

Prospects for Membrane Deoxygenation of Alkanolamine CO₂ Solvents to Prevent Their Degradation (A Minireview)

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Abstract—Absorption by aqueous solutions of alkanolamines is the most mature technology for removing carbon dioxide (CO₂) from natural gas, off-gases from power engineering, and other mixtures. The possible presence of oxygen in the gas mixture being treated or air leakage into the absorption treatment system leads to oxidative degradation of the amine and deactivation of the absorption liquid with the formation of a wide range of corrosion-active degradation products. The paper discusses modern views on the effect of dissolved oxygen on the degradation of amine solvents. The main methods for preventing oxidative degradation of alkanolamines and fighting against it are described. Direct removal of oxygen (deoxygenation) from absorption liquids in compact and modular membrane gas–liquid contactors is suggested as an alternative countermeasure. The retrospective and state-of-the-art of membrane deoxygenation of aqueous media are presented. Advanced studies in the field of oxygen removal from amine CO₂ solvents used for flue gas treatment are described.

Keywords: membrane contactor, composite membrane, deoxygenation, oxygen, degradation, alkanolamine, carbon dioxide

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INTRODUCTION

Treatment of natural and process gases to remove acid impurities (primarily carbon dioxide CO₂ and sulfur compounds) is an important task of industrial gas-refining technologies. Furthermore, there are trends in the world toward economic stimulation of the reduction of anthropogenic CO₂ emissions to mitigate their effect on climate, in particular, by introducing carbon tax and cross-border carbon taxes [1]. Therefore, the carbon dioxide removal from various process streams is topical from both economic and environmental viewpoints. Despite active studies on the development of new efficient methods for carbon dioxide capture, the most technologically mature industrial technology in the world is amine treatment, namely, CO₂ absorption by aqueous solutions of various alkanolamines [2–4]. On the other hand, regeneration of the solvents (CO₂ desorption) at elevated temperatures (100–120°C) is accompanied by gradual chemical degradation of the amines, initiated by oxygen dissolved in the absorption liquid [5]. Oxygen can be present in a

gas mixture being treated. For example, off-gases from metallurgy, petrochemical processes, and power plants can contain up to 15 vol % O₂ [6]. Oxygen can get into the amine during its storage in an untight vessel (contact with air) or air leakage into the amine treatment system [2].

OXIDATIVE DEGRADATION OF ALKANOLAMINE SOLVENTS

The presence of dissolved oxygen in amine solvents intensifies the equipment corrosion [7–11] and leads to direct oxidation of amines (oxidative degradation of amine solvents). As a result, their loss can reach from 0.2 to 3.65 kg per ton of the recovered CO₂ [12]. The oxidation mechanism includes numerous reactions starting, apparently, with the attack of free oxygen radicals on molecules of amines or amine carbamates [6, 13, 14]. These reactions are relatively slow, but under the solvent regeneration conditions (100–120°C) their rate appreciably increases. The reactions yield a wide spectrum of degradation products such as carboxylic acids, amino acids, amides, amines, aldehydes, ammonia, etc. [13].

