Environment Protection Engineering

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SIMULTANEOUS REMOVAL OF NO_x, SO_2, CO AND Hg FROM FLUE GAS BY OZONATION. PILOT PLANT STUDIES

The results of pilot plant investigations of simultaneous removal of NO_x, SO_2 and Hg from flue gas with ozone as the oxidizing agent and the spray tower absorber with NaOH solution as the absorbent have been presented. Flue gas was delivered into the pilot plant at the flow rate of 200 m^3/h from the coal fired OP-430 boiler. The effectiveness of NO_x removal was over 95% when the molar ratio O_3/NO reached 2.0. Sulfur dioxide was practically completely washed out from flue gas in the absorber at the liquid-to-gas ratio 7.5 dm^3/m^3. The effectiveness of Hg removal in the studied system was approximately 80%. It was pointed out that CO concentration in flue gas could considerably increase the ozone consumption. The effect of carbon dioxide in flue gas on the chemistry of absorption due to hydroxides conversion into carbonates and bicarbonates was noticed.

1. INTRODUCTION

According to the IE Directive [1], the Polish energy generating sector is obligated to fulfil the emission limit values 200 mg/m^3 (O_2 content 6%) for SO_2 and NO_x from coal fired boilers after January 1st 2016. Until now, almost exclusively primary methods of reduction of NO_x emission, called low NO_x combustion systems, were used. They were appropriate to meet the actual emission limits at relatively low costs [2], however, the emission limit 200 mg NO_2/m^3 (O_2 content 6%) cannot be executed using only the low NO_x combustion systems in bituminous coal fired and old lignite fired boilers.

The emissions of NO_x from coal fired power plants in the developed EU countries are controlled applying the selective catalytic reduction method (SCR) which has a status of the Best Available Technology (BAT) [1]. The SCR method is very effective in controlling the NO_x emissions, however, it has also some disadvantages: its capital and exploitation costs are considered high [3] and the use of ammonia induces

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risk of the ammonia slip. When biomass is co-firing, the catalysts could be poisoned by potassium and the lifetime of catalysts is limited due to fly ash erosion.

These disadvantages made that alternative to the SCR methods have been updated for the highly efficient and low cost methods of denitrification of flue gas. A promising alternative are wet processes for simultaneous removal of NO_x and Hg, which could be combined with the wet flue gas desulfurization (FGD) methods [4]. Unfortunately, NO and Hg^0 practically are insoluble, therefore they require pre-oxidation to soluble forms before of a scrubber. A lot of efforts has been devoted to examine efficient, safe and economic NO oxidizers in recent two decades [5]. A most considerable amount of works have been done on the ozone based methods for NO_x control, however the methods have not got the commercial status in power plants yet. The main obstacles are of economic nature; ozone generation is energy consuming (10–12 kWh/kg O_3) and requires expensive ozonizers [6]. Therefore, to make the methods cost-effective further studies on reduction of the ozone demand are necessary.

The majority of studies on NO ozonation were made in the lab scale. Nelo et al. [7] showed that efficient NO_x removal requires a substantial ozone excess. Chironna and Altshuler [5] suggested that slow oxidation rate of nitrogen oxide by air could be greatly improved by adding ozone. Jaroszynska-Wolińska [8] showed experimentally a significant acceleration of NO removal from waste gases in a two stage oxidation –absorption process by ozone addition. Fu and Diwekar [6] conducted the cost-effectiveness analysis of the low temperature oxidation (LoTOx^{TM}) process of NO. Mok [9] and Mok and Lee [10] examined experimentally a two-stage ozonation –reduction process of NO_x removal in which NO_2 was reduced by sodium sulfide. Higher than 95% efficiency of NO_x removal was achieved. Wang et al. [11] performed lab scale studies on the oxidation–absorption process of NO, SO_2 and Hg^0 applying ozone. They proved the possibility of simultaneous capturing of NO_x and SO_2 as well as 80% oxidations of elemental mercury.

