

The Fate of Ammonia and Hydrogen Cyanide During Flameless Combustion of Low Calorific Value Gases*

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Abstract

In this paper, a review of the experimental investigations on the fuel-NO_x formation during flameless combustion is presented. The first series of experiments described in the paper were conducted using ammonia doped synthetic gases with different compositions. During these experiments, the influence of gas composition on the conversion of ammonia (NH₃) to NO_x is investigated. The second series of experiments were conducted using product gas generated in a fluidized bed gasifier. These results show the dependencies between the gasifier operating parameters, product gas composition and final NO_x emissions. Moreover, the concentrations of the ammonia and hydrogen cyanide (HCN) in the product gas were measured in order to calculate the conversion ratios of these compounds to NO_x. The results show the significant influence of the gas composition and the gasifier process parameters on the final NO_x emissions. In particular, the hydrocarbon content influences the ammonia to NO_x conversion. The lowest NO_x emissions and therefore the lowest conversion ratios were measured while burning gases with a low hydrocarbon content. An increase of the hydrocarbon concentration in the gas corresponded to a rapid increase in the conversion ratios.

Keywords: Fuel-NO_x; Flameless Combustion.

1. Introduction

The wide-ranging applicability of the Flameless oxidation (FLOX[®]) technology for the utilization of natural and low calorific value gases has already been proven and reported in the literature (Wüning et al., 1997; Galletti et al., 2007; Cavaliere et al., 2004). This technology became of interest due to its great potential in reducing thermal nitrogen oxides (thermal-NO_x) while burning natural gas. In addition to excellent burnout, the fact that fluctuations in the fuel composition do not lead to combustion instabilities is a reason for the high interest in combusting low calorific value gases (LCV) in flameless burners. However, the tests with biogenous gaseous fuels have shown a limited ability to reduce NO_x, where most of the NO_x emissions originate from the fuel-bound nitrogen (Schuster et al., 2007a, Schuster et al., 2007b). During the gasification processes, the nitrogen contained in the solid fuel is partly released to the product gases as ammonia (NH₃) or hydrogen cyanide (HCN). These compounds are formed as a result of thermal destruction of proteins and amino acids contained in the biomass (Leppälähti et al., 1995; Becidan et al., 2007). The NH₃ and HCN compounds are either converted to NO_x or reduced to neutral N₂ in the combustion process. The selectivity of this process depends on the gas composition, temperature and mixing of the reactants during combustion. In this paper, a review of the experimental investigations on the fuel-NO_x formation during flameless combustion is presented. The first series of experiments described in the paper were conducted using synthetic gases with different compositions. During these experiments, ammonia was added to the fuel stream in order to investigate the fuel nitrogen conversion. The aim of these experiments was to investigate the influence of the gas composition on the

conversion of ammonia to NO_x. Moreover, experiments in which product gas generated in a bubbling fluidized bed (BFB) gasifier is subsequently combusted in a FLOX[®]-Burner are presented. Since the gasifier product gas composition depends on the gasification process parameters such as air ratio (AR) and temperature, the final NO_x concentration is also influenced by these factors. The aim of these experiments was to investigate the dependences between gasifier operating parameters, the product gas composition (combustibles, NH₃ and HCN concentration) and final NO_x emissions during flameless combustion of these gases.

2. Experimental

In this section the test rigs, the tested fuels as well as the analysis and measurement methods are presented.

2.1. Burner test rig

The experimental facility consists of a 20 kW FLOX[®]-Burner coupled either with the gas mixing unit during experiments with synthetic gases or with the fluidized bed gasifier during experiments with solid fuels. The burner shown in Figure 1 has been originally developed for natural gas and was subsequently modified to combust low calorific value gases. During start-up the combustion chamber is heated in flame mode up to 850 °C using natural gas. Above this temperature the burner can be switched to flameless mode and fired either with low calorific value gas prepared in the gas mixing unit or with product gas generated in the gasifier. The combustion air is preheated while passing through a ceramic recuperator pipe. The temperature of the combustion chamber is controlled using an air-cooled pipe. In flameless burners the preheated

combustion air is introduced separately to the fuel into the combustion chamber. The air velocity of about 70 to 100 m/s causes high internal recirculation of the hot flue gases. These hot products are partially mixed with the fuel and oxidizer before the mixture ignites. As a result, there is no identifiable flame front. The reactants are strongly diluted and the temperature field is homogeneous without high peaks (Wünning et al., 1997).

