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Nitrosamines and nitramines in Carbon Capture plants

Nitrozoaminy i nitroaminy w instalacjach wychwytu ditlenku węgla

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Abstract
The use of amine absorption in carbon capture technologies is related with the formation toxic and potentially carcinogenic amine degradation products such as nitrosamines and nitramines. These substances can be created within both the solvent and the atmosphere when air components (mainly NOx) and amines react with each other. These substances may pose environmental and health risks depending on the level and duration of the exposure. In this paper, formation and occurrence of nitrosamines and nitramines in carbon capture plants were described. Emission reducing technologies have been also mentioned. Furthermore, an overview of experimental data of emission of nitrosamines and other major degradation products has been pointed out.

Streszczenie
Zastosowanie absorpcji aminowej do wychwytu ditlenku węgla związane jest z tworzeniem się toksycznych i potencjalnie rakotwórczych produktów degradacji amin, takich jak nitrozoaminy i nitroaminy. Produkty te mogą powstawać w roztworze absorpcyjnym jak i tworzyć się w atmosferze w wyniku reakcji emitowanych amin ze składnikami atmosfery (głównie NOx). W zależności od stężenia substancji toksycznych, mogą one stanowić zagrożenie dla środowiska i tym samym dla zdrowia ludzi. W artykule opisano problematykę występowania nitrozo- i nitroamin w instalacjach wychwytu CO2 (carbon capture) stosujących absorbenty aminowe. Ponadto opisano metody redukcji emisji amin i produktów ich degradacji, a także przedstawiono eksperymentalne wartości emisji tych związków, dostępne w światowej literaturze.

1. INTRODUCTION

Owing to changing climate and the European Union CO_2 emission regulations (i.e. reduce emission of greenhouse gases by 20% by 2020 and by 40% by 2030 compared with 1990), employment of carbon dioxide capture from flue gases is becoming increasingly feasible [Więcław-Solny et al., 2012b, Więcław-Solny et al., 2012a]. The chemical absorption using amine solvents such as monoethanolamine (MEA), N-methyldiethanolamine, diethanolamine (DEA), 2-amine-2-methylpropanol (AMP) or piperazine (PZ) (Wilk et al., 2017) is a well-known and mature technology for CO_2 capture.

The concept of a demo plant capturing 1.5 million tonnes of CO_2 annually from flue gas drawn from the coal-fired boiler was presented in a paper [Tatarczuk et al., 2016]. The use of amine absorption technology that contributes to the mitigation of greenhouse gases, however, is related with the emission of amine and its degradation products into the atmosphere [Spietz et al., 2015].

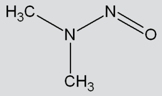
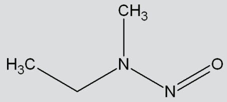
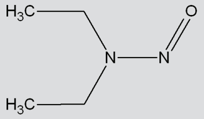
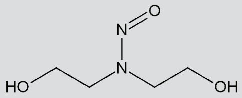
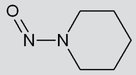
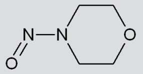
In amine-base carbon capture plant, losses of amines result from physical phenomena and solvent degradation. Solvent (MEA) losses are approximately 0.01–0.8 kg/tonnes of CO_2 removed (without water wash) or 0.01–0.03 kg/tonnes of CO_2 removed (with water wash systems) [Karl et al., 2011]. Physical phenomena responsible for amine losses include evaporation, droplet entrainment and aerosol (mist) formation. The effect of these on emissions has been described in detail in papers [da Silva et al., 2013, Spietz et al., 2015, Khakharia et al., 2014].

As a result of chemical reaction of amines with oxygen and other compounds present in the flue gas (oxidative degradation) and because of high regeneration temperature of the solvent (thermal degradation), degradation products are formed. Owing to their volatility, they have negative effects on the environment in and around the area of the carbon capture plant [Helgesen and Gjernes, 2016]. Furthermore, they cause operating problems such as corrosion and fouling and thus increase the operating cost [Khakharia, 2015]. Ammonia is very volatile, primary degradation product produced as a result of oxidative degradation of amines. Other volatile degradation products include acetaldehyde, acetone, ethylamine (EA), methylamine (MA), formaldehyde, diethylamine and acetic acid [Strazisar et al., 2003, Gouedard et al., 2012]. In turn, thermal degradation contributes to the formation of complex molecules such as N-(2-hydroxyethyl) ethylenediamine, N-(2-hydroxyethyl)-2-imidazolidone and thermally stable salts that decrease the concentration of the solvent [Gouedard et al., 2012].