Several studies on the chemical mechanism of NO ozonation were conducted. The process of NO oxidation with ozone in the well-stirred reactor was numerically simulated by Puri [12]. Wang et al. [13] studied the ozone injection process for NO control by direct numerical simulation incorporating 65-step kinetic mechanism. Jaroszyńska--Wolińska [8] studied numerically the chemical mechanism of the nitrogen oxide oxidation with ozone. Skalska et al. [14] directly measured products of NO ozonation. Skalska et al. [15] proposed kinetic model of NO ozonation and the related rate constants based on the lab scale experiment. Modliński et al. [16] examined the influence of the ozone injection pattern on the effectiveness of NO oxidation applying 2D numerical modelling.

The pilot plant studies were less frequent. Cannon Technology Inc. in collaboration with BOC Gases developed a low temperature oxidation (LoTOx) for NO_x removal by ozone injection [17]. Gostomczyk and Krzyżyńska [18] examined effectiveness of simultaneous removal of NO_x, SO_2 and Hg from flue gas using O_3. Stamate et al. [19] tested

plasma based deNO$_x$ process in a gas engine and small biomass fired power plants. Jakubiak and Kordylewski [20] observed discrepancy between the effectiveness of the ozone based deNO$_x$ processes carried out in the lab and pilot scale.

The overall goal of the pilot scale investigations was to determine the ability of simultaneous removal of NO$_x$, SO$_2$ and Hg from flue gas applying ozone as the oxidizing agent and the wet scrubbing system with an alkaline absorbent.

2. EXPERIMENTAL

Pilot plant. The tests of pollutants removal from flue gas were carried out in the pilot plant installation, consisting of the following components (Fig. 1): a water cooler of flue gas (1), fabric filter (4), ozone generator (6), spray tower absorber (19) and exhaust fan (15). Ozone was produced using an ozone generator (6), OZAT CFS-3 2G type of Ozonia. The generator (6) was fed with oxygen from a steel cylinder (5). The ozonation reactor was approximately horizontal flue gas channel connected to the container of absorbent (12). The residence time in the oxidizing reactor was ca. 2 s.

The pollutants were captured from flue gas in the absorber (19), which was a spray tower of the inner diameter of 190 mm and 4 m high. The absorbent (NaOH solution) was injected into a counter current flow spray absorber tower through the nozzles (11, 17, 18, 20) on four levels. The volumetric flow rates of absorbent through the injectors were measured using rotameters (14).

Gaseous pollutants removal. Flue gas was received at the volumetric flow rate of 200 m^3/h from the flue gas channel after an electrostatic precipitator of the coal fired OP-430 boiler. In order to control the temperature of ozonation, a water cooler (1) of flue gas was used. A fabric filter (4) was applied to reduce fly ash sedimentation in the absorbent container (12).

Ozone (1–5 vol. % of O$_3$ in oxygen) was injected into the flue gas channel under the pressure of 0.07 MPa with a lance (10) at the volumetric flow rate in the range of 1–2 m^3/h. The ozone to nitrogen oxide ratio (X = O$_3$/NO mol/mol) in flue gas was controlled by the method described elsewhere [20].