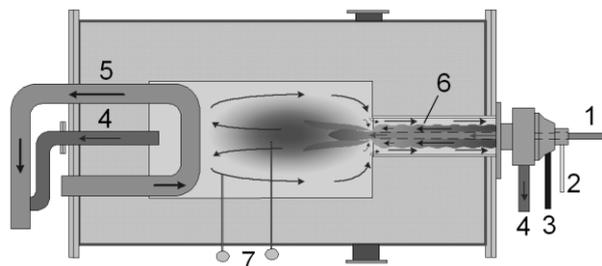


Figure 1. Scheme of FLOX[®]-Burner test rig (1 – natural gas or LCV gas with ammonia, 2 – natural gas, 3 – combustion air, 4 – flue gas, 5 – cooling pipe, 6 – recuperator pipe, 7 – thermocouples).

2.2. Gas mixing unit

The gas mixing unit, where the fuel gas is prepared, allows mixing of CH₄, CO, CO₂, H₂ and N₂. The flow of each gas is controlled by a digital mass flow controller. The gases were mixed and supplied to the burner at room temperature. Additionally, ammonia was introduced to the fuel stream to simulate the fuel nitrogen oxides precursors.

2.3. Bubbling Fluidised Bed (BFB) gasifier

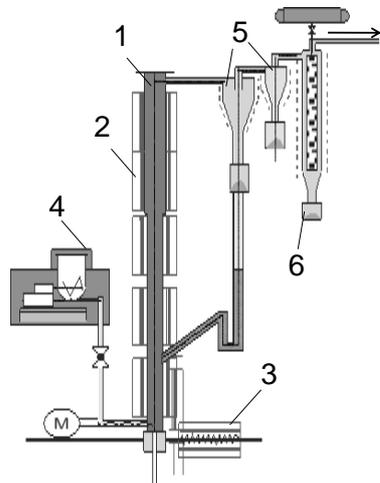


Figure 2. Scheme of BFB gasifier (1 – reactor tube, 2 – electrical heaters, 3 – air preheater, 4 – fuel feeding unit, 5 – cyclones, 6 – candle filter).

The reactor shown in Figure 2 is made of high-temperature steel and is heated by five electrical heating zones which can be controlled separately. The heating zone at the bottom of the reactor vessel, where the bed material is fluidized and the biomass is fed has a heating capacity of 11 kW_{el}. Every other heating zone above has a heating capacity of 4 kW_{el}. The maximum heating zone temperature is 1000 °C, while the resulting maximum process temperature can be 950 °C. The fluidization air can be preheated up to 900 °C in a two zone electrical gas preheater. The absolute pressure in the BFB is controlled by a pressure control valve in the range of 0-100 mbar. The

fuel feeding system is a screw feeder system where the fuel volume flow rate is controlled. The fuel rate can be set between 1 and maximum 7 kg/h depending on the type of fuel used. The product gas exits the reactor vessel and proceeds to two electrically heated cyclones connected in series, followed by a candle filter. As a result, dragged particles like ashes, char and bed material are removed. After the candle filter, a part of the product gas is led through a suction point to gas analysis. The main part of the product gas is led via a stainless steel pipe heated to 350 °C to the FLOX[®]-Burner.