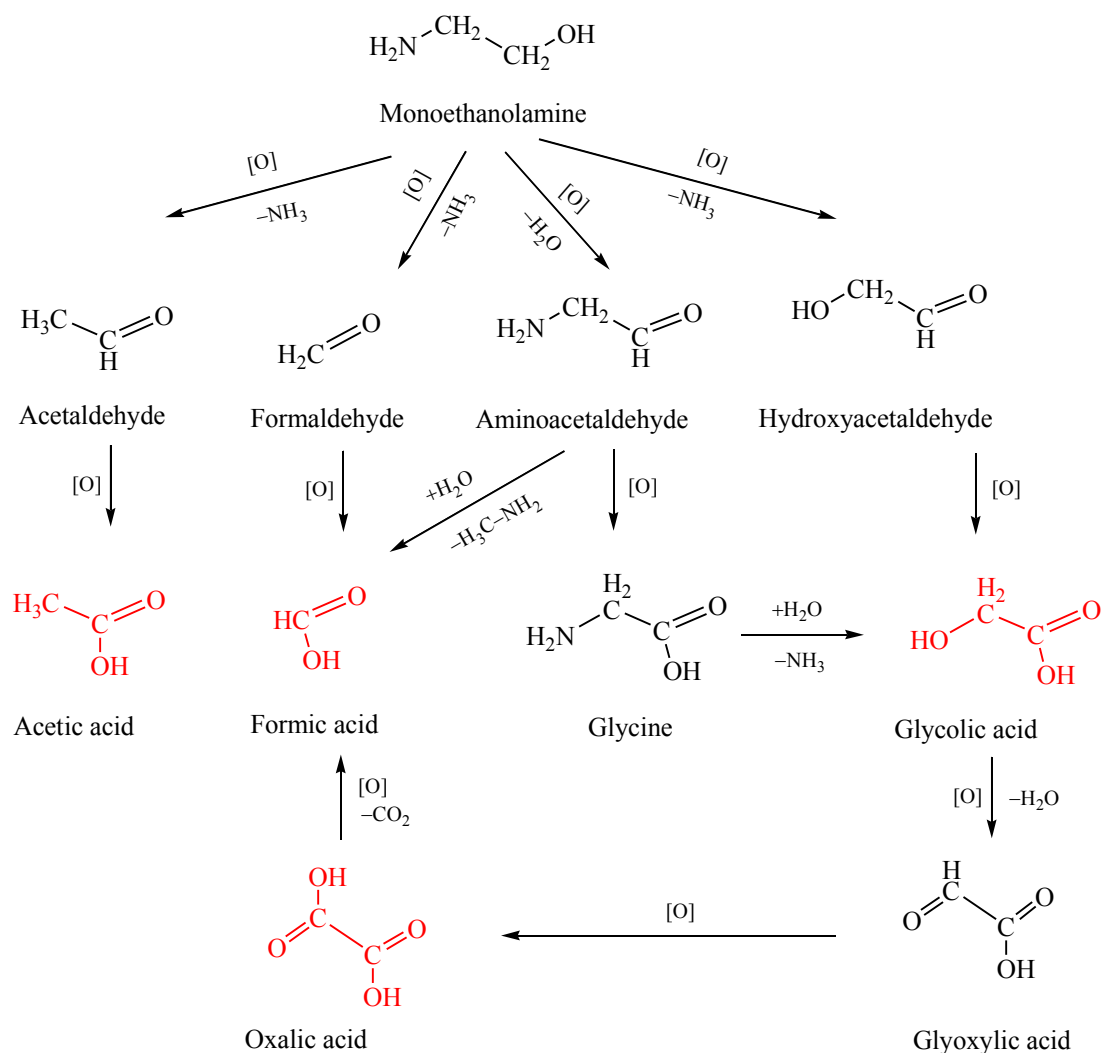


Fig. 1. Scheme of oxidative degradation of monoethanolamine to form carboxylic acids (data adapted from [13, 16]).

Carboxylic acids (formic, acetic, glycolic, oxalic, etc.) prevail, because, being heat-stable, they are formed in the final step of diverse chemical transformations of alkanolamines [13, 15, 16]. The modern review of the results of 29 pilot campaigns with various amines on 18 pilot plants for CO₂ removal from flue gases throughout the world [6] confirms the presence of these compounds in all the cases under consideration. The scheme of oxidation reactions yielding carboxylic acids is shown in Fig. 1 for monoethanolamine as an example.

In addition, the degradation is caused by the reaction of amines with impurities in the mixture being purified (e.g., sulfur and nitrogen oxides) or in the liquid, originating from the equipment corrosion and feeding

of water of poor quality [5]. Side reactions lead to the formation of heat-stable salts (HSSs) of protonated alkanolamine with anions of organic and inorganic acids [17, 18], which do not decompose during the CO₂ desorption [3, 15]. Oxidation products alter the physicochemical properties of the solvent and lead to its foaming and to the equipment erosion and contamination; some of them irreversibly bind the active amine into nonregenerable compounds (in particular, HSSs), which, when accumulated in the system, decrease its overall performance [5, 16, 18]. Being strong corrosive agents [5, 18], these compounds lead to an increase in the content of equipment corrosion products (iron, chromium, nickel ions, etc.) in the solution; these ions, in turn, catalyze

further oxidation of the amines [19, 20], actually effecting autocatalytic degradation of the absorbent.