The concentrations of O$_2$, NO, NO$_2$, CO and CO$_2$ in flue gas were measured after the absorber demister (21) with a gas analyser (23), Testo xl350 of Testo, Inc. The SO$_2$ concentration was measured using the Testo s300 analyser. Electrochemical sensors of the gas analyzers were protected against the residual ozone by the thermal destructor of ozone (22). The concentrations of ions in the absorbent solution were determined by the following methods: SO$_4^{2-}$ – the ion chromatography, NO$_2^-$ and NO$_3^-$ – spectrophotometry, SO$_3^{2-}$, CO$_3^{2-}$ and HCO$_3^-$ – titration analysis. The main experimental parameters are given in Table 1.
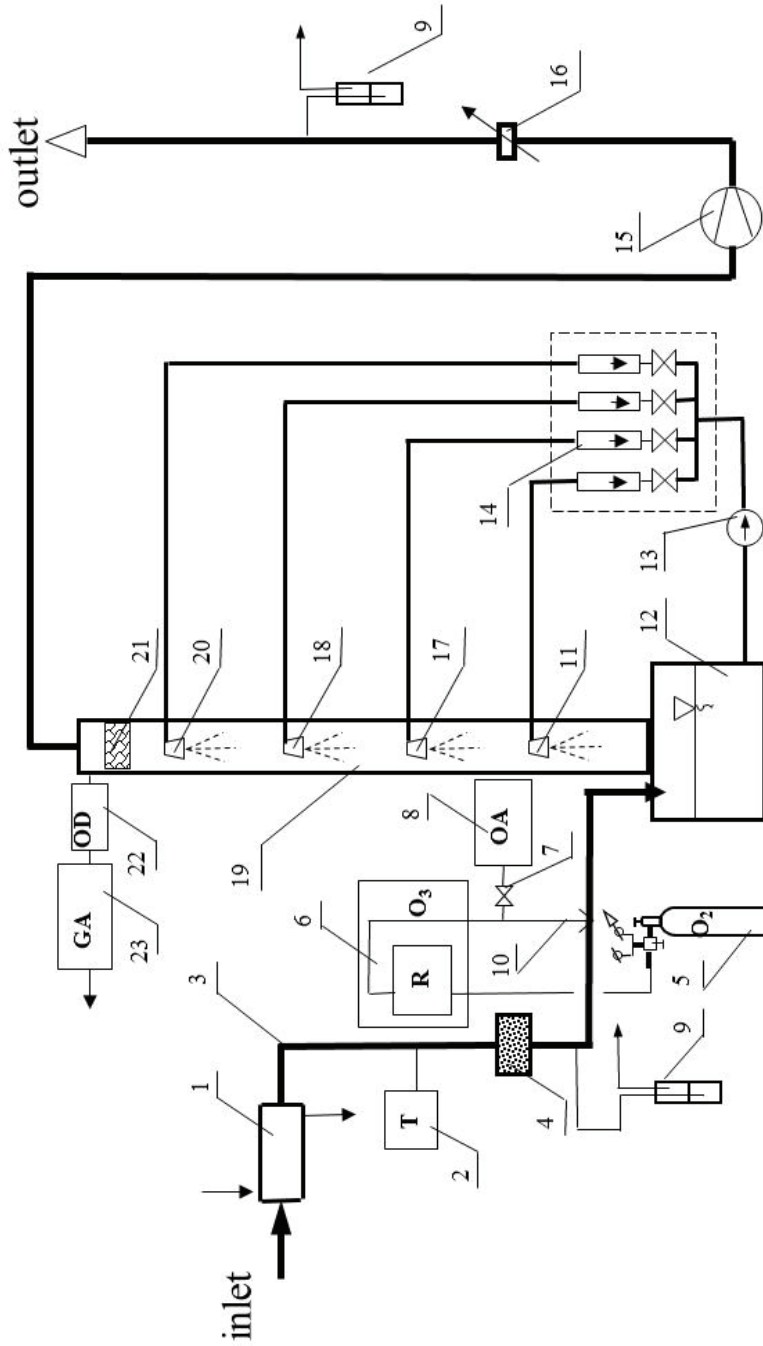
Fig. 1. Scheme of the pilot plant: 1– flue gas cooler, 2 – electronic thermometer (PT-100), 3 – flue gas duct, 4 – fabric filter, 5 – steel cylinder of O_2, 6 – ozone generator, 7 – valve, 8 – ozone analyser, 9 – bubble washers, 10 – ozone lance, 11, 17, 18, 20 – nozzles, 12 – container, 13 – pump, 14 – rotameters, 15 – fan, 16 – measuring orifice plate, 19 – absorption column, 21 – demister, 22 – ozone destructor, 23 – gas analyser