2. TOXICITY OF NITROSAMINES AND NITRAMINES

The amine used as a solvent (in amine scrubbing technology) can be emitted to the atmosphere where it react with hydroxyl radical (*OH), ozone and nitric oxides presented in the air, producing nitrosamines and nitramines. Aliphatic amines, during the day, undergo a very rapid reaction with the hydroxyl radical

Table 1. IARC classification of nitrosamines formed in Carbon Capture plants.

IARC classification (IUR)*, NSRL** µg/day	Compound acronym and CAS	Chemical formula	Structure
Group 2A (4.6×10^{-3}) 0.04	NDMA (62-75-9)	$C_2H_6N_2O$	
Group 2B (6.3×10^{-3}) 0.03	NMEA (10595-95-6)	$C_3H_8N_2O$	
Group 2A (1.0×10^{-2}) 0.02	NDEA (55-18-5)	$C_4H_{10}N_2O$	
Group 2B (8.0×10^{-4}) 0.3	NDELA (1116-54-7)	$C_4H_{10}N_2O_3$	
Group 2B (2.7×10^{-3}) 0.07	NPIP (100-75-4)	$C_5H_{10}N_2O$	
Group 2B (1.9×10^{-3}) 0.1	NMor (59-89-2)	$C_4H_8N_2O_2$	

*IUR, inhalation unit risk $(\mu g/m^3)^{-1}$; NSRL**, No significant risk level according to OEHHA (Office of Environmental Health Hazard Assessment)

(photolysis); hydrogen is abstracted from either carbon or nitrogen atom and amino radicals are formed. During the night, ozone and NO_2 cause further amine degradation what leads to the formation of different kinds of compounds. The main products of atmospheric degradation are amides, aldehydes as well as nitrosamines and nitramines [Karl, 2010, Shao et al., 2009].

Nitrosamines and nitramines, although their emission is very low (2–150 ng/m^3 [Kolderup et al., 2011]; 2–50 ng/m^3 [Ravnum et al., 2014]), should be taken into consideration because of their potential carcinogenic and mutagenic properties [Lin, 1990, Tricker and Preussmann, 1991, U.S. EPA, 2017]. N-nitrosamines are formed primarily by the reaction of secondary and tertiary amines with nitrosonium cation NO^+ (from nitrous acid). Primary amines do not form stable nitrosamines. Nevertheless, primary amines as a result of degradation processes may form secondary amines (e.g. MEA degrades into DEA) [Fostås et al., 2011]. Nitrosation of DEA leads to the formation of nitrosodiethanolamine (NDELA) [Silva et al., 2013].

Nitramines can be formed from primary, secondary and tertiary amines and can be stable in sunlight. Nitrogen oxides presented in process gases (NO, NO_2, N_2O_3, N_2O_4), react with amines in the solvent and form both nitramines and nitrosamines. The study [Mitch, 2002] shows that N_2O_3 can limit the rate of nitrosation of secondary amines. In addition, the NO_2^+ ion formed in the solution, in the presence of formaldehyde as a catalyst, may react with amines to form nitramines [Silva et al., 2013]. As previously mentioned, the formaldehyde is one of the main degradation products of amines.

Finally, the emission of the solvent from the amine scrubber plants indirectly affects the concentration of nitramines and nitrosamines in the atmosphere. Some of the detected nitrosamines and nitramines in carbon capture system are N-nitrosodimethylamine (NDMA), N-nitrosodiethylamine (NDEA), NDELA, N-nitrosomorpholine (NMor), N-nitrosopiperidine (NPIP), mononitrosopiperazine (MNPZ), dinitrosopiperazine, N-methylnitramine (MNA) and N-dimethylnitramine (DMNA) [Ravnum et al., 2014, Hong et al., 2017]. The IARC (International Agency for Research on Cancer) carcinogenicity classification of nitrosamines [IARC, 1978, 2017] and the cancer risk factors [OEHHA, 2017, U.S. EPA, 2017] have been shown in Table 1.

