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Study of the Behaviour of a Catalytic Ceramic Candle Filter in a Lab-Scale Unit at High Temperatures

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Abstract

Solid particles and tars are among the non-desirable products of synthesis gas produced during biomass gasification. Removal of fly ashes is necessary in order to comply with emission limits as well as avoid their deposition in downstream units. Condensation of tars, on the other side, can cause clogging. A catalytic hot gas filter can remove both solids and tars, when operating at temperatures as high as 850°C. Catalytic hot gas filter elements are under development in order to solve this issue.

A lab-scale filtration unit has been designed and constructed at Delft University of Technology. The unit contains one ceramic hot gas filter candle which is made of a SiC porous structure coated with a mullite membrane. The integration of a Nickel-based catalyst layer allows the dual function of particle filtration and tar cracking. The filter vessel is part of a set-up that is equipped with a tar evaporator and a pre-heater, both located upstream of the filter unit.

This paper presents the results of the first set of experimental tests that have been performed with this unit. A dust-free model gas was used and consisted of a mixture of CO (14%), CO₂ (14%), H₂ (7%), CH₄ (5%), and varying concentrations of N₂ (30, 40, 50%) and H₂O (30, 20, 10%). Naphthalene (varying concentrations up to 9 g/Nm³) was adopted as model compound in order to study the catalytic conversion of heavier hydrocarbon species to H₂. A gas face velocity of 2.5 cm/s and 3 cm/s was selected for tests performed at atmospheric conditions and at operating temperatures varying between 700 °C and 850 °C. The pressure drop through the

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filter candle was continuously monitored during the process. The gas composition was measured upstream and downstream of the filter unit by means of an on-line micro-GC, while naphthalene concentration was attained with the SPA method.

The following findings were obtained: higher naphthalene conversion with increasing temperatures and better conversion at any temperature with lower concentrations. Tests at 850°C and 30 vol% H_2O produced a conversion of 99.4% with 2.5 g/Nm³ while 98.5% with 7.8 g/Nm³. Experiments with higher steam content showed higher conversion values. Methane concentration was also affected thus indicating that reforming reactions took place as well. Low toluene concentration was detected as a product of the reactions while no benzene was identified.

KEYWORDS: catalytic candle, gasification, tar

Introduction

Biomass is widely considered to be a major potential fuel and renewable resource for the future. In terms of size of resource, there is the potential to produce at least 50 % of Europe's total energy requirement from purpose-grown biomass using agricultural land no longer required for food, and from wastes and residues from agriculture, commerce and consumers (Bridgwater A.V., (1995)). The need to comply with legislative regulation to reduce CO₂ emissions from power generation together with the well-known availability and sustainability of biomass raised the interest and the economic investments towards power generation from biomass. Within this context, gasification of biomass is becoming one of the favourite routes and a concrete alternative to energy production from fossil fuels. In many industrialized countries primary interest shifted towards integrated gasification combined cycle (IGCC) systems and several studies (Oakey J. et al., (2004)) focus on improvement of the system efficiency. In recent years biomass gasification offered also alternative paths. Significant researches have been developed in the production of transportation fuels derived from the produced synthesis gas. A wide variety of secondary products (Methanol, DME, H₂, Fischer-Tropsch) can be obtained with gas cleaning and upgrading treatments. This development is targeted at complying with the fact that transport will continue to represent the primary demand for hydrocarbons (Clift R., (2007)). However, the produced synthesis gas contains, together with the major gas components (H₂, CO₂, CO, N₂, CH₄, H₂O), non-desirable products such as tars, ammonia, alkali, heavy metal compounds and solid particles (De Jong, W., (2008)). Therefore, removal of the fly ash in gas cleaning units is of primary importance in order not only to meet the environmental emission limits but also to protect the downstream units such as reformers, where the catalysts might be deactivated as a consequence of carbon deposition, heat exchangers and turbines from fouling and corrosion. The required specifications of particles concentration in the product gas for gas turbines application can vary from < 1 ppm up to < 30mg/Nm³ depending on different sources (Knoef H.A.M., (2005)).

The development of technologies for particulate removal from gases at high temperatures has been extremely rapid over the last years. The reasons for choosing hot cleaning systems rather than cold can be summarized in the preference to remain above acid gas dew points, avoid tar condensation, and maintain high temperatures for downstream catalytic units. Additional motivations are the improvement of the thermodynamic efficiency and of the versatility of the overall process (Seville J.P.K. et al., (2003), Woudstra T. et al. (1995)).

