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Designing large-scale CO_2 capture units with assessment of solvent degradation

Grégoire Léonard[a,b,*], Cyril Crosset[a], Marie-Noëlle Dumont[a], Dominique Toye[a],

[a]*Department of Applied Chemistry, University of Liège, Allée de la chimie B6a, Liège Sart Tilman, 4000, Belgium*
[b]*Lenfest Center for Sustainable Energy, Department of Earth and Environmental Engineering, Columbia University, 500 West 120th Street, New York, NY 10027, USA*

Abstract

Solvent degradation is one of the main operational drawbacks of post-combustion CO_2 capture with amine solvents. Although the different degradation mechanisms have been largely studied in recent years, it is still impossible to predict the solvent losses and the emissions of degradation products that may occur in a CO_2 capture plant depending on its size and on its operating conditions. In the present work, we experimentally study the degradation of MEA (monoethanolamine) under accelerated conditions implying high temperature, continuous gas feed and vigorous agitation. A special focus is set on the oxidative degradation of MEA, which is studied in the absence of CO_2. Based on the experimental results, we propose a kinetic model to describe both MEA oxidative and thermal degradation pathways. The degradation kinetics is then included into a global model of the CO_2 capture process, enabling solvent losses and emissions of degradation products to be predicted as a function of the process operating conditions. The predicted MEA loss is in the same order of magnitude as reported in degradation measurements from pilot plants, although lower by a factor 3. This kind of model assessing solvent degradation could and should be used for the design of large-scale CO_2 capture plants in order to simultaneously consider the energy consumption of the process and its environmental impact related to the emissions of degradation products and amine solvent. Further developments shall consider the effect of SO_x, NO_x and dissolved metals on MEA degradation.

Keywords: Post-combustion CO_2 capture; monoethanolamine degradation; Modelling of MEA consumption; Prediction of ammonia emission

* Corresponding author. Tel.: +1-212-851-0241; fax: +1-212-854-7081.
  E-mail address: gl2497@columbia.edu

1. Introduction

CO$_2$ capture and storage technologies represent one of the main solutions to rapidly reduce the anthropogenic emissions of carbon dioxide in response to the increasing environmental concerns and to the growing world energy demand. Among other capture methods, the CO$_2$ post-combustion capture with amine solvent treats the flue gas after the combustion so that already operating power plants can be retrofitted to rapidly reduce their emissions. Besides the high energy requirement of the process, the degradation of the amine solvent and its consequences represent the second main operational drawback of post-combustion CO$_2$ capture. First, the cost of solvent make-up necessary to compensate for solvent losses may already represent up to 22% of the process operative costs [1]. Then, the degradation of amine solvents leads to the formation of a large range of products that may modify the solvent properties and decrease the process efficiency. Finally, the emission of amine solvents and volatile degradation products like ammonia is a critical issue in CO$_2$ capture plants. Although emission reduction technologies exist (among others the (acid) water washing of the flue gas at the column outlet), the problem of volatile products emissions is still significant in large-scale operating plants [2]. As underlined by [3], there is no advantage at capturing CO$_2$ if this implies the emission of other products like ammonia.

So far, the process energy penalty and the degradation of amine solvents have been studied separately and previously published models of the CO$_2$ capture process did not consider solvent degradation at all. The only one significant model of the process that took solvent degradation into account has been proposed by [4]. This model is based on literature data for the degradation of 30 wt% monoethanolamine (MEA, the benchmark solvent for post-combustion CO$_2$ capture). MEA thermal degradation has been modeled using the kinetic model described by [5] while data for MEA oxidative and SO$_x$ degradation have been provided by [6]. Nitrosamine formation from NO$_x$ has been included in the model based on the data published by [7], although very few experimental points were available. However, this model did not lead to relevant results and it could not be validated to predict industrial scale degradation. Such a model is however essential for a proper process evaluation and design.