IARC coordinates and conducts both epidemiological and laboratory research into the causes of human cancer. National health agencies can use this information as scientific support for their actions to prevent exposure to potential carcinogens. The IARC Monographs identify environmental factors that can increase the risk of human cancer. These include chemicals, complex mixtures, occupational exposures, physical agents, biological agents and lifestyle factors. They can be classified as group 1, carcinogenic to humans; group 2A, probably carcinogenic to humans; group 2B, possibly carcinogenic to humans; group 3, not classifiable as to its carcinogenicity to humans; group 4, probably not carcinogenic to humans [IARC, 2017].

According to the Norwegian Institute of Public Health (NIPH), amine compounds are not mutagenic in most cases, but with further reactions in the human body and within the atmosphere, they can form compounds of greater toxicity [Låg et al., 2011]. The WHO (World Health Organization), Health Canada and U.S. EPA (United States Environmental Protection Agency) estimated a cancer risk for the exposure to NDMA in drinking water, based on the extensive study [Peto et al., 1991]. In regard to these studies and inhalation study [Klein et al., 1991], the NIPH estimated the admissible levels of nitrosamines and nitramines associated with negligible risk level for cancer (i.e. 10^{-6}, means there is a risk of one additional occurrence of cancer in one million people, at the given exposure over a lifetime). Permissible total concentrations of nitrosamines and nitramines proposed by NIPH are 0.3 ng/m^3 in air and 4 ng/l in drinking water. According to WHO, Health Canada and U.S. EPA, the NDMA limit in drinking water are 100 ng/l (risk 10^{-5}), 4 ng/l (risk10^{-6}) and 0.7 ng/l (risk10^{-6}), respectively [Låg et al., 2011].

In contrast to nitrosamines, data on chronic toxicity of aliphatic nitramines are very limited and there is not sufficient toxicological information for a proper evaluation of their health hazard. Although nitramines are less mutagenic and carcinogenic than their corresponding nitrosamines, they should also be considered as highly toxic [Låg et al., 2011]. DMNA, N-diethylnitramine (DENA) and MNA should still be regarded as carcinogen of high potency. Many research on nitramines have shown their carcinogenic potential in animals [Pliss et al., 1982, Goodall and Kennedy, 1976, Scherf et al., 1989]. The studies [Fjellsbø et al., 2014] confirm the toxicity of some nitramines. Their results exhibited that amongst MEA-NO_2, 2-nitramine-2-methylpropanol and nitropiperazine, only MEA-NO_2 showed positive mutagenic effect. The other two nitramines were found not to be mutagenic. In turn, mutagenic potential of DMNA was not confirmed.

3. EMISSIONS IN CARBON CAPTURE PLANTS

During the carbon dioxide capture process, it is expected that a small proportion of amine solvent and their degradation products escape from the absorber and are released to the atmosphere together with the cleaned flue gas. Moreover, these compounds have a tendency for adsorption to soil and thus may cause damage to the soil dwelling organisms or in case of mobility to groundwater, they may contaminate the sources of drinking water [Sørensen, 2013]. A review of available studies indicated that different kinds of products are formed [Shao et al., 2009, Berglen et al., 2010, Brakstad et al., 2010]. Some published data on nitrosamines and nitramines emissions refer to measurements near or below the detection limit. Few studies concern quantitative determination of nitramines. Table 2 shows the experimental data of nitrosamine emission and major amine degradation products measured at pilot plants.