Table 1

Experimental conditions for the gaseous pollutants removal tests

Parameter	Value
Volumetric flow rate of flue gas, m^3/h	200
Concentration of NO in flue gas, mg/m^3	~290
Concentration of NO$_2$ in flue gas, mg/m^3	16–20
Concentration of SO$_2$ in flue gas, mg/m^3	500–650
Concentration of O$_2$ in flue gas, %	9.5
O$_3$/NO ratio (*X*), mol/mol	0–2
Absorbent volume, dm^3	200
NaOH concentration in the absorbent, mol/dm^3	0.1
Flow rate of absorbent, dm^3/h	0–2000
Liquid to gas ratio (*L*/*G*), dm^3/m^3	0–10
Flue gas temperature in the oxidizing reactor, °C	40
Flue gas temperature in the absorber tower, °C	35

The effectiveness of NO$_x$ removal from flue gas was calculated from the following formula:

$$\eta = \left(1 - \frac{\left[NO_x \right]}{\left[NO_{x,\,ref} \right]} \right) \times 100 \quad [\%]$$

The reference concentrations of NO$_{x,\,ref}$ denoted the values of [NO$_x$] measured in flue gas after the demister (21) when ozone was not generated using the ozonizer (6) and the absorbent was not delivered into the absorber. The same formula was used for NO, SO$_2$, CO and CO$_2$. The effectiveness of NO$_x$ removal from flue gas has been evaluated depending on the molar ratio *X*, and the influence of ozonation on the outlet gaseous pollutants such as NO, NO$_2$, SO$_2$, CO and CO$_2$ has been determined.

Mercury removal. In order to evaluate the effectiveness of mercury removal from flue gas by the ozonation method, the mercury concentration was determined in the flue gas samples, which were collected after the cooler (1) and the exhaust fan (15) (Fig. 1). The sampling apparatus contained: the titanium probe (*L* = 500 mm, ∅ = 8 mm) inserted into the flue gas channel, two Dreschel's washers (9) and two gas aspirators: ASP 3-II and ASP 3-B types (LAT Company). Mercury was captured from flue gas into the Dreschel bubble washers containing 100 cm^3 of solution prepared according to the Polish standard PN-EN 13211+AC. The solution samples were delivered into the Centre of Environmental Standards Analysis of the Wrocław University of Environmental and Life Science in order to measure the mercury concentration.

These experiments were conducted separately from the tests carried out to examine removal of gaseous pollutants. To ensure the necessary concentration of mercury

in the solution, the aspiration time of flue gas into the washers was 8–9 h. Basic parameters of these experiments are given in Table 2.

Experimental conditions for the mercury removal tests

Parameter	Value
Volumetric flow rate of flue gas, m^3/h	200
Flow rate of the absorbent, dm^3/h	2000
Liquid to gas ratio (L/G), dm^3/m^3	7.5
NaOH concentration in the absorbent, mol/dm^3	0.1
Mean temperature after the flue gas cooler (1), °C	70
Flue gas temperature in the absorber tower, °C	35
Volume of solution in the washers, cm^3	100
Flow rate of flue gas through the washer (9), m^3/h[a]	0.140
Flow rate of flue gas through the washer (9), m^3/h[b]	0.170
Concentration of mercury in burnt coal, mg/kg[c]	0.0347±0.010

[a]After the cooler (1).
[b]After the fan (15).
[c]Air dry according to PN-EN 14774-3:2010.

3. RESULTS

3.1. THE EFFECTIVENESS OF REMOVAL OF GASEOUS POLLUTANTS

The molar ratio $X = O_3/NO$ was gradually increasing from $X = 0$ up to $X = 2.0$ by the ozone concentration growth in oxygen after the ozonizer (6). The liquid to gas ratio (L/G) was kept constant at 7.5 dm^3/m^3 in the absorber (19). The concentrations of NO, NO_2, CO, CO_2 and O_2 in flue gas after the demister (21) vs. X are presented in Fig. 2. The sulfur dioxide concentration was not shown in this figure because SO_2 does not react with ozone in gas phase [11]. It was completely washed out in the absorber for every value of the molar ratio X.

When X increased up to 1.1, the concentration of NO was gradually decreasing approximately to 13 mg/m^3 (Fig. 2). The NO_2 concentration reached maximum at the value $X \approx 1.1$ and then decreased suddenly to attain the level of a few mg/m^3 when X reached 2.0. Carbon monoxide disappeared at the similar rate like NO. The carbon dioxide and oxygen content practically remained unchanged.