PREVENTION OF THE OXIDATIVE DEGRADATION OF ALKANOLAMINES AND METHODS OF FIGHTING AGAINST ITS CONSEQUENCES

In principle, the oxidative degradation can be prevented by introducing oxidation inhibitors such as sodium or potassium sulfite or bisulfite [21, 22], hydrazine, or hydroxylamine [23, 24] into amine solutions. However, these inhibitors are toxic for humans and environmentally hazardous; furthermore, they can cause solution foaming. New human-safe inhibitors of corrosion and amine oxidation were suggested recently [25]: carbonylhydrazide, 2-butanone oxime, and α,α' -(1-methylethylenediimino)di-*o*-cresol as oxidation inhibitors and 1,3-diaminopropane-*N,N,N',N'*-tetraacetic acid, pyrogallol, and tricine as complexing agents. Monoethanolammonium and mercaptoethylammonium tartrates were used for this purpose in [26], and 2,5-dimercapto-1,3,4-thiadiazole, diethylenetriaminepentaacetic acid, and hydroxyethylenediphosphonic acid, in [27, 28]; these compounds allow the monoethanolamine oxidation to be decreased by more than 90%. Sodium metavanadate is widely used in industry for preventing the corrosion caused, in particular, by amine oxidation products [2]; however, this compound, on the contrary, catalyzes the oxidative degradation of amines [29, 30].

A number of reviews on the existing methods for removing oxidative degradation products and HSSs [5, 16–18]. The main methods for reducing the negative effect of degradation products and HSSs are partial replacement (“purging”) of the solvent, its alkalization, and vacuum distillation reclaiming [31, 32]. In the case of vacuum distillation reclaiming, water and pure alkanolamine are transported in the vapor phase into the desorber under the so-called blind plate, and HSSs and other amine degradation products remain in the reclaiming sludge. This technique is the most power-consuming, and the reclaiming sludge is toxic and requires special utilization measures [33–35]. Alternative reclaiming methods are based on sorption; among them, the technique for removing HSS anions on ion-exchange resins has been commercially developed [36, 37]. These methods require large amounts of reagents (acids and alkalis) for resin regeneration, which leads to the

formation of a large amount of dilute waste solutions [16]. The new approaches within this field include the use of new generations of sorbents based on biodegradable natural materials (chitosan and sodium alginate [38–41]) and capacitive deionization (an electrosorption process with porous electrodes for HSS “accumulation”) [42, 43]. The third route is based on membrane processes, which are less power- and reagent-consuming. For example, nanofiltration is promising for HSS preconcentration [44, 45], and electro dialysis reclaiming is a commercial process for HSS removal from solutions of secondary and tertiary amines in gas processing [5, 46] and from amine plant wastewaters [47, 48]. Electro dialysis is suitable for removing HSSs from solvents for flue gas treatment based on monoethanolamine (a primary amine) [49–52], but the transfer of HSSs from solutions, especially from those with low HSS content, is hindered because of the presence of residual bound CO_2 , which leads to large amine loss. This loss can be reduced by using two-step schemes of electro dialysis reclaiming [53, 54]. A new method of HSS extraction with hydrophobic organic extractants was suggested recently [55, 56]; these extractants allow reclaiming of solvents with low HSS content (1000 ppm) without heat supply and with minimal power consumption. Solutions of amines or quaternary ammonium salts with long side substituents ($C \geq 8$) in higher alcohols ($C \geq 6$) immiscible with amine solutions were used. Akkarachalanont et al. [55] have shown that tricaprilmethylammonium hydroxide in 1-octanol binds HSSs by the acid–base mechanism. The extractant performance can be increased by using branched 2-ethyl-1-hexanol as a solvent [56].

MEMBRANE DEOXYGENATION OF AQUEOUS MEDIA: RETROSPECTIVE AND IMPLEMENTATION FEATURES

Membrane technologies are power-saving alternatives to traditional separation methods [57] and can be used for solving technology problems of CO_2 removal with amines. In particular, to prevent the negative effect of oxygen, it is promising to remove it directly from amine solutions by deoxygenation (removal of molecular dissolved oxygen) in membrane gas–liquid contactors. A membrane contactor is an apparatus for separation or chemical conversion in which the membrane acts as a phase contact surface. In the case of a system with mass transfer from a gas into a liquid, the liquid phase contains a selective chemisorbent [58]. In particular,

the use of hollow fiber membranes in contactors allows reaching high (up to 5000 m²/m³) specific surface area of the membrane in the apparatus [59]. This leads to a 400–1500% increase in the mass transfer area per unit volume of the apparatus [60], compared to the traditional separation equipment. Therefore, membrane degassing contactors have up to 10 times smaller size [61]. The additional advantages are as follows: no mutual dispersion of phases and, correspondingly, no problems with the mixing and subsequent separation of the phases; independent control of the phase flows; simple assembling/operation and low operation expenditure; modular design allowing the productive capacity of separation units to be linearly increased or decreased depending on the process demands [61, 62].