2.4. Fuel composition – experiments with synthetic gases

During typical air gasification the main combustible components in the product gas are carbon monoxide and hydrogen. The amount of methane and higher hydrocarbons is usually relatively small. However, the composition of the gasification product gas depends on the solid fuel used, gasifier operating parameters and gasifier type. During the experiments with synthetic gases the composition of the investigated fuels is similar to the product gases from the gasification processes. In order to cover such a wide range of different gas compositions three synthetic gases with CO/H₂ ratios of 0.5, 0.7 and 1.5 were investigated. For each of the CO/H₂ ratios, the methane content was varied between 0 and 10 vol-% to investigate the influence of hydrocarbons on the ammonia to NO_x conversion. The concentration of CO and H₂ was kept constant for each CO/H₂ ratio. Therefore, increasing the methane content in the mixture led to a decrease in nitrogen (N₂) concentration and an increase in lower heating value of the fuel. The ammonia concentration of 700 ppmv was constant for all experiments. This concentration is related to the total mixture of fuel and combustion air to ensure the same ammonia partial pressure for each mixture. All of the mentioned experiments were carried out at the same power input of 18 kW. The compositions of the tested synthetic fuels are summarized in Table 1.

Table 1. Composition and lower heating value (LHV) of the synthetic fuels.

	Gas 1	Gas 2	Gas 3
CO (vol-%)	18	18	27
H ₂ (vol-%)	16	25	18
CO ₂ (vol-%)	15	15	15
CH ₄ (vol-%)	0-10	0-10	0-10
CO/H ₂ (-)	0.5	0.7	1.5
LHV (MJ/m ³ _{stp})	6.2-9.7	5.0-8.5	5.4-8.9
NH ₃ (ppmv)	700	700	700

2.5. Solid fuel composition and gasifier settings – experiments using BFB gasifier

In the second series of experiments the flameless burner was fired using product gas from the gasifier. Wood pellets and rape cake pellets were used as a fuel for the tests. During the experiments air was used as a gasification agent. The characteristics of the solid fuels are given in Table 2.

The rape cake pellets were chosen due to the high nitrogen content in the fuel. Both of the presented fuels were gasified under two different temperatures and gasifier air ratios. All experimental conditions are presented in Table 3.

Table 2. Characteristics of the tested biomass.

	Wood pellets	Rape cake pellets
Moisture (wt-% ar)	7.36	8.10
Ash (wt-% ar)	0.54	5.94
C (wt-% ar)	46.43	45.00
H (wt-% ar)	5.77	6.67
N (wt-% ar)	<0.3	4.98
S (wt-% ar)	<0.3	0.48
LHV (MJ/kg dm)	20.20	17.76

Table 3. Gasifier operating parameters.

Fuel	Gasification parameters	
	T (°C)	Air Ratio
Wood pellets	750	0.15
	875	0.22
Rape cake pellets	750	0.15
	875	0.22

2.6. Analysis and measurement methods

In order to evaluate the fuel-N conversion ratios to NO_x the following equation was applied

$$CR_{N \rightarrow NO_x} = \frac{[NO_x]_{fluegas} \cdot \dot{V}_{fluegas}}{[N]_{fuel} \cdot \dot{V}_{fuel}}, \quad (1)$$

where: $[NO_x]_{fluegas}$ - NO_x concentration in the flue gas in ppm_{dry}, $[N]_{fuel}$ - concentration of NH₃ and HCN in the fuel in ppm_{dry}, $\dot{V}_{fluegas}$ - flue gas flow in m³_{stp}/h, \dot{V}_{fuel} - fuel flow in m³_{stp}/h. During all tests the exhaust gas was continuously sampled and analysed at the combustion chamber exit with respect to O₂, CO₂, CO, NO, NO₂ and NO_x. During the experiments in which the burner was coupled with the gasifier the product gas was analyzed continuously with respect to CO, CO₂, CH₄, H₂ and O₂. Moreover, a gas chromatograph was used to determine the content of ethene (C₂H₄), ethane (C₂H₆) and propene (C₃H₆). The ammonia and hydrogen cyanide concentrations in the product gas were measured in order to investigate the dependencies between gasifier operating parameters, product gas composition (combustibles, NH₃ and HCN concentration) and final NO_x emission during flameless combustion of these gases. The concentration of ammonia and hydrogen cyanide was measured using the wet chemistry method. The product gas was sampled in the vicinity of the burner inlet. The gas sampling pipe was heated up to 350 °C to avoid ammonia and tar condensation. The gas was flowing through a heated filter and was led to three impinger bottles placed in an ice bath. A 0.01 M H₂SO₄ solution was used for ammonia and a 2 M NaOH solution was used for hydrogen cyanide absorption. The gas flow, temperature, pressure and oxygen concentration in the gas was measured to ensure high quality of the sampling. The content of NH₃ and HCN in the solutions was afterwards analysed according to DIN38406, (1983) and DIN38405, (1981). For each gasifier setting two gas samples were collected. The content of NH₃

and HCN is given as an average value of the analysed samples.