Ceramic filters have been defined as the most promising devices for particles removal and many studies have been performed already. The influence of the filter's characteristics, such as its permeability (Kanaoka C. et al. (2001)), on the dust cake formation, and, on the other side, the effect of dust properties in the regeneration of the filter (Koch D. et al. (2001)) and in the increase of the pressure drop (Kamiya H. et al. (2001)), have been studied in depth. Recently, research groups started working on the idea of catalytic candle filters for hot biomass gasification gas cleaning where particles removal and decomposition of tar and ammonia are integrated in one process unit. New generations of ceramic material with a catalytic function have been developed (Draelants D.J. et al., (2000), Rapagnà S. et al., (2008)) in order to solve the problem connected to the presence of tars by converting them at high temperatures. Catalytic tar conversion opens the possibility to avoid clogging of downstream equipment due to their condensation but also to increase the H₂ content in the product gas. Intense research is conducted in the development of catalyst and support material in order to improve tar conversion. Since many years nickel catalysts have been studied for steam reforming of hydrocarbons (Rostrup-Nielsen J.R., (1973)). Both steam and dry reforming reactions have been observed during tests performed with gasification gas rich in CO₂ and H₂O where toluene was the tar model compound (Simell P.A. et al. (1997)). Benzene was identified as product from naphthalene cracking over nickel-activated filter under gasification conditions (Zhao H. et al. (2000)). Parallel research on other catalysts is also being performed. A comparison study showed that Co/MgO catalyst has higher activity in naphthalene steam reforming compared to Ni/MgO (Furusawa T. et al. (2005)). Steam pretreatment of the catalyst but also higher operating conditions (900 °C) has shown to reduce coke formation (Boudouard reaction), which can cause fast deactivation of the catalyst (Pfeifer C. et al. (2008)). A drawback of nickel is that it is particularly sensitive to H₂S poisoning. Sulphur chemisorption on the catalyst surface can cause its deactivation (Hepola J., (2000)). New types of nickel-based catalysts provided promising results in methane conversion even with a degree of sulphur coverage of the nickel surface close to 1 (Koningen J. et al.(1998)). Recent studies on improved catalyst supports have shown high naphthalene conversion in presence of 100 ppm H₂S (Nacken M. et al. (2007), Ma L. et al. (2008)).

The present project, focused on gas cleaning at high temperatures, is part of a European project called "Chrisgas", which aims to study the production of synthesis gas with high hydrogen content from biomass gasification. This paper provides initial findings of tests performed with a filtration unit at atmospheric pressure and high temperatures. The aim was to study the catalytic effect of a filter element on conversion of tar under steam reforming gasification conditions.

Experimental setup

The experimental facility was designed and built for this project at the Laboratory of Process and Energy at Delft University of Technology (DUT). It consists of three sections (see Figure 1): a cold section, an evaporating section, and a high temperature section.

The first part consists of three gas lines that, via mass flow controllers, supply gas mixtures, resembling different kinds of gasification product gases. For these tests dust-free model gas was obtained by using pressurized gas bottles for the mixture of CO, CO_2 and H_2 , and for CH_4 separately, while N_2 was provided from a fixed line. Shut-off valves positioned at the inlet of the system, close instantaneously the system in case of accidental overpressure. Mass flow controllers are used to provide the desired flow and gas composition. The presence of various open-close valves along the lines gives flexibility to the system.

The evaporating section consists of a tar evaporator and a bubble flask. Tars (such as naphthalene) and light hydrocarbon species (such as benzene and toluene) can be evaporated in the two units, respectively, and added to the main gas stream. Both evaporators can be easily disconnected and used independently.