The objective of the present work is to develop a model of the CO$_2$ capture process that assesses solvent degradation based on own experimental work, so that the influence of process operating conditions on solvent degradation may be better understood. As a result, solvent degradation may be considered more accurately during the design of CO$_2$ capture units, which is especially relevant for large-scale plants. In previous studies, we first described appropriate experimental equipment and procedures that were developed to accelerate solvent degradation [8]. Indeed, solvent degradation is a slow phenomenon taking place over months in industrial capture plants, so it has to be accelerated in order to enable its study within a reasonable timeframe at the lab scale. The relevance of accelerated degradation conditions could be evidenced by reproducing in one-week lab experiments similar degradation pathways as observed in industrial CO$_2$ capture pilot plants over several months. Then, using this equipment, the influence of the process operating conditions was experimentally studied, leading to a kinetic model for MEA oxidative and thermal degradation [9]. However, some assumptions made in first approach may be improved. The present work thus provides additional experimental data to refine this model and proposes an updated kinetic model for MEA oxidative degradation. Finally, the resulting kinetics is included into a global model of the post-combustion CO$_2$ capture process, so that the final model provides a useful tool for assessing solvent degradation during the design stage of large-scale CO$_2$ capture plants.

2. Experimental study

In this section, we first briefly describe the equipment and the procedures that have been developed for the study of monoethanolamine oxidative degradation. Then, the results of the experimental study are presented and discussed. Finally, the kinetic model for MEA oxidative degradation developed in [9] is improved to take these additional results into account.

2.1. Material and methods

The study of MEA oxidative degradation requires a continuous gas feed since this phenomenon is limited by the rate of gas-liquid transfer [10]. Thus, oxidative degradation experiments were conducted in an experimental Degradation Test Rig with continuous gas flow. This test rig consists in an agitated pressurized reactor with continuous gas feed. It allows the degradation of solvents at temperatures up to 140 °C and pressures up to 2 MPa, with flexible gas composition and variable agitation rate. Typically, 300 g of 30 wt% MEA (1.47 mol MEA and 11.67 mol H_2O) are weighted into the reaction vessel. Contrarily to previously reported experiments [9], amine solutions are not loaded with CO_2 in the present study. The experiment runs for one week at 120°C, 0.4 MPa (gauge) and 600 rpm with a continuous gas flow rate (160 NmL/min) usually composed of 5% O_2 and 95% N_2. In order to characterize the degraded solvent samples, different analytical methods have been developed. The MEA content is determined by high performance liquid chromatography (HPLC) while the liquid degradation products are quantified using gas chromatography (GC). Gaseous degradation products are quantified by Fourier transformed infra-red spectroscopy (FTIR). The main quantified compounds and the corresponding analytical techniques are reported in the Appendix A. Besides these components, the concentration of dissolved metal ions (Fe, Cr, Ni and Mn) is measured in liquid solvent samples by Atomic Absorption Spectrometry (AAS). A detailed description of the equipment and procedures has been published previously [8].

2.2. Experimental results

The oxidative degradation model described in [9] relies on experiments that have been systematically performed in the presence of CO_2. The reason for that is that an inhibitive effect of CO_2 on MEA oxidative degradation was evidenced by [10], but this inhibitive effect appeared to be independent of the CO_2 concentration in the gas feed [8]. Thus, as CO_2 is always present in CO_2 capture applications, we initially considered that conducting oxidative degradation experiments in the presence of CO_2 was more representative of industrial conditions. However, small quantities of typical thermal degradation products were reported at 120°C and 140°C under oxidative degradation conditions. Thus, in the present work, the oxidative degradation experiments described in [9] have been repeated in the absence of CO_2 in order to completely decouple the oxidative and thermal degradation pathways. This leads to more accurate data regarding the influence of oxygen and temperature on the oxidative degradation of MEA.