3. Results

In this section the experimental results achieved using ammonia doped synthetic gases and product gases generated in the BFB gasifier are presented.

3.1. NO_x emission during NH₃ doped synthetic gas combustion

In order to investigate the dependencies between hydrocarbon concentrations in the fuel and fuel nitrogen to NO_x conversion, experiments with a varying methane content were conducted. The air ratio was set to 1.5. Since the high dilution of the reaction zone caused by the internal recirculation of the hot flue gases significantly decreases the temperature peaks in the chamber, the combustion of all tested fuel mixtures occurs at a very similar and homogeneous temperature. When increasing the methane content the temperature was slightly changing in the range of 960 to 980 °C. However, such small temperature differences do not have a considerable influence on ammonia to NO_x conversion ratios (Zieba et al., 2009b). Figure 3 shows the NO_x concentration in the flue gas for three different CO/H₂ ratios as a function of methane concentration in the fuel. A rapid increase of NO_x emissions can already be observed at the lowest investigated methane concentrations of about 1 vol-%. At the methane concentration of approx. 2 vol-% the slope of the trend curve decreases. However, the NO_x emissions increase with increasing methane concentration across the whole investigated range. The measured NO_x emissions are higher in gases with a lower CO/H₂ ratio.

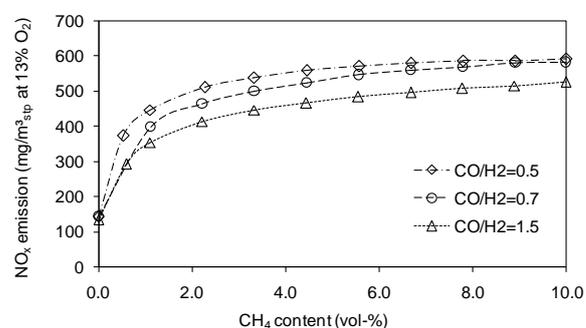


Figure 3. NO_x concentration in the flue gas as a function of methane concentration in the fuel; (*t*_{comb}=970 °C, AR=1.5).

Figure 4 presents the calculated ammonia to NO_x conversion ratios. The results show that the investigated CO/H₂ ratios had no significant influence on the conversion ratios. The higher NO_x emissions by lower CO/H₂ ratios were observed due to the changing content of water vapour in the flue gas resulting from the hydrogen oxidation. Varying the methane concentration, the conversion ratios increase from about 0.16 for methane-free mixtures to above 0.6, when 10 vol-% of methane was present in the fuel.

In order to investigate the influence of the burner air ratio on the NO_x emissions, ammonia doped fuel with a CO/H₂ ratio of 0.7 and varying methane concentration was burned at two different air ratios of 1.3 and 1.6. The methane content was varied between 0 and 4.5 vol-%. Figure 5 shows the NO_x emissions obtained in these experiments. The tests were carried out at 970 °C, with a

power input of 18 kW. Similar to the previous experiments the NO_x emissions increased with increasing methane content in the fuel for both investigated air ratios. However, for methane-free mixtures the air ratio had a minor influence on the NO_x emissions. Increasing the methane content in the fuel saw the influence of burner air ratio slightly increase.

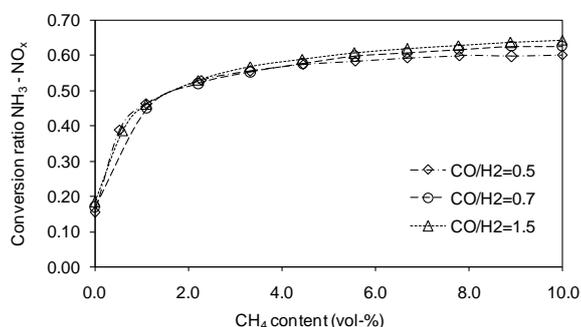


Figure 4. Ammonia to NO_x conversion ratio as a function of methane concentration in the fuel; ($t_{\text{comb}}=970^\circ\text{C}$, $\text{AR}=1.5$).