The advantages of membrane contactors were clearly demonstrated in membrane degassing of liquids, primarily in the removal of dissolved oxygen from water. This problem should be solved in pharmaceutical, food, biotechnological [63], and semiconductor [64] industry and in power engineering [65]. For example, in power engineering, the removal of dissolved oxygen from water to the required level of 5 ppm allows the corrosion of boilers and pipelines of heat and power plants to be considerably reduced. In microelectronics, this level is lower by an order of magnitude and can reach 0.1 ppb in the case of preparing ultrapure water for washing silicon wafers for microchips [64].

The need for oxygen removal from water became one of decisive vectors of the development of the membrane contactor technology. For example, as early as 1986 [66], Yang and Cussler presented countercurrent and crosscurrent designs of hollow-fiber contactor modules, performed the first experiments on water deoxygenation, and obtained mass transfer correlation equations, which appeared to be comparable to the previously obtained dependences for heat and mass transfer problems. The main conclusion from their study consisted in that the key parameter of hollow-fiber contactor modules should be the mass transfer area per unit volume of the apparatus rather than the mass transfer coefficient, because the former parameter considerably exceeds that reached in traditional separation equipment, whereas the latter parameter is on the similar level. The same authors [67] used modules with hollow polypropylene fibers (Celgard X20, Questar, Charlotte, NC membranes; inside diameter 200 μm; wall thickness 25 μm; porosity 30%; mean pore size 30 nm; number of fibers in the module 2700–10000)

to recover dissolved oxygen from water for maintaining the livelihood of living bodies in a closed space (artificial gill technology). They demonstrated principal possibility of membrane oxygen supply of small animals: hamsters, rats, and one dog [67]. It was shown under the Cussler's guidance that the optimum size of fibers of hollow-fiber modules from the viewpoint of cost per unit weight of the transferred oxygen is 100–200 μm [68]. At smaller fiber size, the power consumption for water pumping through the module becomes too high, whereas at larger fiber size the membrane becomes unduly expensive [68]. Finally, the possibility of using membranes for preparing ultrapure deoxygenated water was demonstrated by Tai et al. [69] and Yagi et al. [70]. In the first paper, the authors reduced the oxygen content of water to 8 ppb using modules based on the above-described hollow-fiber polypropylene membranes, whereas in the second paper the membrane modules were used in combination with sorption or nitrogen bubbling to obtain water with the residual oxygen content of 10 ppb for producing submicrometer ultra large integrated circuits.

There are three ways to ensure the driving force of the transmembrane oxygen transfer: (1) vacuum pumping of the gas part of the contactor [64, 65, 71–76]; (2) stripping with an inert gas [71, 77–79]; (3) combination of both [71, 78]. In the first case, the highest degree of deoxygenation is reached when applying a vacuum from both sides of the membrane module. In the second case, an inert gas (most frequently nitrogen) is fed into the module in the countercurrent mode. This method is efficient, but deep purification requires high-purity nitrogen, and large amount of water is evaporated in the process, which makes the process power-consuming; furthermore, the water being purified is saturated with nitrogen. The third approach allows control of the residual concentration of the purging gas in water. The use of solutions of antioxidants (e.g., sodium sulfite, ascorbic acid, etc.) as an alternative to evacuation or purging is possible in principle, but we found no data on implementation of this process in the literature.