At the Loy Yang pilot plant (Australia), emission of ammonia is so high because of intense degradation of the MEA. The measured concentrations of other ions were at the following level: formate 1,485 mg/l; oxalate 380 mg/l, nitrate 660 mg/l and propionic acid 800 mg/l [Azzi et al., 2014]. However, DEA, dimethylamine (DMA), methylamine and ethylamine were not detected in the treated gas. At the Maasvlakte pilot plant (the Netherlands), the concentrations of nitrosamines are in some cases higher behind BDU (brownian demister unit) than before it. Researchers suspect that these compounds may be additionally formed on the wetted surface of the filter. As can be seen from Table 2, the most commonly measured MEA degradation products are ammonia, formaldehyde and acetaldehyde. The concentration of nitrosamines in the purified gas is significantly lower than 1 μg/m^3 (except in one case –CCPilot100+, the UK). In the solvent, the content of these compounds is much higher (NDELA: 2–5 mg/l).

4. THE TECHNOLOGY OF EMISSIONS REDUCTION

Reducing amines and their degradation products can be achieved either by removing them by emissions reducing systems or by limiting amine degradation (by appropriate process parameters, solvent selection, etc.) [Hoff et al., 2013].

Most amines used in carbon capture systems and amine degradation products are water soluble [Shao et al., 2009]; hence the water wash systems are widely used. Additionally, acid aqueous solution or addition of strong oxidants such as potassium permanganate may be used [Goetheer and Da Silva, 2012]. Knitted wire mesh, vane demisters, Brownian demister unit, wet electrostatic precipitators, multicyclons, adsorbers and condensers are also used [IEAGHG, 2012, Mertens et al., 2014, Khakharia et al., 2014, Knudsen et al., 2013].

In regard to research [Goetheer and Da Silva, 2012], nitrosamines are present in the vapour phase. Typical options for lowering the emission would be using additional water wash steps. Because nitrosamines do not have an alkaline nature, an acid wash would not be so effective as in case of ammonia and amines reduction. UV irradiation can be used to remove nitrosamines in the process liquids [IEAGHG, 2012]. However, the decomposition of these compounds is heavily affected by the colour of the solution and amine concentration. An increase in amine concentration decreases the nitrosamines decomposition [Knuutila et al., 2013]. Nonetheless, UV irradiation can be used to limit the concentration of NDMA and NDELA in process wastes [Plumlee et al., 2008] as well as in the water wash sections [Knuutila et al., 2013]. It has been observed that the acidic solution favours UV absorption and thereby the decomposition of nitrosamines [Xu et al., 2009b]. According to studies [Shah et al., 2013], the use of both UV irradiation and O_3 can lead to the removal of about 90% of all N-nitrosamines. On the other hand, findings of the research [Lee et al., 2007, Xu et al., 2009a] shown that ozone was not effective in destroying NDMA – only 12% is removed at neutral pH, but using the mixture of O_3/H_2O_2 may lead to removal of up to 80% of NDMA within 5 min. Nonetheless, under these conditions, MA is the main product of NMDA decomposition [Lee et al., 2007]. The use of UV/O_3 method leads mostly to the formation of NO_3^-, whereas UV radiation leads to the formation of DMA. Thus, the latter may be more favourable for reducing nitrosamine.

The use of biological methods is associated with long reaction times. Moreover, nitrosamines can be converted to nitroamines [Brakstad et al., 2011].

Furthermore, it should be noted that photolysis is a natural phenomenon that limits these compounds in the atmosphere. On the sunny day, owing to the *OH radicals presented in the atmosphere, nitrosamines are rapidly degraded. The average lifetime of compounds in the atmosphere is as follows: NDMA, <1 h; DMNA, 2 days (decomposition by photolysis is negligible); NDEA, DENA and MEA-NO_2 decompose within 1 day, formaldehyde, several hours; acetaldehyde less than 1 day [Ge et al., 2011].

Table 2. An overview of nitrosamine and other major degradation products found in carbon capture plants.