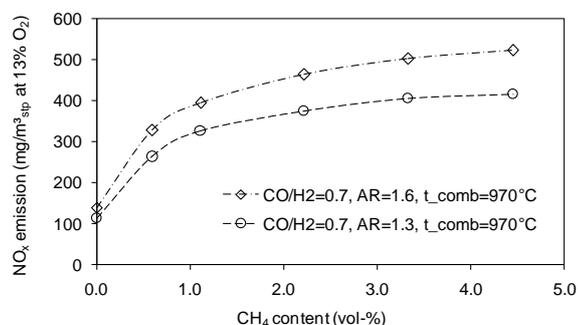


Figure 5. NO_x concentration in the flue gas as a function of methane concentration in the fuel; ($t_{\text{comb}}=970^\circ\text{C}$).

3.2. NO_x emission during flameless combustion of product gases generated in the BFB gasifier

In order to investigate if similar dependencies described in Section 3.1 can also be observed using product gases generated from biomass gasification, a series of experiments using wood pellets and rape cake pellets as a gasifier feedstock were conducted. The product gas generated in the gasifier was subsequently combusted using the flameless burner. During the experiments the product gas composition, including NH_3 , HCN and the flue gas composition were measured simultaneously. The composition of the product gas from gasification process can not be as precisely controlled as synthetic gas from a mixing unit. Therefore, to influence the product gas composition the solid bio-fuels were gasified at 750°C and 875°C with an air ratio of 0.15 and 0.22 in the case of wood pellets and 0.22 in the case of rape cake pellets.

Figure 6 shows the measured hydrocarbon concentrations for both fuels for all tested operating gasifier parameters. With a rising temperature and air ratio the total amount of hydrocarbons was similar and decreased slightly for both fuels. During the gasification of rape cake pellets much higher amounts of C_2H_4 were present in the product gas in comparison to wood pellets product gas. Higher gaseous hydrocarbons than C_3H_6 and tar were not measured.

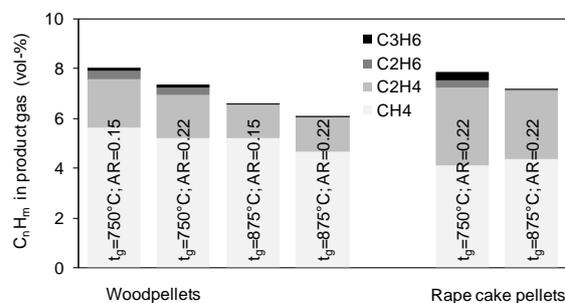


Figure 6. Hydrocarbon concentrations in the product gas during wood and rape cake pellets gasification.

3.2.1 Fuel- NO_x precursors and NO_x emission - wood pellets

The measurement results of fuel NO_x precursors obtained during wood pellets gasification are presented in Figure 7. Ammonia was the main nitrogen containing compound present in the gas. Its concentration increased with the gasifier temperature. The concentration of HCN behaves in a quite opposite manner as it decreases with increasing temperature. The influence of the gasifier air ratio was minor. However, the concentration of both of the fuel- NO_x precursors shows a tendency to increase with higher gasifier air ratio and temperature.

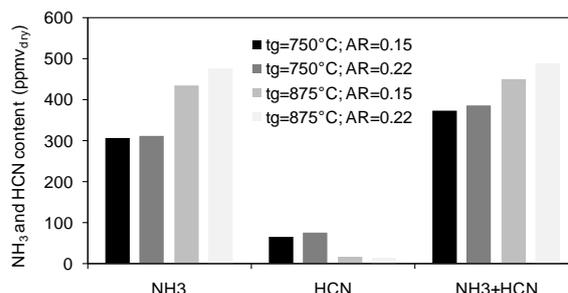


Figure 7. NH_3 and HCN concentrations in the wood pellets gasification product gas.