Figure 1 (left) represents the influence of the oxygen concentration in the gas feed on the MEA loss and the emission of ammonia. Since the gas feed consists in a mix of O_2 and N_2, the variation of the O_2 concentration was compensated for with N_2 in order to keep a constant gas feed flow rate. As expected, both the MEA loss and the ammonia emission increase with the oxygen concentration. Figure 1 (right) evidences that HEEDA and HEIA are not observed. Since they are typical thermal degradation products [11], this confirms that no thermal degradation takes place in the test conditions. It also appears that HEI is the second largest quantified degradation product after ammonia, and that the formation of HEI dramatically increases with the O_2 concentration. This result is expected since HEI has been identified as a major oxidative degradation product in [12]. Moreover, it seems that the formation of HEPO and BHEOX does not depend on the O_2 concentration in the gas feed.
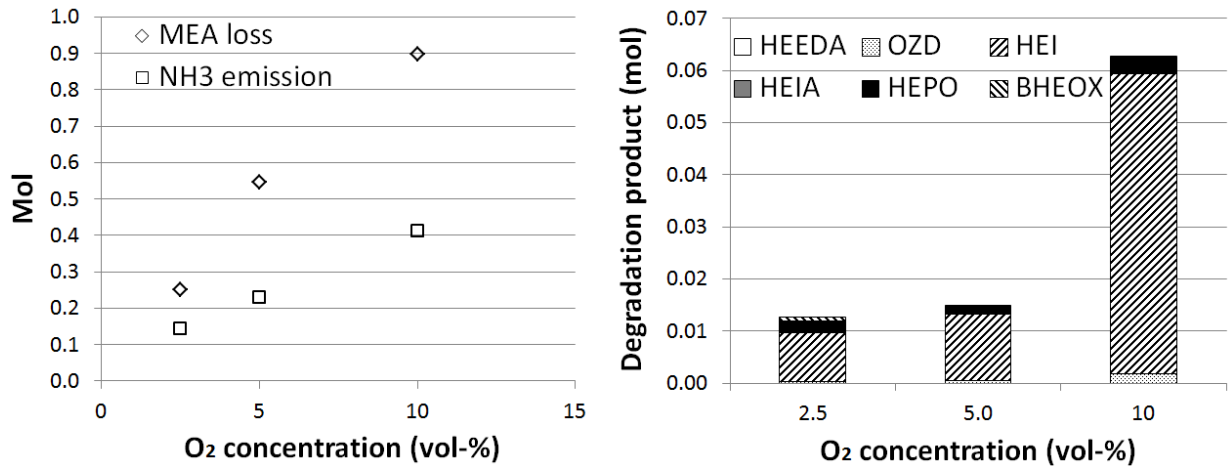
Figure 1. Influence of the O_2 concentration in the gas feed on MEA degradation and NH_3 emission (left) and on liquid degradation products (right) (300 g, 30 wt% unloaded MEA, 120°C, 0.4 MPa (gauge), 600 rpm, 160 NmL/min gas feed, 1 week).

The effect of temperature on the MEA loss and the ammonia emission is evidenced in Figure 2 (left). The MEA loss increases from 0.05 mol at 55°C to 0.70 mol at 140°C (initial MEA content: 1.47 mol). At the same time, the NH_3 emission increases from 0 to 0.39 mol. Figure 2 (right) shows again that no HEIA or HEEDA may be observed. The influence of the temperature on the formation of HEPO confirms the condensation mechanism proposed in [7]. The lower amount of HEI formed at 140°C can be explained by its lower thermal stability [12].
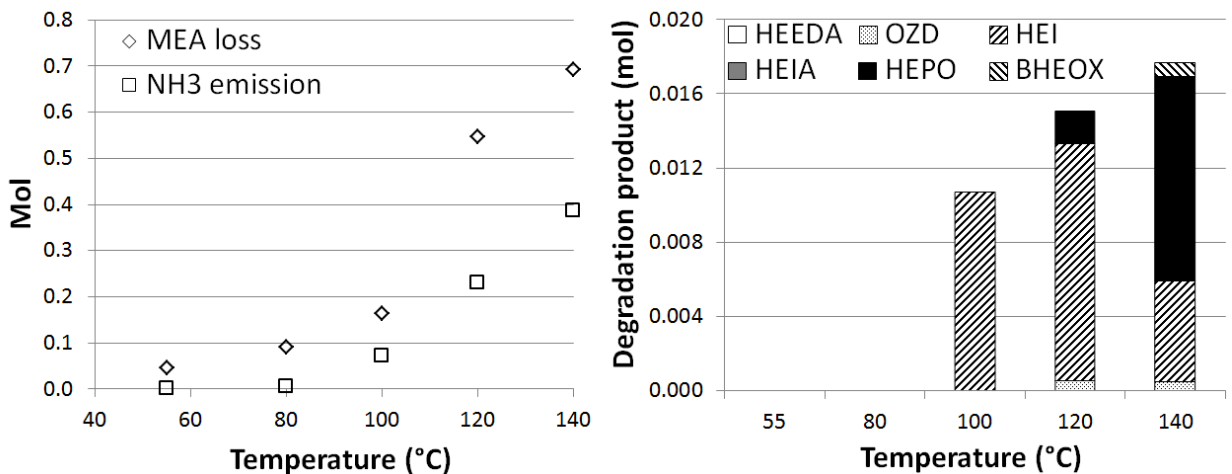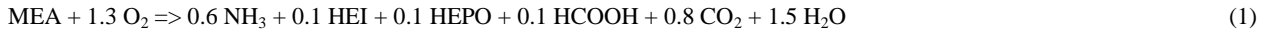