Source of emission	Concentration of nitramines, nitrosamines and other major degradation products	Other useful information
Loy Yang carbon capture pilot plant at power station in Latrobe Valley, Victoria. (Azzi et al., 2014)	Absorber outlet: NMor: 0.32; 0.04* μg/m^3_N NDMA, NDEA, NDELA: nd NH_3: 250 mg/m^3_N; MEA: 311; <0.3* mg/m^3_N Acetaldehyde: 1 mg/m^3_N In the solvent: NMor 16; 6.4^W μg/l; NDELA 2.8 mg/l; nd^W	Captures 50 kg CO_2/h (flue gas rate: 200 m^3_N/h). Solvent: 30% MEA, flue gas drawn from a brown coal-fired power plant, without $DeNO_x$ and FGD.
Carbon capture pilot plant at the Maasvlakte coal power plant in Netherlands. (Kolderup et al., 2012, Khakharia, 2015)	Absorber outlet: NDELA: 11–47*; 16–20^B ng/m^3_N NDMA: 7–10*; 6,5–11^B ng/m^3_N NMor: 18*; 3 ng/m_N^3 MEA: 372; 219*; 1–4^B mg/m^3_N NH_3: 13–70^B mg/m^3_N In the solvent: NDELA 2.0 mg/l; MEA-NO_2: 1.40–1.85 mg/l MEA-NO_2^{W}: <5 μg/l	Captures max. 250 kg CO_2/h. Flue gas flow rate 800–900 m^3/h BDU and WW were installed on the pilot plant. Absorber top temperature 25–35°C. Solvent 30% MEA.
Simulation in Aspen Plus, including both vapour and droplet phase. (Thong et al., 2012)	NDELA<1*pg; NMor 2.76* ng/m^3_N MEA: 0.12* mg/m^3_N; DEA 25* ng/m^3_N NH_3: 0.12* mg/m^3_N; MA: 0.21* mg/m^3_N formaldehyde: 0.27* mg/m^3_N acetaldehyde: 0.29* mg/m^3_N	Total emissions in gas stream after WW, predicted at 25°C, for 30% MEA.
	Absorber with condenser (38°C): AMP: 5.4* mg/m^3_N; PZ: 1.13* mg/m^3_N Absorber without condenser (36°C): AMP: 38* mg/m^3_N; PZ: 19.2* mg/m^3_N	Total emissions in gas stream after WW, predicted for 15% AMP+10% PZ.
Mobile test unit. Tests in Norway and Scotland (SEPA, 2013)	NH_3: <5 ppm (in Norway); >50 ppm (in Scotland); MEA: ppb levels Nitrosamines <1 μg/m^3	Flue gas flow rate: 1,000 m^3/h CO_2 inlet: 4% (Norway) and 12% (Scotland). Solvent 30% MEA. Aker Clean Carbon carried out at three different test campaigns, one in Norway and two in Scotland
Pilot Plant at SSE's Ferrybridge Power Station (CCPilot100+) in the United Kingdom. (Fitzgerald et al., 2014)	Total emission of nitrosamines: 20 μg/m^3; the highest concentration nitrosamine: 10 μg/m^3	Capture 100 tonnes CO_2/day. RS-2 solvent. Water wash located at the absorber top. FGD polisher installed.
Technology Centre Mongstad in Norway. (Morken et al., 2014)	MEA 13–17 μg/m^3; MA: 2.6–3.6 μg/m^3 NH_3: 6–11 mg/m^3; Nitrosamines: <1 ppb Acetaldehyde: 310 μg/m^3 In the solvent and process liquids: NDELA: 4.16 mg/l; MEA-NO_2: 2.12 mg/l Nitroso-2-hydroxyethylglycine: 477 μmol/l Total nitrosamines: 797; 0.13^W μmol/l MA 3,700^W/1600^W μg/l; EA: 270^W μg/l MEA 1,600^W/37^W mg/l Nitrosamines in water wash <1 μg/l	30% and 40% MEA used as a solvent. Flow rate of flue gas 50,000 m^3/h Wash water 1/wash water 2.
Solvent degradation rig (SDR). Absorber–desorber system. (Haugen et al., 2014)	NH_3: 150-270 mg/m^3_N In the solvent: MA: 3-16 mg/l; DMA: 0.5–2.5 mg/l NDMA In the condensate: 2–27 μg/l	Emission measurement from SDR. MEA 30% as a solvent. Flue gas temperature 40°C. Flue gas contained O_2: 12–18 vol.%, NO_x 5–50 ppmv.
Aminox rig (laboratory unit for degradation) (Fostås et al., 2011)	In the solvent: NDELA: 10–1,000 ng/g NDMA: about 5 ng/g DEA: 10–612 μg/g	100h degradation test at 44°C. The effect of NO_x on 35% MEA degradation. Gas flow rate 30 m^3/h. Inlet gas contained: NO_x 5–25 ppmv and O_2: 0–14 vol.%.
Pilot plant at the Pickle research Campus in Austin, Texas (USA) and Pilot Plant2 (PP2) (Nielsen et al., 2013)	In the solvent: MNPZ 10; 140 (PP2) mg/kg MNPZ max. 325 mg/kg DEA 12.7; 9.33 (PP2) mmol/kg PZ nitrates 0.14; 4.82 (PP2) mmol/kg	Synthetic flue gas, 12% CO_2 at the inlet, 8m PZ as a solvent. 'Pilot Plant 2' – real flue gas drawn from a coal-fired power plant