The product gases were combusted in the flameless burner at the average combustion chamber temperature of 1050°C . Although the quality and quantity of the product gas was changing due to gasification process instabilities the combustion process was very stable. The amount of combustion air was varied, resulting in flue gas excess oxygen in the range between 1 to 8 vol-%. Complete combustion was first observed at 1.5 vol-% O_2 in the flue gas, much earlier than expected. By further decreasing excess oxygen, particularly below 1 vol-%, a rapid increase in CO emissions was measured. Figure 8a shows the NO_x emissions observed during the experiments. The black crosses represent NO_x emissions while burning product gas generated at a gasifier temperature of 750°C , whereas the grey crosses show the emissions achieved at a gasifier temperature of 875°C . The gasifier air ratio in both cases was 0.15. The NO_x emissions are plotted against the excess oxygen in the flue gas.

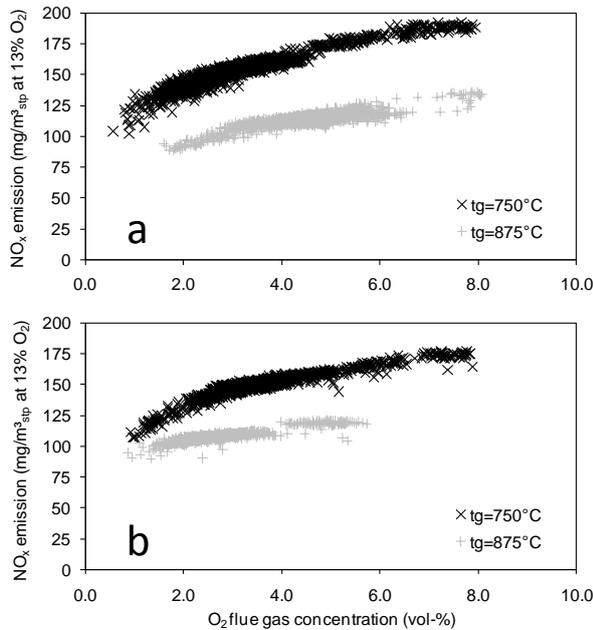


Figure 8. NO_x emission during product gas combustion from wood pellets gasification; a – gasification air ratio 0.15, b – gasification air ratio 0.22

Although, the NH_3 and HCN content in the product gas generated at 875 °C was higher, the emissions of NO_x are significantly lower. Due to lower hydrocarbon concentrations much less of nitrogen containing compounds in the product gas are converted to NO_x . Very similar results were observed for a higher gasifier air ratio. The results are presented in Figure 8b. Also in this case a higher NO_x emission level was measured during the combustion of product gases generated at a lower gasifier temperature, thus containing more hydrocarbons. Higher excess oxygen during combustion slightly enhanced the NO_x formation. The influence of the gasifier air ratio was minor. However with a higher amount of gasification air the emissions were slightly lower. The results also confirm that during combustion of gasifier product gases the content of hydrocarbons is crucial for the ammonia and hydrogen cyanide conversion ratios. The calculated conversion ratios for combustion of wood pellets are presented in Figure 9. The conversion ratio values higher than one indicate that a certain percentage of NO_x emissions are not originating from the fuel nitrogen. It can be assumed that this part of NO_x emissions is a thermal- NO_x which mostly depends on the temperature and its contribution to the total NO_x emissions is the same for all tested conditions and fuels. Since the exact amount of the thermal part of NO_x is unknown it could not be extracted from the total NO_x emissions.

3.2.2 Fuel- NO_x precursors and NO_x emission –rape cake pellets

Contrary to wood pellets the rape cake pellets contain a high amount of fuel-bound nitrogen. Therefore, high concentrations of fuel- NO_x precursors in the gasifier product gas and accordingly high NO_x concentrations in the flue gas were expected. The results are presented in Figure 10.

