

Reduction of amines emission and their volatile degradation products

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Introduction

In light of the decisions made by the European Commission with regard to reduction of greenhouse gas emission, what is becoming increasingly realistic is the application of the CO₂ capture technology at the existing coal-fired power units [1, 2]. One of the technologies already applied to absorb carbon dioxide is the amine absorption method, commonly used in the industry to remove CO₂ and other acid gases from the natural gas stream [3].

As the amine absorption method was improved through selection of new sorbents intended to increase the CO₂ removal efficiency and reduce the energy consumption involved in the process [4], more and more attention has been paid within the recent years to aspects connected with emission of amines and volatile products of their degradation (VDP) [5, 6, 7].

There is a sorbent (amine) loss problem occurring in the course of the system operation, triggered by thermal and oxidation-related degradation [8] as well as by physical phenomena of evaporation, liquid entrainment and mist formation. Specific compounds are formed due to sorbent degradation, reducing the latter's concentration, and furthermore, many of these substances are highly volatile, hence their escape from the system into the atmosphere along with the gas being purified, consequently leading to environment pollution [9].

The compounds formed as a result of amine degradation (VDP) vary in terms of volatility, and those having the highest importance for monoethanolamine (MEA) have been provided in Table 1. The degradation product emitted in the largest amounts is ammonia, formed as a result of oxidative MEA degradation [8]. Mertens's research [10] implies that after 1,000 hours of operation of a pilot system running on the MEA solution, the NH₃ concentration in purified gas increased nearly twofold. An increase in this concentration followed a proportional increase in the concentration of iron ions in the sorbent solution, contributing to accelerated sorbent degradation [11].

However, it is the emission of potentially carcinogenic nitrosamines and nitroamines that poses the largest threat, even though its volume is relatively low (0.5 ppb – 6 ppb/l ppm of MEA emission [6]). One may avoid the emission of these compounds by making appropriate selection of sorbents which do not form such stable bonds under conditions of the CO₂ capture process. Nevertheless, assuming specific conditions, atmospheric emission of amine particles only may trigger formation of toxic degradation products [12, 13]. Regulations aware that the sum of concentration of nitrosamines and nitramines should not exceed 0.3 ng/m³ for air concentrations and 4 ng/l for fresh water sources (Norwegian Climate and Pollution Agency, 2011) [14]. Nevertheless, the emission standard have not been established for MEA or the other likely amine solvent species [15].

Table 1

Approximate emission of products degradation of MEA [6]

| Degradation product | Maximum emission of compound in ppm per 1 ppm emission of MEA |
|---|---|
| The compounds of high volatility | |
| Ammonia | 1 |
| Methylamine | 0.006 |
| Formaldehyde | 0.08 |
| Acetaldehyde | 0.02 |
| Ethylamine | 0.01 |
| Diethylamine | 0.01 |
| N- nitrosodimethylamine | 0.02 |
| Dimethylnitroamine | 0.02 |
| Compounds with medium volatility | |
| Formamide | 0.007 |
| 2-oxazolidin | 0.004 |
| 1- (2-hydroxyethyl) -2-imidazolidinone | 0.003 |
| Oxamide | 0.003 |
| 2- methylaminoethanol | 0.01 |
| 1- (2-hydroxyethyl)imidazole | 0.03 |
| 2-(nitroamino)ethanol | 0.006 |
| N- nitrosodimethylamine | 0.0005 |
| The compounds of low volatility | |
| Formic acid | 0.003 |
| Acetic acid | 0.03 |
| Oxalic acid | 0.003 |

Emission mechanism

Emission of amine particles and products of their degradation may proceed through direct emission of vapours (evaporation), entrainment of liquid droplets along with the gas stream as well as mist formation.

Direct vapour emission related to sorbent evaporation depends on volatility of the amine used, the CO₂ lean loading and temperature in the absorption column. The amine vapour pressure and its evaporation increases as the temperature rises. Formation of carbamate and amine protonation reduces the sorbent volatility. Emission of this type may be easily reduced by application of a water washing column [6, 10].