The high temperature zone consists of a preheater and a filter unit, both electrically heated. All gases are mixed and heated in the preheater until a maximum temperature of 800 °C. A water pump is connected to the preheater, thus providing the required amount of H_2O . The filter unit (height 600 mm, diameter 200 mm) was designed with a typical cylindrical shape and a conical bottom part for facilitating ashes collection. It is therefore made of a "dirty" and "clean" side, separated from each other by a plate, on which a ceramic candle is fixed (see Figure 2). It is planned that in a later stage of the project this unit will be tested also for filtration of particles. Therefore, two "side" flanges have been designed with the purpose of observing the filter cake while building up on the candle's surface. The filter candle can be easily removed by opening the vessel at the separation plate. This catalytic filter element (type Dia-Schumalith[®], see Table 1) had dimensions 250/60/10 mm (total length/outer diameter/thickness). The porous catalyst support structure was made of MgO-Al₂O₃ and impregnated with 60 wt% NiO catalyst. For more details concerning the catalytic activation see Nacken M. et al. (2008). A mullite membrane, with lower porosity compared to the monolith filter support structure, is sintered on the candle's surface. Properties of the candle are given in Table 1. Gas sampling points are located upstream and downstream of the filter unit and are connected to a micro gas chromatograph (Varian 4900 micro-GC) and an FTIR (Thermo Nicolet 5700 with a gas cell and an optical length of 2m) for gas analyses. Gas temperatures are monitored in key points along the system, though the flow diagram of the unit does not indicate

their location in detail. Two thermocouples are located inside and outside the candle, in the centre and close to the outer surface, respectively. The system temperatures, flows and pressure drop are monitored and controlled with LabviewTM.

Table 1: Properties of Dia-Schumalith [®]								
SPECIFICATIONS								
Filtration fineness of gases (µm)	0.3							
Support material	SL M 20							
Membrane type	Mullite, type 10							
Mean pore size (support) (µm)	50							
Mean pore size (membrane) (µm)	10							
Porosity support material (%)	38							
Material density (g/cm ³)	1.90							
Specific permeability (10^{-13} m^2) for air at 20 °C	21.9							

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Figure 1: Flow diagram of the lab-scale experimental facility



Figure2: Detail of the candle's fixation

Experimental tests

These initial tests used model gas with the following composition: 14 vol% CO, 14 vol% CO₂, 7 vol% H₂, 5 vol% CH₄, and a varying composition of H₂O (10, 20 and 30 vol%) and N₂ (50, 40 and 30 vol% respectively). The filter's temperature varied from 700 °C to 850 °C with 50 °C step increases. Naphthalene was selected as tar model compound. Solid naphthalene (crystals) was evaporated while the total CH₄ flow was led through the evaporator. A full set of tests for all temperatures and a face velocity of 2.5 cm/s was completed and additional tests at 800 °C and 850 °C were performed at 3 cm/s. Owing to fact that the setup is still being optimised, it was not always feasible to obtain the desired concentration of naphthalene at the filter's inlet which, therefore, varied between 0.1 and 9 g/Nm^3 in the total set of experiments. The applied concentrations, though, reflect the range of tar concentrations encountered for different biomass gasifiers. Dry and wet gas compositions were measured for at least 45 minutes upstream and downstream of the filter. The sampled gas used for dry gas analysis measurements was cooled down by passing through two condensers placed in iced water, a silica adsorbent and a paper filter. In this way, removal of tar and water was guaranteed. One column (type COX, length 1m) of the micro-GC provided the separation of the main gas components (H₂, CO, CO₂, CH₄ and N₂), with subsequent quantification using a TCD detector, while a second column (type CP-Sil 5CB, length is 4m) was used for BTX separation and quantitative analysis using an FID detector. Gas composition was available every three minutes. FTIR measurements on dry gas were also taken with FTIR and used only for comparison. The water

content was measured during wet analysis. Details of the FTIR quantification method can be found in (De Jong, W. (2005)). In this case the sampled hot gas was transported in a heated line (150 °C) and five measurements were taken with FTIR each time. Solid phase adsorption method (SPA, Brage C. et al. (1997)) was used to determine naphthalene concentration. A gas volume equal to 100 ml was sampled from the main gas stream while gas temperature at the sampling point was taken. At least three samples were collected from the inlet and outlet gas flows for each experiment Averaged values were calculated afterwards for all analyses. The integrated Ni catalyst of the catalytic filter candle was placed in the vessel, the whole unit was heated up to 850 °C for one night before the tests started.