The measurement data shown in Fig. 2 was used for calculating the effectiveness of NO oxidation and NO_x removal. The results are presented in function of the molar ratio X in Fig. 3. The effectiveness of NO_x removal was over 95% for the molar ratio X close to 2.0.

The collective effects of flue gas treatment by ozone and absorption into the NaOH solution are presented in the Table 3, where the values of NO, NO_2, CO, SO_2 and CO_2 concentrations (O_2 content 6% according to the EU standards [1]) were compared with the reference values for the molar ratio $X = 0.0$ and 2.0 and the liquid to gas ratio $L/G = 0.0$ and 7.5 dm^3/m^3.
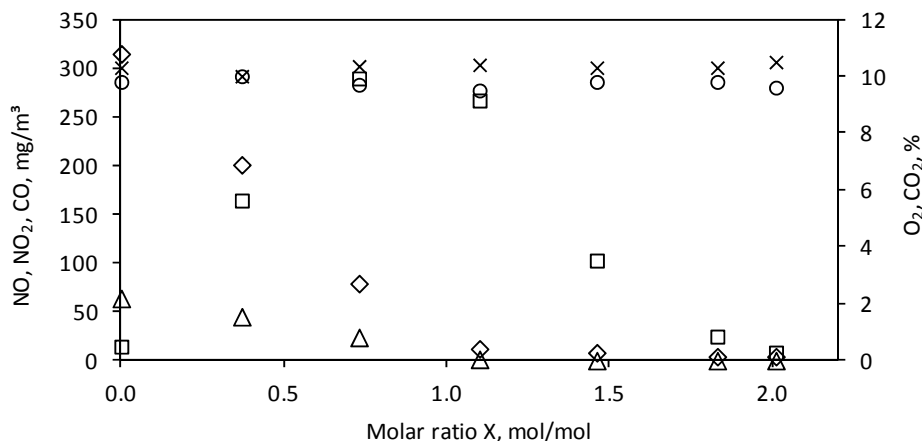

Fig. 2. The concentrations of NO ($\diamond$), NO_2 ($\square$), CO ($\triangle$), CO_2 ($\times$) and O_2 ($\circ$) vs. the molar ratio $X = O_3/NO$
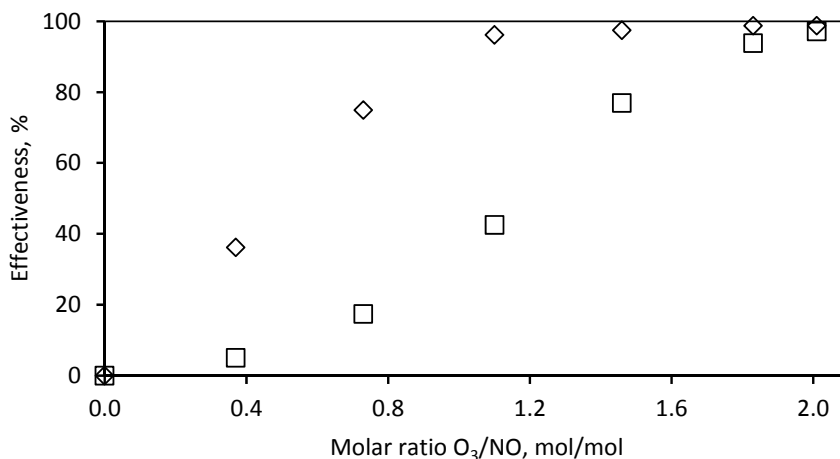
Fig. 3. The effectiveness of NO oxidation ($\diamond$) and NO_x removal ($\square$) vs. the molar ratio X ([$NO_{x, ref}$] $\approx$ 470 mg NO_2/m^3, $L/G = 7.5$ dm^3/m^3, 0.1 M NaOH)

The obtained results indicate that the combination of ozonation and absorption into alkaline solutions leads to complete removal of sulfur dioxide and carbon monoxide from flue gas. The effectiveness of NO_x removal was approximately 97%.