Nguyen [9] reports that volatility of sorbents rises in the following sequence: MDEA(methyldiethanolamine)<DGA(diglycolamine)<PZ(piperazine)<2-MPZ(2-methylpiperazine)<MAPA(methylaminopropylamine)<EDA(ethylenediamine)<MEA<DAP(1,2-diaminopropane-)<1-MPZ(1-methylpiperazine)<AMP(2-amino-2-methylpropanol).

Vapour pressure over amine solution p_{Am} may be described with the following equation [6]:

$$p_{AM} = \gamma P_{Am}^0 (1 - 2\alpha)x_{MEA}$$

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where: P_{Am}^0 – vapour pressure of pure amine (Pa), α – CO₂ loading (CO₂ mole/amine mole), x_{MEA} – mole fraction of MEA in solution, γ – activity factor depending on the amine type and temperature. For a diluted MEA solution, the activity factor equals ca. 0.2 for 40°C and it increases up to 0.4 at 100°C. Values of vapour pressure for pure components may be found in the literature [9].

Entrainment of liquid droplets is a natural phenomenon occurring wherever there is contact between gas and liquid. Inside the absorption column, the gas flowing from the bottom column section comes into contact with the liquid flowing down from the top. Under perfect conditions of the filling material spraying, the liquid forms a thin and smooth film, however, in reality the liquid film is subject to disruption as a consequence of the gravitational force impact, thus triggering formation of droplets. Intense formation of droplets also occurs in liquid distributors [6].

The amount of droplets entrained is a function of the column loading with a stream of gas and liquid. Increasing the gas flow rate triggers an increase in the amount of liquid suspended on the bed and contributes to formation of droplets. Raising the flow of liquid increases the film thickness as well as the extent of separation of liquid droplets and streams. Therefore it is so important to establish optimum operating conditions for the column. In order to reduce the emission of droplets, demisters are used, ultimately removing droplets with the diameter $> 10 \mu\text{m}$ [16, 17].

The most undesirable phenomenon related to amine emission is the formation of mist, i.e. suspension of liquid droplets in gas. On account of the small size of the liquid particles (with diameter of several μm), they permeate through water wash sections and traditional demisters.

The prerequisite of mist formation is a state of oversaturation, i.e. a situation when gas, at the given temperature and pressure, contains more moisture than in the state of equilibrium. This state is unstable and, under appropriate conditions, excess of the liquid present in gas is subject to outdropping, which leads to formation of mist.

Nucleation, i.e. formation of the first droplets, may be homogeneous, when condensation nuclei are only formed from the gas component being condensed, or heterogeneous, in which case there are also other particles contributing to the nucleation. Compared to homogeneous nucleation, heterogeneous nucleation occurs already in states of small oversaturation of the system [6, 18].

Heterogeneous nucleation takes place particularly in industrial processes, since process gases contain multiple different substances of nanometric dimensions, on surfaces of which the first droplets may condense. Kamiyo [19] established that already small amounts of SO₃ present in flue gas, i.e. on the level of 3 ppm, act as condensation nuclei forming mist, and consequently increasing the MEA emission from 0.8 to 68 ppm. Khakharia [20] also found that the presence of soot, ash and H₂SO₄ aerosol exerts major impact on the amine emission increase in the CO₂ removal process using amine scrubbing. Since the flue gas composition changes on ongoing basis, it is extremely difficult to estimate the emission of amine which has taken the form of mist.

In an amine-based CO₂ removal system, formation of mist takes place primarily in the top part of the absorber where heated gas saturated with moisture comes into contact with cold sorbent [18, 21]. Emission of amine in the form of aerosol increases as the temperature difference between the warmest and the coldest bed section rises. Therefore, the warmer the amine solution flowing into the absorber, the lower the aerosol emission, however, the vapour emission rises. Nevertheless, the research implies that, in this particular case, the total emission value is lower [7].

Mertens [10] emphasises that the mist formation risk also occurs near liquid distributors and in the water washing column. He also claims that ca. 50% of the ethanolamine present in purified gas occurs in the form of mist, whereas the remaining part assumes the form of vapour.