Figure 2. Influence of the temperature on MEA degradation and NH3 emission (left) and on liquid degradation products (right) (300 g, 30 wt% unloaded MEA, 120°C, 0.4 MPa (gauge), 600 rpm, 160 NmL/min gas feed, 1 week).

2.3. Kinetic model of MEA degradation

A kinetic model for MEA thermal and oxidative degradation has been proposed in [9]. In the present work, we keep the thermal model unchanged since no additional thermal degradation experiments have been performed. However, the oxidative degradation model is updated based on the experiments reported in the previous section. Since oxidative degradation mechanisms are complex and still partially unknown, the oxidative degradation of MEA is described by an apparent reaction mechanism that is based on several irreversible reactions implying the main identified degradation products. From the experiments above, we consider ammonia, HEI, HEPO and formic acid

(HCOOH) as the main degradation products resulting from MEA oxidative degradation. Formic acid is the acidic form of formate, and it is included in the model in order to consider the formation of heat stable salts. Indeed, although it was not identified in our test conditions, formate is the main carboxylic acid identified in degraded MEA samples from industrial pilot plants and its formation was reported in many experimental studies (among others in [11] and [13]). The resulting apparent reaction of MEA with oxygen is given by Equation (1). It is weighted in accordance with the experimentally observed distribution of degradation products to lead to an overall reaction balance.

$$MEA + 1.3\ O_2 => 0.6\ NH_3 + 0.1\ HEI + 0.1\ HEPO + 0.1\ HCOOH + 0.8\ CO_2 + 1.5\ H_2O \tag{1}$$

In order to determine its kinetics based on Arrhenius' equation, the influence of MEA on the degradation kinetics is neglected in a first approach since MEA is not the limiting reactant and is present in large excess in the bulk. Similarly, the influence of CO_2 on MEA oxidative degradation is not considered in the present model. The inhibitive effect of CO_2 on MEA oxidative degradation that has been observed in [10] is assumed to be due to a lower solubility of O_2 in CO_2-loaded solutions. As a consequence, the oxidative degradation reaches a lower extent in the presence of CO_2 but its kinetic rate itself is not affected by CO_2. This is supported by the fact that CO_2 is not a reactant of MEA oxidative degradation. Finally, the dissolved O_2 concentration in the solvent has been approximated by Henry's law for oxygen in 30 wt% MEA according to [14]. Based on these assumptions, the reaction rate of the MEA oxidative degradation (in mol/L.s) can be expressed by Equation (2):

$$-r_{MEA,\ Oxidative} = 1.360\ 10^6 \cdot e^{-55\ 111/RT} \cdot [O_2]^{1.03} \tag{2}$$

R is the universal gas constant (8.314 J/mol.K), T the temperature (K), and $[O_2]$ the concentration of dissolved oxygen (mol/L). The value for the activation energy is given in J/mol and the pre-exponential unit is $(mol/L.s)/(mol/L)^{1.03}$. The pre-exponential constant, the activation energy and the kinetic order of oxygen appearing in Equation (2) have been determined by minimizing the sum of squared differences between the model-predicted degradation rates and the observed degradation rates based on a set of seven degradation experiments. The parameter regression has been performed with the solver tool embedded in the Microsoft Excel 2010 software. The operating conditions of the degradation experiments used for the regression are reported in Table 1. For each experiment, the observed degradation rate is compared to the predicted rate which has been calculated by dividing the mol number of degraded MEA by a run time of one week (604 800 s) and a solvent volume of 0.3 L. Although the initial quantity of solvent equals 0.3 kg, the effect of density has been neglected since the density of fresh 30 wt% MEA has been measured as 1.004 kg/L. The mean error between predicted and observed values equals 19%, mainly due to high experimental uncertainties at low degradation rates.