For removing oxygen from water, the overwhelming majority of researchers use Liqui-Cel® (3M) contactors based on cheap hydrophobic polypropylene hollow-fiber membranes with the geometric parameters of 0.3/0.2–0.24 mm and mean pore size of 30 nm [71, 73, 76, 78, 80]. Nevertheless, hydrophobic porous hollow fibers of polyethylene [72], polyvinylidene fluoride [81], and polysulfone [77] have also found use. Nonporous hydrophobic polydimethylsiloxane membranes [64, 74]

and composite membranes with a thin selective layer of polydimethylsiloxane on a polyvinylidene fluoride support [82] and of perfluorodimethyldioxole–tetrafluoroethylene copolymer on a polypropylene support [80] also show promise. The modern achievements in the field of hollow fiber membranes allow preparation for this purpose of three-channel porous hollow polyvinylidene fluoride fibers [83, 84] and of helical porous hollow-fiber polypropylene membranes [85] with the pore size of 2–200 nm. The process upscaling allows removal of dissolved oxygen from recycled water streams of heat and power plants with the productive capacity of up to 5 m³/h, with simultaneous reduction of the corrosion activity of water. It was demonstrated in pilot trials of degassing apparatuses with porous hollow fiber poly-4-methyl-1-pentene membranes (membrane area 40 m²) [65] and polypropylene membranes (membrane area 42 m²) [75]. Even under semicommercial conditions, the authors were able to reduce the oxygen content of water by three orders of magnitude [71]; the cost of water degassed by the membrane technique is two times lower than the cost of water degassed by heating [78]. Finally, the efficiency of the deoxygenation in contactors can be considerably increased with catalytic hollow fibers. For example, contactors based on commercial hollow-fiber polypropylene membranes coated with palladium nanoparticles catalyzing the reaction of oxygen dissolved in water with hydrogen fed from the internal side of the membranes were suggested at the Topchiev Institute of Petrochemical Synthesis, Russian Academy of Sciences [86, 87]. This considerably improves the deoxygenation kinetics even at initially low oxygen concentrations in water.

MEMBRANE DEOXYGENATION OF CO₂ SOLVENTS

Taking into account the aforesaid, the membrane deoxygenation of amine solvents is apparently a topical problem. However, by now, wide studies in this direction have been initiated by the Netherlands Organization for Applied Research in the Field of Natural Sciences (Nederlandse Organisatie voor Toegepast Natuurwetenschappelijk Onderzoek, TNO) [88, 89], which is one of the world leaders in the field of post-combustion CO₂ capture. First, as shown by researchers [88], standard commercially available electrochemical and optical O₂ sensors can be used for direct measurements of the concentration of dissolved oxygen in aqueous

solutions of alkanolamines; variation of the alkanolamine type and concentration only weakly influences the O₂ solubility. A simple model was developed for predicting the O₂ solubility in carbonated ethanolamine solutions, and the possible salting-out effect on this quantity was outlined [88]. Second, Monteiro et al. [89, 90] reported the results of DORA membrane contactor process trials (Direct Oxygen Removal Apparatus, device for removing dissolved oxygen) developed by TNO to prevent oxidative degradation of post-combustion CO₂ capture solvents. The DORA technology was tested in combination with a TNO mobile pilot plant for CO₂ capture from simulated flue gases with the productive capacity of 5 m³_(STP)/h, shown in Fig. 2. The deoxygenizer was a Liqui-Cel™ SP Series (2.5 × 8 extra-flow model) hollow-fiber membrane contactor module with the total membrane area of 1.4 m². The use of the membrane contactor for the solvent deoxygenation reduced the ammonia emission from the absorber by approximately 70%, which suggests reduced degradation. The potentialities of the technology were demonstrated under industrial conditions in treatment of flue gases from a diesel generator with a 30 wt % aqueous monoethanolamine solution. The results show that the deoxygenation in the membrane contactor reduces of the absorbent degradation. Figure 3 shows how the content of acetate and oxalate in the solvent vary with time. As can be seen, the switching-on and operation of the membrane deoxygenizer allow the acetate and oxalate formation rate to be considerably reduced and their concentration to be maintained on a constant level for ~700 h (shown by a yellow rectangle). Switching-off of the deoxygenizer leads to sharp intensification of the solvent degradation, reflected in acceleration of the acetate and oxalate formation.

As the developers indicate, the process can be implemented not only in the traditional version with vacuum/stripping, but also using liquid oxygen scavengers from the other side of the membrane (liquid–liquid contactor mode) [91]. It should be noted that the DORA technology was tested using standard Liqui-Cel® membrane contactors based on porous hollow polypropylene fibers (porosity 25%, inside/outside fiber diameter 0.2/0.3 mm, pore size 20–30 nm [92]). The authors noted that the contact with the degraded absorbent led to wetting of membrane pores and to the solvent penetration into the gas phase, which deteriorates the oxygen mass transfer [90].