Using the water wash column (section) contributes to reduction of nearly a half of the total amount of the MEA emitted, since only the gaseous form of ethanolamine is removed in the water wash. Submicronic MEA particles forming mist pass through demisters and water wash columns which hinders the gas post-treatment from the perspective of removal of these types of contaminants. Khakharia et al. [16] claim that a water wash and a knitted-mesh demister are not fully efficient in aerosol removal, since the droplet diameter is typically 0.04-4 μm .

Apparatus used for emission reduction

The water wash column

Using the water wash section at the absorber top is one of fundamental and most popular methods of reducing amine emission [10, 12, 22]. In the water wash, vapours of amine and VDP contact with the cool water stream in the counter-current, where they become condensed and absorbed. Water is injected into the apparatus by means of a sprinkler and delivered to the structural packing (e.g. Mellapak 250Y manufactured by Sulzer Chemtech [23]) which ensures appropriate area of contact between gas and liquid [24, 6].

It is for the water balance configuration and economic aspects that one cannot use large quantities of fresh water, hence the application of recirculation. Fresh water accounts for only a small part of the recirculating stream. After being saturated with amines, part of the circulating water is transferred to the absorption column as replenishment for the evaporated water.

In order to reduce the circulating water temperature, coolers are installed in the liquid circuit. Temperature of the water used in the water wash section should be ca. 5-10°C lower than gas temperature [16]. This temperature reduction increases the absorption of amines and VDP as well as contributes to outdropping of the moisture part contained in gas. The part of water outdropped from gas constitutes the fresh water fraction in the recirculation system.

Unfortunately, the circulating water stream causes that the process driving force is inconsiderable, since the amine concentration is almost equal at the top of the bed section as well as under it. For the sake of the foregoing, one often uses more than a single water wash section [6]. It is in that case that fresh water is delivered to the highest section, and then water from the first water washing section is delivered to the section located below. Also the aforementioned water cooling is aimed at increasing the driving force.

Standard height equivalent to theoretical plate (HETP) for the Mellapak 250Y Sulzer Chemtech packing is 0.7 m. Laboratory studies [6] prove that using the height of packing 1.5 m, under conditions of equilibrium, makes it possible to attain the capture efficiency of 99% of the compounds emitted.

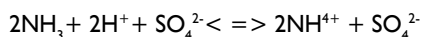
Kolderup [6] conducted model studies for a plant capturing ca. 73 t of CO₂/h (90% efficiency of CO₂ capture). Disregarding the phenomenon of entrainment and mist formation, the MEA emission before the water wash section came to ca. 155 ppm. Once two water wash sections had been used, the emission dropped to 0.6 ppm (99.6% efficiency). After the first water wash section, the MEA content was 9.5 ppm (93.8% efficiency). Using three water wash sections allowed for the MEA emission to be reduced to less than 1 ppb (99.999% efficiency)

The acid wash columns

For more volatile compounds, it is recommended that acidic solution be used instead of water. Reducing pH of the solution contributes to far more efficient removal of basic components, and these include amines and numerous compounds formed in the process of their decomposition (methylamine, ethylamine). Using acidic solution is particularly recommended for removal of volatile ammonia,

since water in a traditional water washing section quickly becomes saturated with ammonia, and hence the very low removal efficiency for this compound [12].

One may notice that pH is maintained on the level of 3 – 4 [6], particularly for the removal of NH_3 . The dissolution equilibrium of NH_3 in acidic solution depends on temperature, concentration and pH:



In order to maintain appropriate pH, the solution of H_2SO_4 and a pump system dosing can be used. In studies conducted on the pilot plant (flow rate of flue gas 1,000 Nm^3/h , 200 kg/h of captured CO_2), Knudsen [12] used 10% solution of sulphuric acid, thus attaining reduction of the ammonia concentration from 8 ppm to 1 ppm as well as of the emission of alkylamines (ethylamine, methylamine, diethylamine and dimethylamine) by ca. 93 – 94%.

Demisters

Demisters are specific gas/liquid type separators used for capturing and outdropping of droplets from gas. One of the most popular demisters is the knitted-mesh type. These separators are successfully used in amine gas treating plants [16], [6], [22].