The concentrations of ammonia and hydrogen cyanide measured in the product gas were much higher than in the case of wood pellets gasification. Concentrations up to 35000 ppmv_{dry} were observed. Contrary to results obtained with wood pellets, a higher NH_3 concentration and a lower

HCN concentration were measured in the product gas generated at a lower gasifier temperature. The total concentration of both fuel- NO_x precursors was lower at higher gasifier temperatures. The product gas was subsequently combusted using the flameless burner. The average combustion chamber temperature of 1050 °C was the same as that during the combustion of the wood pellets product gas. The amount of combustion air was varied in order to investigate the influence of the burner air ratio on NO_x emissions. In the case of the rape cake pellets product gas, complete combustion was possible beginning at an excess oxygen level of 2 vol-%. The NO_x emissions were about one order of magnitude higher in comparison to wood pellets product gas combustion. The results are presented in Figure 11. Similarly to the wood pellets case higher NO_x emissions were measured at a lower gasifier temperature. The difference was growing with the excess oxygen in the flue gas. A higher combustion air ratio significantly enhanced the NO_x formation.

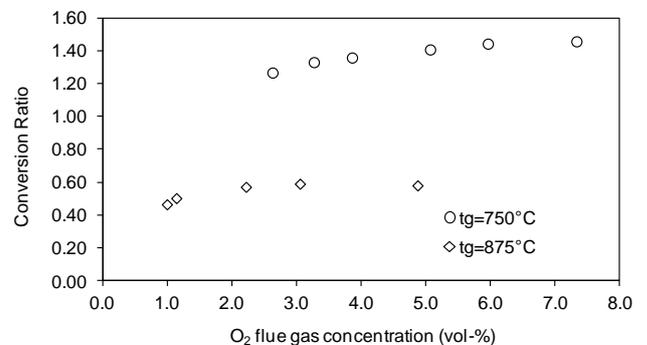


Figure 9. NH_3 and HCN to NO_x conversion ratios during product gas combustion from wood pellets gasification (AR=0.22).

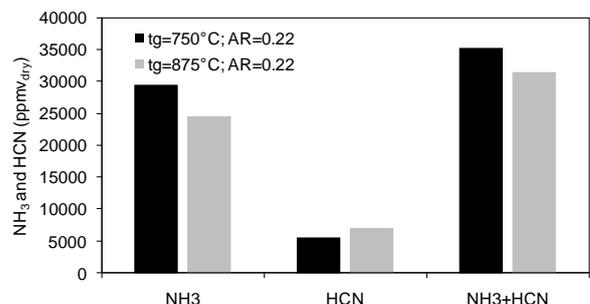


Figure 10. NH_3 and HCN concentrations in the rape cake pellets gasification product gas.

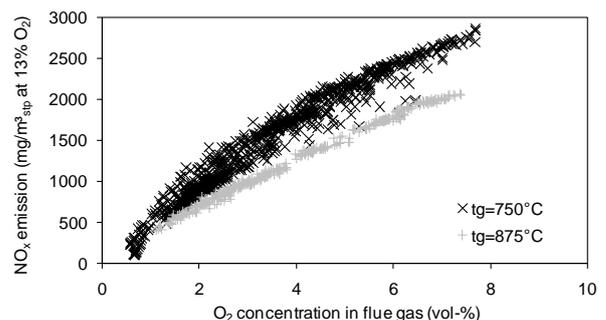


Figure 11. NO_x emission during product gas combustion from rape cake pellets gasification (AR=0.22)

The lower NO_x emissions at a higher gasifier temperature were not only observed because of the lower concentration of NO_x precursors in the product gas. Similar to the wood pellets experiments this case also saw lower hydrocarbon concentrations lead to lower conversion ratios of fuel-nitrogen. Figure 12 shows the calculated conversion ratios for product gas from rape cake pellets gasification.