The effectiveness of flue gas cleaning for selected conditions of ozonation
($X = 2.0$) and scrubbing ($L/G = 7.5$)

L/G [dm³/m³]	O_3/NO_{ref} [mol/mol]	NO [mg NO_2/m³]	NO_2 [mg NO_2/m³]	NO_x [mg NO_2/m³]$_x$	SO_2 [mg/m³]	CO [mg/m³]	CO_2 [%]	η_{NOx} [%]
0.0	0.0	700	5	705	586	86	13.6	–
7.5	0.0	645	19	664	0	68	13.8	0.0
7.5	2.0	8	11	19	0	0	13.8	97.3

When the experiment was prolonged, changes in pH of the absorbent were observed: the initial pH was 12.64, after 27 min – 11.50 and after 41 min – 10.0. It was the effect of the reactions of carbon dioxide with sodium hydroxide in which sodium carbonates were produced [21]. Because of high concentration of CO_2 in flue gas NaOH was quickly removed and first sodium carbonate and next sodium bicarbonate predominated in the solution. Table 4 presents the comparison between the amounts of the selected ions in fresh absorbent (0.1 M NaOH in 200 dm³ of the Odra River water) and after 1 h of the experiment in which flue gas was ozonized at the rate of $O_3/(NO + CO) = 1.8$ and blown through the absorber tower.

Amounts [mol] of the selected ions in the absorbent
before and after the experiment

Time [min]	NaOH	SO_4^{2-}	NO_3^-	CO_3^{2-}	HCO_3^-
0	20[a]	0.146[b]	0.02[b]	–	0.157[b]
60	0	2.45	2.72	4	10

[a]Due to initial concentration of NaOH in the absorbent (0.1 M).
[b]Impurities in the Odra river water.

The amounts of ions SO_4^{2-}, NO_3^-, CO_3^{2-} and HCO_3^- at the moment 0 min are the contaminations in water used to prepare the adsorbent.

3.2. EFFECTIVENESS OF MERCURY REMOVAL

Elemental mercury is effectively oxidized with ozone to water soluble oxidized mercury which can be removed in wet alkaline scrubbers [11]. Stoichiometry of the chemical reaction of elemental mercury and ozone results from the equation:

$$Hg + O_3 \rightarrow HgO + O_2 \tag{1}$$

Because the concentration of mercury in combustion gases from coal burning is at least three orders lower than the concentration of nitrogen oxides (Tables 3 and 5), thus the reaction (1) practically does not affect the process of NO ozonation. The effectiveness of NO_x removal from air, which was gas carrier doped with NO from the cylinder [22], was not better than in these investigations.

Table 5 shows the mercury concentrations in flue gas collected before and after the pilot plant and the calculated effectiveness of Hg capture. The results were presented vs. the molar ratio $X = O_3/NO$ because ozone is consumed mainly in the reactions with nitrogen oxides in flue gas.

T a b l e 5

Effectiveness of mercury removal from flue gas vs. molar ratio X

No.	X [mol/mol]	Concentration of Hg [$\mu g/m^3$] in flue gas after		Effectiveness of Hg removal [%]
		the cooler (1)	the fan (15)	
1	1.0	1.3	0.19	85.4
2	1.5	0.86	0.22	74.4
3	2.0	0.87	0.09	89.7

The evaluated effectiveness values of mercury removal (Table 5) are in accordance with the results of investigations performed by Western Research Institute [23] to determine the ability of reduction in elemental mercury in the bench-scale LoTOx^{TM} unit. A slipstream of coal-derived flue gas from the approx. 70 kW combustion facility was treated with ozone in the quartz reactor. Over 80% reduction of the inlet mercury concentration (nearly 12 $\mu g/m^3$) was achieved.