Demisters of this type assume the form of knitted mesh, usually made of stainless steel wire 0.152 mm or 0.279 mm in size. They may also be made of glass or polyester fibre with the core diameter of 0.01 mm or of plastics (PE, PP, PTFE) with the thread thickness of 0.40–0.66 mm. It is a relatively cheap solution, it is easy to apply and ensures high efficiency of droplet separation. The apparatus types discussed allow for efficiency of >99% to be obtained for droplets > 10 μm in diameter. Unfortunately, they do not prove equally effective in separation of droplets with smaller diameters.

In order to ensure successful operation of a demister, high gas flow rates are applied, since as the rate of the gas flow increases, the diameter of particles capable of passing through a demister declines. The recommended gas flow rate (for vertical arrangement) comes to 3.1–4.6 m/s. Demisters of this type feature ca. 98 – 99% of free space, hence their low flow resistance, and so the pressure drop is lower than 250 Pa. Standard thicknesses of demisters are as follows: 25, 50, 100, 150 and 200 mm [17].

In an amine-based CO_2 removal system, one may also use mesh made of glass fibre which is hydrophilic in nature. Owing to the foregoing solution, amine particles, being hydrophilic themselves, may be captured more efficiently [6].

Filter bed demisters

High efficiency of removal of even very small droplets, even those of 0.1 μm in diameter, can be achieved using a filter bed demister. This demister consists of a number of filters referred to as candle filters. They are cylinders, usually 1 m in length and 0.5 m in internal diameter. A filter of this type is composed of millions of interwoven fibres, and although the efficiency of a single filter is not impressive, each apparatus comprises a number of those, and their combined capability ensures good efficiency of aerosol particle removal reaching 99% [16].

It performs the droplet capture function by operating on the diffusion mechanism basis, meaning that, as an effect of chaotic thermal motions, a particle may stop while flowing next to a fibre. On account of the foregoing, for the sake of correct operation of a demister of this type, the gas flow rate must be rather small. This apparatus is mainly intended for removal of aerosol particles with the diameter smaller than 2 μm [16].

According to studies conducted at a pilot plant, this filter triggers reduction of the MEA emission from 160 mg/m^3 (ca. 59 ppm) to 1.2 mg/m^3 (0.45 ppm). The filter is installed downstream the water wash section featuring a knitted mesh type demister. Using a combination

of devices, the MEA emission could be successfully reduced from 408 mg/m^3 (ca. 150 ppm) to 1.2 mg/m^3 (0.4 ppm). In the case discussed, the water washing section efficiency came to 41%, whereas that of the BDU demister – 99.2% [16].

Khakharia [16] also studied the BDU (Brownian Demister Unit) efficiency in the function of ammonia and nitrosamine removal. And as expected, this apparatus did not contribute to reduction of emission of the compounds studied, which has only confirmed the fact that these compounds occur in the vapour form and not as droplets, and consequently they are not removed.

A disadvantage of this apparatus type is the high pressure drop on a very low gas flow rate (compared to a knitted mesh demister). For the gas flow rate of 0.13 m/s, Khakhira [16] claims the pressure drop to equal 5,000 Pa. For the sake of comparison, for a mesh type demister with the density of 145 kg/m^3 , with the gas flow rate of 4 m/s, the pressure drop does not exceed 150 Pa [25].

The high pressure drop in the case of the BDU demisters contributes to a necessity of incurring higher operating costs due to the fan's increased energy expenditure. As reported in publication [16], in this case, the electric energy cost (€/tonne of CO_2 captured) will rise by 26% or even by as much as 52% on the pressure drop by 10 kPa. Nevertheless, in terms of removal efficiency for sub-microscopic aerosol particles, this demister proves unmatched.