Apparently, the use of porous membranes in the case of deoxygenation of alkanolamine solvents is not appropriate,

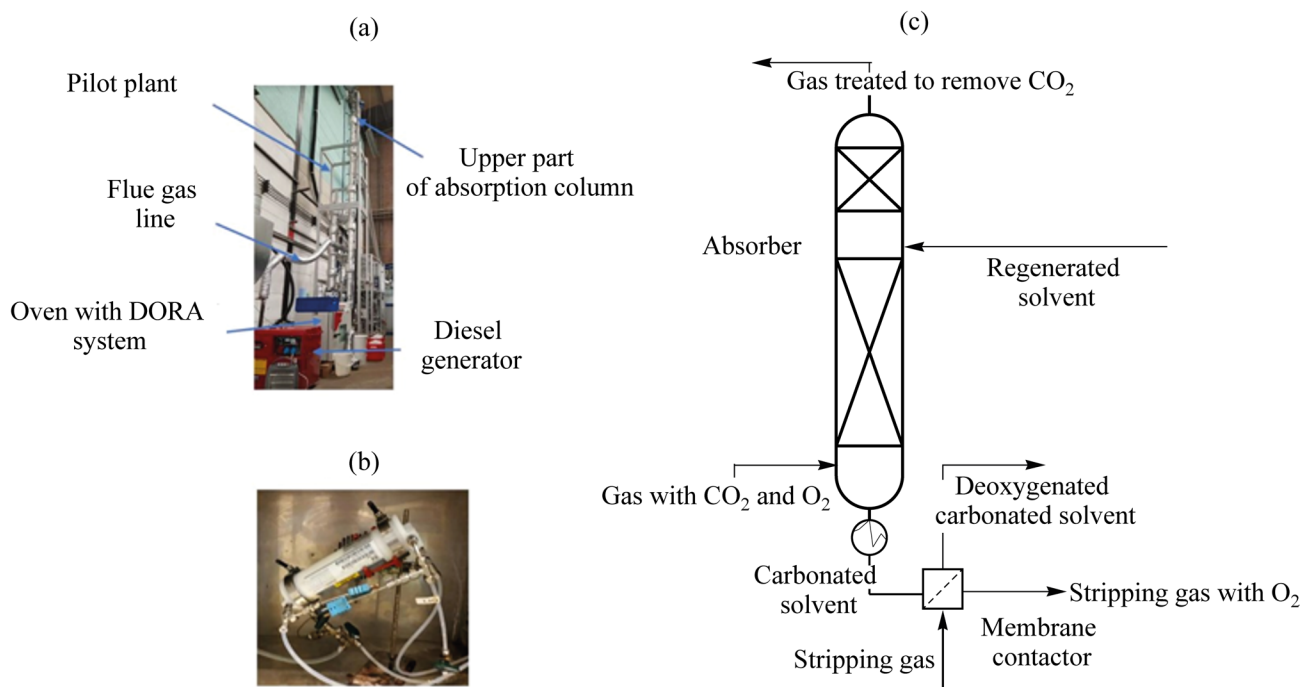


Fig. 2. Mobile pilot plant of TNO for CO₂ capture from simulated flue gases: (a) general view of the pilot plant for CO₂ removal from flue gases, (b) general view of the DORA membrane contactor-deoxygenizer, and (c) scheme of DORA deoxygenizer inclusion in carbonated solvent lines (adapted from [90]).