Tar analysis

The samples taken with the SPA method and consisting of an SPE adsorption column (Bakerbond, 7088-03), were kept at freezing conditions until they were analysed with an offline custom-made gas chromatograph (GC with flame ionization detector FID, Interscience 2000). Each sample was removed from the freezer a few hours before being eluted. An elution procedure was selected and applied to each sample. Each syringe, containing 500 mg of amino-phase adsorbent, was initially eluted twice with 0,5 ml of Dicloromethane. The solvent was left percolating through the SPA by gravity, and collected in a vial. After observing the elution of few samples, a waiting time of two minutes was estimated to be long enough in order to have the maximum amount of solvent percolating through the porous media. In fact, drops were observed coming out of the syringe throughout. In a second stage, each sample was eluted for a third time with 0,5 ml of Dicloromethane while pressurized N2 was applied to the sample for about 5-10 seconds. Within this time bubbles' formation indicated that all the solvent passed through the medium. Each vial was weighed before and after being filled with the eluted sample, and net volumes of the eluted samples were calculated. Naphthalene concentrations at standard conditions were calculated from the concentrations acquired with the GC.

Results and Discussion

The inlet and outlet dry gas composition was determined for every test from an average of values obtained during sampling time. The measured inlet H_2O content was compared with the expected value, thus giving an error of maximum 1%.

This was valid for 10 and 20 vol % H_2O , while 30 vol% was above the detection limit of the FTIR and could therefore not be identified in that way. The measured water content of the outlet flow was always less than 20 vol%, thus detected. The wet gas composition was determined by using the calculated water content at the inlet and the measured values at the outlet.

When these tests were performed, the set-up was not yet equipped with a gas flow meter downstream of the filter unit. Nitrogen concentration (wet basis) showed an error of maximum 1%. Hence, this inert gas was used to calculate backwards the molar flow rates of the other gas components. Methane conversion and CO, H_2O , CO_2 and H_2 yields were calculated based on the molar flow rates of each gas as indicated in Zhao et al. (2000):

$$X = \frac{\left(F_{in} - F_{out}\right)}{F_{in}} \times 100 \tag{1}$$

$$Y = \frac{\left(F_{out} - F_{in}\right)}{F_{in}} \times 100 \tag{2}$$

Results of the tests performed at 800°C and 850°C, with a face velocity of 3 cm/s, and very low concentration of naphthalene (range 0.1-0.9 g/Nm3) are shown in Figures 3 and 4.



Figure 3: CH_4 conversion and CO_2 and H_2O yield, as a function of temperature and H_2O inlet concentration. Tests performed at a face velocity of 3 cm/s and inlet naphthalene concentration in the range 0.1-0.9 g/Nm³

The resulting gas composition can be explained, realizing that the water-gas shift reaction (ΔH_{800}^0 = 34 kJ/mol) plays a significant role:

$$CO + H_2O \Leftrightarrow CO_2 + H_2$$
 (3)

Figure 4: Yield of CO and H_2 as a function of temperature and H_2O content. Tests performed at a face velocity of 3 cm/s and inlet naphthalene concentration in the range 0.1-0.9 g/Nm³

The reaction shifts towards the left when the temperature increases. This translates in a decrease of the H_2 and CO_2 yield and a consequent increase in the CO yield (Figure 4). This is valid for 20% and 30% H_2O , while with 10% H_2O CO shows a different behaviour. Tests at 800 °C show that yield of H_2 increases with increasing H_2O content and constant temperature, thus following the principle of Le Chatelier. On the other side, the yield of H_2O appears to be involved in other reactions. Methane conversion, shown in Figure 3, was particularly high. This can be explained with the fact that naphthalene concentration was very low. Thus, this outcome confirms the competition for the Ni active sites when naphthalene is present that translates in a reduced methane conversion.

Considering the wide variety of naphthalene inlet concentration, these were grouped in the following five ranges: 0.1-0.9, 1-1.8, 1.9-3.5, 3.6-6, and 6.1-9 g/Nm³, dry gas (see Table2).

concentrations (vol%, wet basis)												
Conc. ranges	Class A (10% H ₂ O)			Class B (20% H ₂ O)			Class C (30% H ₂ O)					
$g/Nm^{3}T(^{\circ}C)$	700	750	800	850	700	750	800	850	700	750	800	850
0.1-0.9	0.86		0.13	0.1			0.82	0.1			0.80	0.12
1-1.8		1	1.63			1	1.63			1	1.63	
1.9-3.5	2.40	3.57	2.87	2.49	2.40			2.49	2.40			2.49
3.6-6						5.12	5.09					
6.1-9				8.64				7.13		6.80	9.01	7.76