Conclusions

In systems based on amine absorption methods, sorbent degradation and physical phenomena, such as evaporation, liquid entrainment or mist formation, are the reasons for atmospheric emissions of harmful substances. Bearing the impact of various process parameters in mind, it is difficult to accurately establish the emission value (Tab. 2). Ammonia, being the main degradation product, constitutes the main emission source due to its volatility. Further amine degradation products of high volatility include formaldehyde, acetaldehyde, ethylamine and diethylamine. On the other hand, on account of their toxicity, there are also particularly hazardous products being formed in the degradation process, namely nitrosamine and nitroamine, even though their emission volume is negligible (i.e. on the level of ppb).

Amine, being the sorbent component, constitutes the main emission component next to degradation products. The rate of amine evaporation increases as the temperature rises and it depends on the volatility of the amine used, whereas it declines as the CO_2 lean loading grows. Entrainment of droplets and formation of mist are natural phenomena occurring in areas where gas comes into contact with liquid. The substances present in flue gas, such as soot, ash, sulphur oxides, being the condensation nuclei, contribute greatly to the mist formation, thus increasing the amine aerosol emission.

In order to reduce the emission, one applies apparatus-based solutions. Water wash sections featuring structural packing and cooling water circulation are used to remove vapours of ammonia, amines and VDP. The ammonia removal efficiency can be increase further on by application of an acid-spray column. Ultraviolet radiation, on the other hand, may be successfully used to remove nitrosamines [6, 13].

Downstream this gas treatment stage, the emission mainly comprises amine in the form of droplets and mist. Research results imply that emission in droplets and mist accounts for at least 50% of the total emission value, and hence the necessity of using demisters. What proves an efficient solution for removal of droplets with the diameter of $\geq 10 \mu\text{m}$ is the knitted-mesh type of demisters, whereas for smaller droplets forming mist, one must use solutions like demisters featuring candle filters. Such demisters allow for reduction of the droplet emission by even as much as 99%. Using a flue gas pre-treatment unit (flue gas desulphurisation, cooling and dust removal) is also a means to reduce emission considerably owing to elimination of condensation nuclei [26, 22].