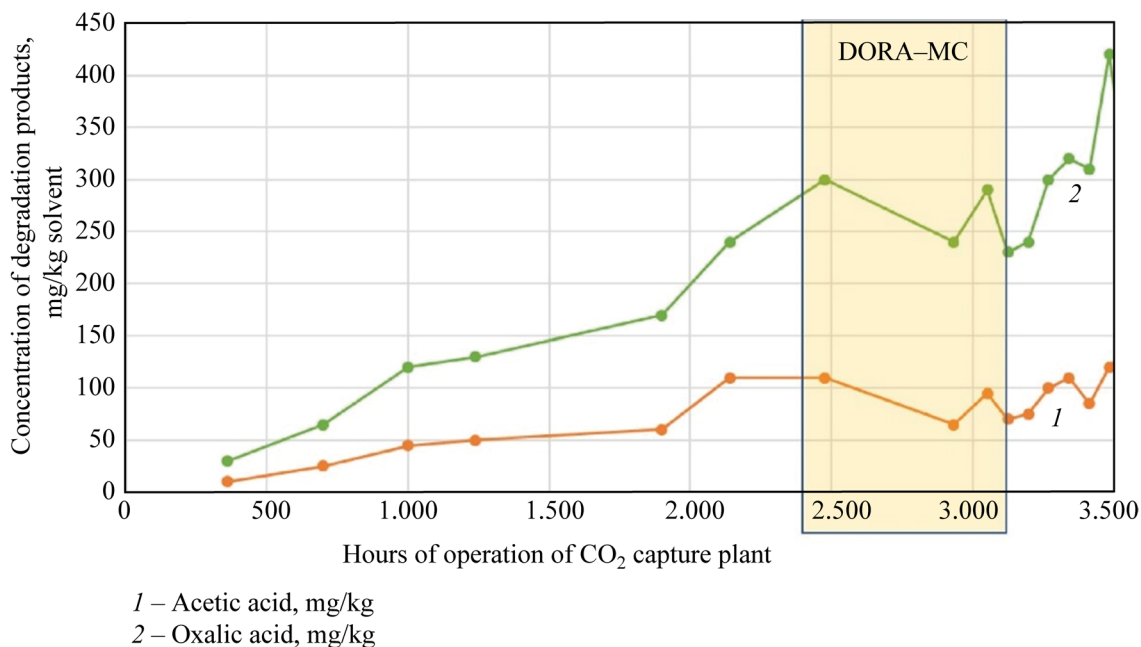


Fig. 3. Variation of the acetate and oxalate concentrations in the solvent of the pilot plant. The period of switching-on and operation of the DORA deoxygenizer is shown by a yellow rectangle (adapted from [90]).

because changes in the morphology of the membrane porous structure, resulting from penetration of amine molecules into the polymer matrix [93–96] and partial swelling of polypropylene [97], have been demonstrated in numerous studies. As a result, the membranes become wetted with the solvent, and the mass transfer becomes drastically deteriorated [98–100]. These membranes are unstable to the amine oxidative degradation products [101]: the presence of oxalic, acetic, and formic acids in the degraded solvent considerably reduces the CO₂ mass transfer rate into aqueous monoethanolamine solutions, which is probably associated with the adsorption of these acids on the surface of polypropylene membranes, making the membranes less hydrophobic. This leads to a 22–31% increase in the membrane pore wetting after 69-h operation of the membrane contactor [101]. Furthermore, there are data that even porous membranes made of the most chemically inert hydrophobic material, polytetrafluoroethylene, are also prone to wetting and to permeation of CO₂ solvents [102, 103]. Therefore, for deoxygenation of CO₂ absorbents, TNO researchers suggested composite membranes with thin selective layers of amorphous high free volume perfluorinated polymers, such as Teflon AF2400 [90, 91].

CONCLUSIONS

Degradation of alkanolamine CO₂ solvents is a factor that impedes further development of the amine absorption for post-combustion CO₂ capture. One of possible causes is the presence of oxygen, which oxidizes the amine, in the absorption liquid. The oxygen removal from solvents in membrane gas–liquid contactors is a very promising approach, as indicated by rich experience of their use to obtain deoxygenated ultrapure water. The few published papers on membrane deoxygenation of CO₂ solvents deal with porous hollow-fiber membrane contactors and report gradual wetting of membrane pores with the amine solution and deterioration of the oxygen mass transfer. Apparently, it is more appropriate to use composite membranes with thin nonporous highly permeable layers preventing the absorbent penetration into the porous structure of the membranes and wetting of the pore surface. Ideally, selective layers of such membranes should have maximal permeability to oxygen and minimal permeability to water and amine vapors. Both the selective layers and the support membranes used should have high chemical and morphological stability in degraded absorption liquids. Such membranes should

be additionally chemically and morphologically stable in aqueous solutions of liquid oxygen scavengers (e.g., aqueous solutions of sodium or ammonium sulfite) in the case of using their solutions in a membrane liquid–liquid contactor. By now, data on such membranes are virtually lacking in the available literature, and such membranes are to be developed.

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CONFLICT OF INTEREST

The author declare no conflict of interest requiring disclosure in this article.

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