Table 1. Experimental points for determining the kinetics of MEA oxidative degradation.

Temperature	O_2 concentration in the gas feed	Observed degradation rate	Model-predicted degradation rate
K	vol%	mol MEA/L.s	mol MEA/L.s
328	4.9	$2.51\ 10^{-7}$	$2.94\ 10^{-7}$
353	4.9	$4.96\ 10^{-7}$	$7.33\ 10^{-7}$
373	4.9	$9.07\ 10^{-7}$	$1.35\ 10^{-6}$
393	5.0	$3.01\ 10^{-6}$	$2.43\ 10^{-6}$
393	2.4	$1.39\ 10^{-6}$	$1.17\ 10^{-6}$
393	9.9	$4.95\ 10^{-6}$	$4.89\ 10^{-6}$
413	5.0	$3.81\ 10^{-6}$	$4.09\ 10^{-6}$

3. Simulation study

In this section, we first describe the basic assumptions that have been made to consider solvent degradation into a global process model of the post-combustion CO_2 capture. The simulation results are then presented and discussed.

3.1. Modelling assumptions

The kinetic model for MEA oxidative and thermal degradation presented above has been included into a global rate-based process model developed in Aspen Plus 8.0 [15]. The flowsheet of the process is shown in Figure 3. This model represents the Esbjerg pilot plant described in [16], treating a flue gas flow rate of 5000 Nm³/h. The underlying assumptions of the present global process model have been previously discussed in details in [17]:

- Degradation reactions are included into a steady-state model of the post-combustion CO_2 capture process
- Degradation reactions only take place in the absorption and stripping columns in order to better reflect the actual process operating conditions. Degradation in other process equipment has been neglected.
- Component data for HEI, HEPO and HEIA are estimated based on the component chemical structures. Moreover, these components have been defined as non-volatile to facilitate the liquid-vapor equilibria calculations. This assumption is supported by the high molecular weights of HEI, HEPO and HEIA, respectively equal to 112.13, 144.17 and 130.15 g/mol.
- Solvent purge and make-up have been neglected as they do not prevent the model from converging. As a consequence, degradation rates are quantified by the reaction rates reported in mass transfer columns since column reactions are precisely calculated by Aspen Plus at the contrary of solvent loop concentrations.
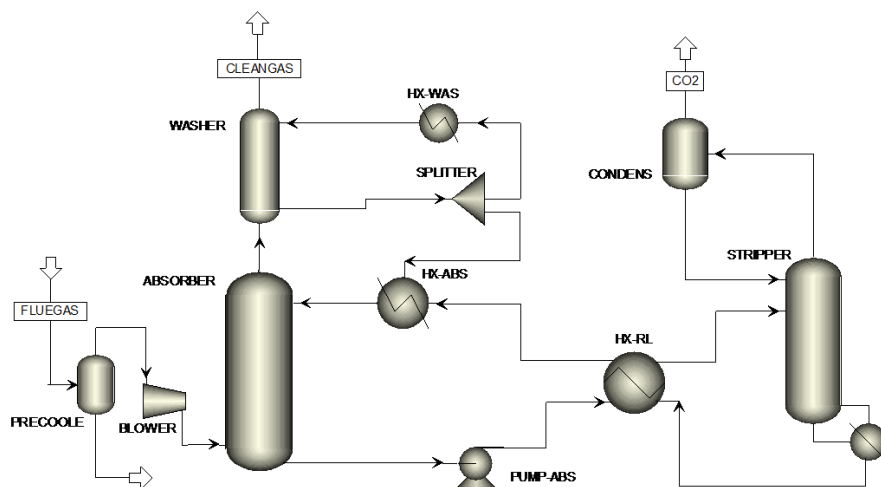

