the Hicapt+[TM] process developed by IFPEN and PROSERNAT. It uses a 40 % MEA solvent with an efficient oxidation inhibitor, thus combining high energy efficiency and low degradation.

Unfortunately, the more efficient oxidation inhibitors interact with the usual stainless grades used in the conventional 30 % MEA processes These inhibitors have a strong reducing capability, which may affect the passive layer of 304L and 316L. Small pilot plant studies conducted at IFPEN have shown uniform corrosion rates close to 0,5 mm/year for 316L coupons in the hot rich solvent. For this reason, it was not possible to test the Hicapt[+(TM)] process at pilot scale to date, since most available pilot units are made of 304L and 316L.

In order to solve this corrosion problem associated with the oxidation inhibitor, two types of solutions were investigated and gave satisfactory results. Efficient corrosion inhibitors were found, enabling the maintain of stainless steel passivity in the presence of the oxidation inhibitor. However, the more efficient corrosion inhibitors were strong oxidants, and they appeared to interact with the degradation inhibitor efficiency. This solution was thus abandoned. Investigations were then dedicated to find corrosion resistant materials. Solutions with a weak economical impact were found, and should be presented in more details in the near future. Small scale pilot plant with appropriate materials are also currently under construction at IFPEN, in order to demonstrate the efficiency of the solution in real operating conditions.

New generation of solvent – DMX$^{(TM)}$ process

Although MEA represents the most easily accessible solvent for short term commercial applications, its global performances remain extremely penalizing with regard to the cost of avoided CO_2. New generation processes must be developed to significantly lower the overall costs, and allow the largest dissemination of post-combustion technologies. A lot of research is in progress worldwide, with the objective to find new amine or blend of amines with improved performances. The great number of daily publications on this field does not allow to propose an exhaustive list of references, but a recent review of some of the most important research programs worldwide was proposed recently by Wang et al. [1].

In order to focus our research on the most important parameters, an analysis of process and design impact on the cost of avoided CO_2 was realized [16].
Cost repartition between OPEX and CAPEX was first established for the reference case using 30 % MEA for a non integrated plant. The main findings are presented in Table 1.

Table 1: Cost repartition for the 30 % MEA process

	CAPEX (€/t$_{CO2}$)	OPEX (€/t$_{CO2}$)	TOTAL (€/t$_{CO2}$)
Stripper	4.7	44.5	49.2
Compressor	5.1	5.1	10.2
Absorber	4.7	/	4.7
Solvent	0.2	2.4	2.6
Washing tower	2.2	/	2.2
Heat exchanger	2.0	/	2.0
Pumps	1.0	0.4	1.4
Blower	0.1	1.0	1.1
Miscelaneous	0.7	0.3	1.0
TOTAL	**20.7 (28 %)**	**53.7 (72 %)**	**74.4**

Steam price: 21.7 €/t
Electricity price: 45 €/MWh

Based on this analysis, individual impact of key parameters for the development of new solvents were discussed. The main characteristics of the solvent can be divided in four main domains:
- the thermodynamic performances, defining the cyclic capacity, the heat of reaction and the stripping energy,
- the kinetic performances,
- the thermal and chemical degradation,
- the operational performances, including volatility, foaming, corrosion, viscosity, ...

Cross analysis with cost impact allowed to determine the most efficient targets for new solvents:
- good thermodynamic properties, with a particular focus on the cyclic capacity,
- low thermal and chemical degradation for possible regeneration under pressure,
- good kinetics.

With these objectives in mind, IFPEN performed intensive high throughput screening (HTS) for thermodynamic and degradation evaluations [18,22,23]. A very interesting class of solvents was discovered in this investigation, which are capable of two-phase separation when

they are heated and loaded with CO_2. This process in now developed by IFPEN and PROSERNAT under the name $DMX^{(TM)}$. In this class of solvents, the CO_2 captured concentrates in one of the two liquid phases, and only this portion has to be sent to the stripper. This allows substantial reduction of the energy requirement for the regeneration of the solvent. In a first step, fine tuning of demixing solvents properties had to be determined, as a function of CO_2 loading and temperature [24]. It was particularly important to make sure that two-phase separation occurred only at high temperature, i.e. could not appear in the absorber where it would strongly affect gas - liquid transfer. Process evaluation with the best candidates showed that the reboiler duty could be reduced down to 2.3 GJ/t_{CO2}, which is a strong gap with 30% MEA (3.7 GJ/t_{CO2}) or with 40% MEA (3.0 GJ/t_{CO2}).