Table 2: Naphthalene inlet concentrations (g/Nm^3 , dry basis) for different H₂O concentrations (vol%, wet basis)

Naphthalene conversion was calculated:

$$X_n = \frac{\left(C_{n.in} - C_{n.out}\right)}{C_{n.in}} \tag{4}$$

Practically complete naphthalene removal was obtained in the lowest range of concentration at 800 °C and 850 °C (Figures 5-7). This group belonged to the tests performed with a face velocity of 3 cm/s. The conversion showed a clear negative peak at 750 °C for the three largest concentration groups, thus indicating that the combination of lower temperatures and higher naphthalene concentrations results in a reduced cracking of this tar compound. On the other hand, increasing H₂O content from 10 vol% to 30 vol% showed to improve the naphthalene conversion. This effect was observed for the concentrations range 1-1.8 g/Nm³ at 750 °C and 800 °C and 6.1-9 g/Nm³ at 850 °C. For example, an inlet naphthalene concentration of 1.63 g/Nm³ led to conversion values of 97.5%, 98.5% and 99.1% conversion at 800 °C with 10%, 20% and 30% H₂O, respectively. But also an inlet naphthalene concentration of 2.49 g/Nm³ had 97.6%, 98.2%, 99.4% at 850 °C. These findings are consistent with the results achieved by Nacken et al. (Nacken M. et al. (2008)).



Figure 5: Naphthalene conversion with 10 vol% H₂O (class A)



Figure 6: Naphthalene conversion with 20 vol% H₂O (class B)

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Figure 7: Naphthalene conversion with 30 vol% H₂O (class C)

It is worth mentioning that among the BTX compounds, the concentration of benzene was of the order of few ppm, which is still within the detection limit of the micro-GC, while toluene showed higher concentrations with values as high as few hundred ppm for some tests. The linear regression coefficient derived from the calibration curves was 0.990 and 0.989 for benzene and toluene, respectively. This result would be in contradiction with what has been shown in literature before (Jess A. (1996), Engelen K. et al. (2003)). Therefore, more tests are required in order to confirm these preliminary results. Pressure drop values monitored during the tests, ranged between 40.9 mbar (at 700 °C) and 42 mbar (at 850 °C) for a face velocity of 2.5 cm/s, and 45 mbar for 3 cm/s (at 800 °C). No visible variation appeared to be depending on varying water contents. The difference between pressure drop values is not high enough in order to be able to discriminate whether carbon deposition occurred during naphthalene reforming reactions. On the other hand, the molar balance of carbon calculated from the inlet and outlet molar flow rates of the gas components closed within 1% error (tests with face velocity 3 cm/s). Hence, further tests as well as elemental analyses will be necessary.

Conclusions

Tests performed concerning a nickel activated filter candle using made-up synthesis gas doped with a model tar component have shown promising results in tar removal under steam reforming gasification conditions. Naphthalene was used as tar model compound at different inlet concentrations (0.1 g/Nm³ to 9 g/Nm³), relevant for different biomass gasification processes. Temperatures varied from 700 °C to 850 °C, which are typical for targeted high temperature gas cleaning. The selected face velocity values were in-line with usual filtration velocities, namely 2.5 and 3 cm/s. The highest yield of H_2 (increasing from 7 vol% at the inlet of the catalytic filter to 21 vol% at the outlet) was obtained at 800 °C and 30% H₂O at low naphthalene concentration (0.1-0.9 g/Nm³). Increasing inlet water content from 10 vol% to 30 vol% showed higher naphthalene conversion at 750 °C as well as at 850 °C. Nearly complete naphthalene conversion (99.4%) was achieved for an inlet concentration of 2.49 g/Nm³ of naphthalene at 850 °C and with 30 vol% H₂O. Toluene was detected as product from naphthalene reforming, while no benzene was identified, contrary to previous studies found in the literature. These findings represent the starting point for a follow up study that will be performed with an improved catalytic filter element which was developed aiming at tests with real biomass gasification gas, where H₂S presence must also be taken into account.

Notation

 $C_{naph.in}$ inlet naphthalene concentration, g/Nm³ $C_{naph.out}$ outlet naphthalene concentration, g/Nm³

F_{in} inlet molar flow rate, mol/min

F_{out} outlet molar flow rate, mol/min

X conversion, %

Y yield, %

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