Figure 3: Flowsheet of the post-combustion CO_2 capture process with assessment of solvent degradation

3.2. Simulation results

The reboiler heat duty predicted by the model equals 3.64 GJ/t CO_2 at a solvent flow rate of 24.44 m³/h. This is in agreement with the values observed during the pilot plant campaign described in [16]. Table 2 lists the formation rates of degradation products as well as the MEA degradation rate in the absorber and the stripper. The formation rates of ammonia and HEIA are reported as an indication of the oxidative and thermal degradation rates respectively. The formation rates of HEI, HEPO and HCOOH can be calculated from the formation rate of NH_3 based on the stoichiometry of Equation (1). The emissions in the cleaned flue gas and the CO_2 product streams are also reported, before and after passing the column washing sections. Since HEI, HEPO and HEIA are not present in the component database of Aspen Plus, they have been assumed as nonvolatile components, so that there are not

present in the gas streams. The liquid temperature and the vapor oxygen content at the top and bottom stages of the columns are also indicated. All degradation and emission values have been normalized by the amount of captured CO_2 which equals 1.24 ton/h in the simulation (90% capture rate, 5000 Nm³/h flue gas).

Table 2. Degradation and emission results of the degradation model.

Parameter	Unit	Absorber	Stripper	Total
MEA degradation	kg/ton CO_2	$7.94 \ 10^{-2}$	$9.64 \ 10^{-5}$	$7.95 \ 10^{-2}$
NH_3 formation	kg/ton CO_2	$1.33 \ 10^{-2}$	$1.53 \ 10^{-5}$	$1.33 \ 10^{-2}$
HEIA formation	kg/ton CO_2	$6.56 \ 10^{-9}$	$5.92 \ 10^{-6}$	$5.93 \ 10^{-6}$
MEA emission before washing	kg/ton CO_2	$2.29 \ 10^{0}$	$1.17 \ 10^{0}$	$3.46 \ 10^{0}$
NH_3 emission before washing	kg/ton CO_2	$1.13 \ 10^{-2}$	$3.16 \ 10^{-3}$	$1.44 \ 10^{-2}$
HCOOH emission before washing	kg/ton CO_2	$1.15 \ 10^{-3}$	$4.09 \ 10^{-4}$	$1.55 \ 10^{-3}$
MEA emission after washing	kg/ton CO_2	$1.10 \ 10^{-3}$	$9.02 \ 10^{-9}$	$1.10 \ 10^{-3}$
NH_3 emission after washing	kg/ton CO_2	$1.02 \ 10^{-2}$	$2.91 \ 10^{-3}$	$1.31 \ 10^{-2}$
HCOOH emission after washing	kg/ton CO_2	$1.44 \ 10^{-4}$	$1.73 \ 10^{-5}$	$1.62 \ 10^{-4}$
MEA consumption (degradation + emission after washing)	kg/ton CO_2	$8.05 \ 10^{-2}$	$9.64 \ 10^{-5}$	$8.06 \ 10^{-2}$
Top stage liquid temperature	°C	57.5	96.7	-
Bottom stage liquid temperature	°C	51.1	115.6	-
Top stage O_2 content (vapor phase)	mol%	6.3	$9.5 \ 10^{-3}$	-
Bottom stage O_2 content (vapor phase)	mol%	6.1	$1.7 \ 10^{-13}$	-

No MEA consumption data were reported for the test campaign described in [16], but MEA consumption and emission data were reported in a study performed at another pilot plant [18]. It should be noted that this latest study could close the MEA balance within a measuring uncertainty of only 10%, so that its results can be discussed with confidence. It appears that the total MEA loss reported in Table 2 is in the same order of magnitude compared with the CO_2 capture plant results, although the predicted values are lower. A MEA consumption rate of 0.284 kg MEA/t CO_2 was reported in the absence of degradation inhibitors in [18] while the model prediction only reaches 0.081 kg MEA/t CO_2. The model also predicts a lower emission of NH_3 (0.010 kg NH_3/t CO_2) than reported in [18] where the ammonia emission varied between 0.089 and 0.156 kg NH_3/t CO_2. The emission predicted by the model is thus one order of magnitude lower than the value reported from pilot plant. However, this result has to be compared to previous attempts to model solvent degradation. Indeed, only one previous model for predicting MEA degradation and ammonia emission in CO_2 capture plants has been identified [4]. The rate of MEA degradation was not reported in [4], but the authors calculated an ammonia emission rate of 1.10^{-13} kg/ton CO_2, which is far away from the value reported [18].