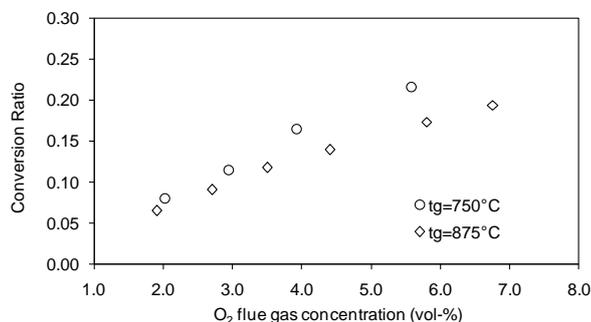


Figure 12. NH_3 and HCN to NO_x conversion ratios during product gas combustion from rape cake pellets gasification ($\text{AR}=0.22$)

The conversion ratios were calculated using Eq. (1). The conversion ratios calculated for combustion of product gases generated at the gasifier temperature of 875 °C are lower than those at 750 °C in the whole investigated range of excess oxygen. The difference between the values for the two temperatures increases with increasing excess oxygen in the flue gas.

4. Discussion

Presented results show that during flameless combustion of N-containing gases, the fuel-N to NO_x conversion ratio strongly depends on the hydrocarbon concentrations. This phenomenon can be explained by the results obtained in previous modeling studies of the authors (Zieba et al, 2009a). They examined the ammonia behavior during flameless combustion of methane-free and methane-containing ammonia doped gases using detailed chemistry. It has been found that the presence of methane strongly influences the ignition behavior and thus the location and the stoichiometry under which the ammonia reacts. In the presence of methane, due to the longer ignition time, ammonia contained in the fuel is reacting with delay in the fuel-lean. Whereas during combustion of methane-free gases, reactions occur rapidly and ammonia decomposes in a substoichiometric region of the reaction zone. In Choudhuri et al., (2003) an investigation of the combustion characteristics of hydrogen-hydrocarbon hybrid fuels is presented. This study confirms that the ignition delay can be significantly influenced by hydrocarbons. Moreover, (Skreiberg et al., 2004) investigated the ammonia chemistry below 1400 K. They also reported that methane in relatively low concentrations can strongly inhibit the ammonia conversion under fuel-rich conditions. Increasing the methane or hydrocarbon content in the fuel can see ammonia and hydrocarbons start to compete for the same radicals occurring in the combustion process. Therefore, the decomposition of ammonia can be shifted toward the fuel-lean side of the combustion, thus enhancing the conversion to NO_x .

5. Conclusions

The presented experimental studies show the fate of ammonia and hydrogen cyanide during flameless combustion of different low calorific value gases. The first series of experiments in which ammonia doped synthetic gases were burned under flameless conditions showed that depending on the gas composition, large differences in the conversion of NH_3 to NO_x can occur. In particular, the methane in low concentrations influences the ammonia to NO_x conversion. The lowest NO_x emissions and therefore the lowest conversion ratios were measured while burning methane-free gas. A rapid increase of NO_x emissions could already be observed at the lowest investigated methane concentrations. The NO_x emissions increased with increasing methane concentrations in the whole investigated range. The tested CO/H_2 ratios in the fuel had a minor influence on the conversion of ammonia to NO_x . During the second series of experiments, product gases generated from two different solid bio-fuels in a BFB gasifier were burned in the flameless burner. The goal of these studies was to show the dependencies between gasifier operating parameters and thus the composition of the product gas and final NO_x emissions during subsequent flameless combustion. The results shows the same tendencies observed during tests with ammonia doped synthetic gases. Also in this case the fuel nitrogen conversion was influenced by the product gas composition. According to investigated gasifier parameters the amount of hydrocarbons and fuel- NO_x precursors was changing. In the case of wood pellets product gas combustion, much higher NO_x emissions were observed with lower gasifier temperatures and thus with higher hydrocarbons concentrations in the product gas. This was observed even when the concentration of NH_3 and HCN was lower at a lower gasifier temperature. In the case of nitrogen-rich rape cake pellets, much higher concentrations of NH_3 and HCN were measured in comparison to wood pellets. This case also saw higher hydrocarbon concentrations lead to higher conversion rates of NH_3 and HCN to NO_x . In all cases a higher combustion air ratio enhanced the NO_x emissions. This was observed particularly during the combustion of the nitrogen-rich rape cake pellets product gas.

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