Once the more promising DMX^{TM} amine was chosen, degradation and corrosion tests were performed. These tests are not easy to perform, since phase separation is expected in closed reactors at high temperature. Weight loss coupons were then placed at two distinct heights in the reactor, where thermodynamic calculations predict two phases.
In order to evaluate the solvent behavior in extreme conditions, corrosion tests were performed at 160°C and under a high pressure of CO_2 (27 and 55 bar). Under these conditions, the global CO_2 loading is respectively 0.3 and 0.7 mol_{CO2}/mol_{amine}. Most of the absorbed CO_2 is in the dense phase.
Corrosion rates determined by weight loss measurements after three weeks exposure are presented in Table 2. The chemical composition of the steel coupons is given in Table 3.

Table 2: Results of corrosion experiments in the DMX^{TM} solvent at high temperature

Test condition	Steel grade	Corrosion rate (µm/an)	
		light phase	dense phase (CO_2 rich)
160°C / 27 bar	AISI 1018	1 ± 2 µm/an	1 ± 2 µm/an
	AISI 316L	3± 2 µm/an	3 ± 2 µm/an
160°C 55 bar	AISI 1018	9± 2 µm/an	8 ± 2 µm/an
	AISI 316L	8 ± 2 µm/an	8 ± 2 µm/an

Table 3: Chemical composition of weight loss coupons (wt. %)

	C	Si	Mn	Ni	Cr	Mo	N	S	P	Al	Cu
AISI 316L	0.021	0.448	1.335	10.295	16.861	2.161	0.047	0.002	0.029		
AISI 1018	0.186	0.223	0.560	0.084	0.106	0.013		0.022	0.009	0.015	0.261

These corrosion rates are extremely promising for the development of the process using this solvent. For comparison, the corrosion rate of carbon steel in CO_2 rich 30 % MEA at 110°C exceeds 0.5 mm/year.
From similar experiments, it was shown also that this solvent exhibited far better resistance to thermal and oxidative degradation than MDEA, which is already considered to have an excellent degradation behavior [16,25].

Thanks to these excellent properties, high temperature regeneration of the solvent can be considered. As a consequence, gaseous CO_2 at the stripper outlet can be obtained at a pressure of several bar, minimizing the cost of compression to 110 bar required before CO_2 transport and storage.

The next steps for the development of this process is to verify that all promising performances seen in the laboratory and in small scale pilot units will be confirmed on large scale pilot experiments. This pilot plant demonstration represents one of the objectives of the EU funded Octavius project which started in March 2012, coordinated by IFPEN and with 16 other partners.

Conclusions

The present paper describes some of the research in progress at IFPEN on CO_2 post-combustion capture with chemical solvents. At present time, monoethanolamine represents the benchmark solvent for this application. The main performances of this process were resumed, showing that the cost of avoided CO_2 remained quite high. It was shown also that this solvent was quite corrosive to carbon steel, and required to use stainless steel as construction material.

Then, two different new generation processes were presented.
The first one consists mainly of an optimisation of the usual MEA process. The energy consumption is lowered by approximately 15%, thanks to an increase of MEA concentration from 30% to 40%. The consequences of this higher concentration are discussed in terms of degradation and corrosion. It is shown that the risks of oxidative degradation increase considerably, but can be significantly reduced by efficient oxidation inhibitors. Corrosion needs also to be taken into account. It was shown that oxidation inhibitor could interact with the passive layer of usual austenitic stainless steel grades, allowing fast uniform corrosion. Solutions with more resistant materials were found to avoid this problem.

The second process uses a radically different type of amine. Furthermore, it presents the very particular trend to separate in two liquid phases at high temperature and when it is loaded with CO_2. Then, only the CO_2 rich liquid phase needs to be heated in the regenerator for CO_2 stripping, which represents a net benefit for energy consumption. This new solvent also presents excellent thermal resistance up to 160 – 180°C, as well as resistance to oxidative degradation. High temperature regeneration allows to produce CO_2 at several bars, allowing substantial compression gains. Finally, corrosion by this new solvent appeared to be much less than with conventional MEA, or even with usual amines used in gas treatment (DEA or MDEA). Corrosion rates as low as 10 µm/year were observed for carbon steel even at the highest temperature expected for the process. Therefore, less costly construction materials should be employed, and the use of expensive proprietary corrosion inhibitors will be avoided.

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