Continued Table 2. An overview of nitrosamine and other major degradation products found in carbon capture plants.

Source of emission	Concentration of nitramines, nitrosamines and other major degradation products	Other useful information
The Tarong CO_2 capture pilot plant in Australia (Cousins et al., 2015)	In the solvent: total nitrosamines: 250–2,500 ppm MNPZ: 230–1,160; 124–1,000^W mg/kg in stripper condensate: 10–50 ppm	Concentrated PZ as a solvent (40%). Flue gas (600 kg/h) drawn from coal-fired power station. 12vol % CO_2 and 200 ppmv NO in the inlet gas.
Mobile Pilot Plant at Łaziska Power station in Poland (Spietz et al., 2017)	NH_3 27–50* ppm formaldehyde <1 ppm	Flue gas flow rate: 200 m^3_N/h. 12% CO_2 in the inlet gas. Flue gas drawn from hard coal-fired boiler. 40% AEEA as a solvent

* After water wash column; ^{B} after BDU and WW; ^{W} in water wash liquor; BDU, Brownian demister unit; nd, non detected; FGD, flue gas desulphurisation; $DeNO_X$ - reduction of nitrgen oxides, mg/m^3_N, milligrams per cubic meter, where N subscript stands for standard conditions in volume flow rate unit – 1,013 hPa, temperature 273 K; ppmv, part per million in volume; ppb, part per billion.

The stability of MEA and piperazine molecules in the atmosphere is estimated at 2.4 h and 1.2 h, respectively [Angove, et al., 2013]. N-nitrosamines and nitramines containing –OH group has the potential to biodegrade in natural freshwater (up to 30 days, about 35% NDELA, and 55% MEA-NO_2 biodegraded). The reason for this is that they are more polar (than the compounds without –OH group) [Sørensen, 2013, Brakstad and Zahlsen, 2011].

5. CONCLUSIONS

During the carbon capture process, amines may react with nitrogen oxides (NO_x) in the flue gas to form degradation products, including nitrosamines and nitramines. According to IARC, nitrosamines are classified as group 2B – possibly carcinogenic to humans – and group 2A – probably carcinogenic to humans (NDEA and NDMA). Nitramines in turn seem to be less potent as mutagens and carcinogens than the corresponding nitrosamines; however, the DMNA, which has been best studied, should still be regarded as a highly potent carcinogen.

Norwegian Institute of Public Health concludes that the total amount of nitrosamines and nitramines should not exceed 0.3 ng/m^3 in air and 4 ng/l in drinking water.

The data reported in the literature show that in most cases, gas emission of nitrosamines and nitramines is much less than 1 μg/m$_3$; however, concentration in solvent is more significant (even 2–5 mg/l, especially for NDMA and NDELA).

Water and acid washers as well as different kind of demisters are mainly used for removing amine emission and volatile degradation products. As nitrosamines are rapidly decomposed by photolysis, applying UV light with O_3 and O_3/H_2O_2 can be promising methods for nitrosamines removal. In fact, using a single method for emission removal may be insufficient in order to reduce undesirable compounds to acceptable levels. At the same time, additional apparatus results in additional capital and operational expenditure. Level of emission depends on used solvent, inlet gas composition, process parameters and even on ambient temperature. Therefore, design and development of gas purification system for each carbon capture plant should be considered separately. Moreover, the regular monitoring of N-nitrosamines is crucial to prevent possible exposure of the compounds to humans.

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