Amine emission from the CO₂ capture plants using amine scrubbing

| Source | Absorbent | Type of study | Tested parameters and apparatus | Emission* |
|--------|--------------|--|---|--|
| [13] | PZ | Tarong Capture Pilot Plant, Australia (600 kg/h) | Influence of temperature of regeneration to concentration of mononitrosopiperazine (MNPZ) in solvent. | PZ :0.48-1.09 ppm MNPZ: 0.01-0.15 ppb (based on Henry's law) MNPZ in sorbent: 2.5-7.5 mmol/kg |
| [20] | MEA | TNO mini plant mobile, Netherland (4 Nm ³ /h) | The influence of dust, soot and H ₂ SO ₄ aerosol. | MEA: 45 mg/Nm ³ MEA:200 mg/Nm ³ (soot in flue gas) MEA: 600-1100 mg/Nm ³ (presence of H ₂ SO ₄) |
| [19] | MEA | (1 tpd CO ₂) Pilot plant, MHI, Japan | The influence of SO ₃ aerosol. | MEA: 0.8 ppm (without SO ₃) MEA: 67.5 ppm (3ppm SO ₃) |
| [18] | MEA | Modeling study (616 kg/s) | Absorption temperature and content of CO ₂ . | MEA: 100-650 mg/Nm ³ (vapour emission) MEA: 40-240 mg/Nm ³ (aerosol emission) |
| [6] | MEA, AMP/PZ | Modeling study (1.2 mln Nm ³ /h) | Temperature of flue gas and CO ₂ loading of lean amine. Efficiency of 1-3 water wash sections. | Not including liquid entrainment. MEA: 43-155 ppm, After 2 WW section: <0.1 ppm AMP+PZ: 217-554 ppm After 2 WW section: 0.55-30 ppm |
| [7] | MEA, AMP/PZ | TNO mini plant mobile, Netherland (4 Nm ³ /h) | Temperature and pH of lean solvent temperature in absorber, H ₂ SO ₄ aerosol, CO ₂ content in flue gas. | Aerosol based emission only: MEA: 1200-1900 mg/Nm ³ AMP:100-2300 mg/Nm ³ PZ: 0-350 mg/Nm ³ |
| [5] | MEA | CO ₂ capture plant at the Maasvlakte coal power plant (1500 Nm ³ /h) | Efficiency of Brownian demister unit and water wash. | MEA emission (in mg/Nm ³): Before WW: 372 After WW and demister: 219 In BDU: 152 Out BDU: 2 Nitrosamines and nitramines: 5-47 ng/m ³ |
| [24] | MEA, AMP/PZ | PCCC Pilot Plant Esbjerg, Denmark (5000 Nm ³ /h) | The influence of temperature, flow rate of water in WW, CO ₂ content in flue gas. Correlation between O ₂ and NH ₃ emission and between NH ₃ and MEA. Efficiency of Water wash. | No absolute emission numbers are presented. |
| [16] | MEA | TNO Pilot Plant at Maasvlakte, 1500 Nm ³ /h | Efficiency of Brownian demister unit (BDU) and water wash. | Before WW: 460 mg/Nm ³ After WW: 250 mg/Nm ³ After BDU: 2.1 mg/Nm ³ NH ₃ emission after WW: 10-70 mg/m ³ N. |
| [12] | MEA, | Aker Clean Carbon, MTU, USA (1000 Nm ³ /h) | Efficiency Aker Clean Carbon's Anti-mist design (AMD) and acid wash. | Before AW NH ₃ : 4-8 ppm After AW: NH ₃ < 1 ppm MEA: 20-40 ppm before AMD After AMD: MEA < 1 ppm Alkyloamines (before/after AW): 0.003-0.006 mg/Nm ³ / <0.0004-0.002 mg/Nm ³ |
| [22] | BASF solvent | Pilot Plant at Niederaussen, Germany (1550 Nm ³ /h) | Dust and particle concentration in flue gas. Water wash temperature, pH acid wash, intercooler temperature, voltage of wet electrostatic precipitator. Dry bed configuration and gas pre-treatment efficiency. | Aerosol formation – amine concentration in lean flue gas. No absolute emission numbers are presented. |
| [21] | MEA | TNO mini plant mobile, Netherland (4 Nm ³ /h) | Number concentration and size of aerosols in flue gas. | NH ₃ : 20 mg/m ³ MEA: 3000 mg/m ³ (presence of H ₂ SO ₄) 45 mg/m ³ MEA (absence of H ₂ SO ₄) |

*ppm – 1 parts per million, (ppm = mg/Nm³·22.42/Molecular weight); mg/Nm³ – milligram per normal cubic meter; WW – water wash; AW – acid wash; AMD – Aker Clean Carbon's Anti-mist design; BDU – Brownian demister unit.

On account of the nature of the emission of amines and their degradation products, post-combustion carbon capture (PCCC) systems require application of several separation techniques in order that the gas purification degree attained is sufficiently high. Solutions such as those addressed in the paper will contribute to considerable emission reduction, however, since additional apparatus must be used, thus increasing flow resistances, these technologies will cause further energy consumption which must be taken into consideration in amine treating plants design.

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RYNEK

LANXESS i Saudi Aramco tworzą joint venture

Koncern LANXESS, producent specjalistycznych środków chemicznych, oraz koncern Saudi Aramco planują ustanowienie spółki joint venture zajmującej się produkcją kauczuków syntetycznych. Koncern LANXESS oraz Aramco Overseas Company – podmiot zależny koncernu Saudi Aramco – będą posiadać po 50% udziałów

w nowej spółce działającej w sektorze, w którym koncern LANXESS w 2014 r. uzyskał ok. 3 mld EUR dochodu. Koncern Saudi Aramco zapłaci ok. 1,2 mld EUR w gotówce za swój 50% udział w spółce po odjęciu zadłużenia i innych zobowiązań finansowych. Wspólne przedsięwzięcie wyceniane jest na całkowitą kwotę 2,75 mld EUR. Transakcja nadal wymaga uzyskania zgody właściwych organów ds. ochrony konkurencji, oczekuje się, że nastąpi to w pierwszej połowie 2016 roku. (kk)

(Komunikat prasowy Lanxess, 24.09.2015)

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