It also appears from Table 2 that more oxidative degradation products (NH_3) are formed than thermal degradation products (HEIA) over the entire process. Moreover, the highest MEA loss is observed in the absorber. These results are in accordance with previous studies evidencing MEA oxidative degradation in the absorber as the main degradation pathway in industrial CO_2 capture units [19]. Furthermore, MEA losses due to solvent emission are much lower than losses due to MEA degradation thanks to the washing sections. The present model only considered basic water washing sections, so it is likely that emissions would be lower with advanced washing sections using acid wash for instance. This may also have an impact on the emissions of NH_3. In the present configuration, almost all the produced NH_3 is emitted.

Finally, the degradation model provides useful information about the influence of operating conditions on MEA degradation and on the emission of degradation products. The differences between the model predictions and the

MEA loss and ammonia emission observed in pilot plant may be due to the assumptions done in first-approach. For instance, the presence of SO_X and NO_X contaminants in the flue gas as well as the presence of dissolved metals in the solvent solution have been neglected in this first step. Since all these components are known to increase the degradation rate [13], they should be considered in further model developments.
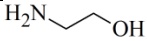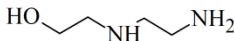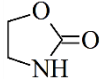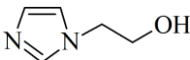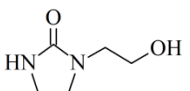
4. Conclusions

The present work proposes a methodology for taking solvent degradation into consideration during the design process of CO_2 capture plants. First, the degradation of MEA is studied under high temperature with a continuous gas feed. The main difference with previous works performed in the same equipment as reported in [9] is that the oxidative degradation is studied in the absence of CO_2 in order to completely decouple the oxidative and the thermal degradation pathways. The influences of the temperature and of the oxygen partial pressure in the gas feed are studied, and the MEA loss and the production of degradation products are quantified. Based on these experimental results, a kinetic model considering both MEA thermal and oxidative degradation is proposed. This kinetic model is then included into a global process model of a CO_2 capture pilot plant in Aspen Plus in order to consider MEA degradation.

The reboiler heat duty predicted by the model is in agreement with the values reported during the pilot plant campaign. The predicted MEA loss is in the same order of magnitude than the loss observed in a recent pilot plant although three times lower. Thus, the present model still has to be refined in order to include the effect of SO_x, NO_x and dissolved metals on solvent degradation. Indeed, these elements have been neglected in first approach, but are known to increase MEA degradation, so that they should be included in further model developments. Finally, this kind of model could and should be used for the design of CO_2 capture plants to consider not only the process energy penalty, but also its environmental penalty. Both aspects are particularly relevant in case of large-scale deployment of the CO_2 capture technology.
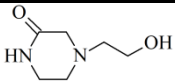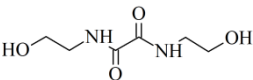
Appendix A. Summary of organic compounds quantified in degraded MEA samples

Abb.	Compound	CAS Number	Structure	Analytical method
MEA	monoethanolamine	141-43-5		HPLC
HEEDA	*N*-(2-hydroxyethyl)ethylenediamine	111-41-1		GC
OZD	2-oxazolidinone	497-25-6		GC
HEI	*N*-(2-hydroxyethyl)imidazole	1615-14-1		GC
HEIA	*N*-(2-hydroxyethyl) imidazolidinone	3699-54-5		GC

HEPO	4-(2-hydroxyethyl)piperazine-2-one	23936-04-1		GC
BHEOX	*N,N'*-bis(2-hydroxyethyl)oxamide	1871-89-2		GC
NH_3	Ammonia	7664-41-7	NH_3	FTIR

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