4. DISCUSSION

The main problem with wet scrubbing of NO_x is low solubility of NO, which constitutes about 95% of NO_x from coal combustion [24]. As a result of flue gas ozonation NO is converted into NO_2 according to the chemical equation:

$$NO + O_3 = NO_2 + O_2 \qquad (2)$$

This is a very fast reaction [8, 13], hence for the molar ratio $O_3/NO \approx 1$, the conversion of NO into NO_2 was almost complete [22]. The solubility of nitrogen dioxide is an order better than the solubility of NO [25] but it is insufficient for entire capture of NO_2 from flue gas in a wet scrubber. More intensive ozonation ($O_3/NO > 1.5$) lead to the further NO_2 oxidation and di-nitrogen pentoxide formation, as follows:

$$NO_2 + O_3 = NO_3 + O_2 \tag{3}$$

$$NO_2 + NO_3 = N_2O_5 \tag{4}$$

Solubility of N_2O_5 is three orders higher than that of NO_2, moreover N_2O_5 reacts with steam in flue gas to form HNO_3:

$$N_2O_5 + H_2O = 2HNO_3 \tag{5}$$

whose solubility is seven orders higher than that of NO_2 and five orders higher than that of SO_2 (Table 6) [25].

Table 6

Solubilities of selected nitrogen compounds
and sulfur dioxide at 25 °C [25]

Compounds	Henry's constant [mol/(dm^3·Pa)]
NO	1.9×10^{-8}
NO_2	1.2×10^{-7}
N_2O_5	2.1×10^{-5}
HNO_3	2.1
SO_2	1.4×10^{-5}

The molar ratio $X = O_3/NO$ necessary to meet an emission limit value of NO_x is an important parameter influencing economy of the method because ozone generation is an important part of the denitrification cost [6]. In the lab scale investigations, NO_x was almost entirely removed from the carrier gas at $O_3/NO > 1.5$ but in the pilot plant studies the molar ratio $O_3/NO \approx 2$ was required [20].

There are several factors which influence the ozone based de-NO_x process in a large scale, i.e. imperfect mixing in flue gas, ozone termination via heterogeneous reactions with metal walls and dust particles, ozone absorption in water droplets and reactions with other compounds in flue gas. When a solid fuel is burnt, the concentration of carbon monoxide in flue gas can be comparable with the concentration of NO_x [24]. Direct oxidation of CO with ozone is non-probable at the moderate temperature (below 500 °C) [26]:

$$CO + O_3 \rightarrow CO_2 + O_2$$
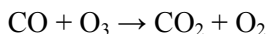
However, carbon monoxide can react with nitrogen dioxide and trioxide [27]. In the presence of ozone, CO is oxidised in fast reaction with the radicals NO_3:

$$CO + NO_3 = CO_2 + NO_2 \tag{6}$$

Although CO does not influence the NO oxidation rate, but it accelerates termination of NO$_3$ radicals and reduces the effectiveness of NO$_x$ removal. Therefore, the ozone demand for NO$_x$ removal from flue gas should also include this additional factor. Particularly, when CO level is high, it is reasonable to use the molar ratio Y which takes into account the CO influence too:

$$Y = \frac{[O_3]}{[NO]+[CO]} \tag{7}$$

In the equation, the process of oxidation of elemental mercury was not taken into consideration because the concentration of Hg^0 in flue gas is very low.

5. CONCLUSIONS

The results of the investigations carried out in the pilot plant scale concerning simultaneous removal of NO$_x$, SO$_2$ and Hg from flue gas lead to the following conclusions:
- Ozone is a very reactive agent oxidizing NO almost completely to NO$_2$ with the molar ratio O$_3$/NO close to 1.
- Over 95% reduction of NO$_x$ can be achieved with O$_3$/NO$_X$ molar ratio of ca. 2.
- Carbon monoxide eliminated nitrogen trioxide which increases the ozone demand.
- Carbon dioxide reacts with alkaline hydroxides to form carbonates and bicarbonates in the absorbent.
- Over 80% reduction in elemental mercury can be achieved with the molar ratio O$_3$/NO$_x$ = 2.0.
- The overall conclusion from these studies is that the combination of flue gas ozonation and wet scrubbing allow meeting the most stringed NO$_x$ emission limit values of the EU Industrial Emissions Directive.

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