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Original Article

## Exposure of refractory materials during high-temperature gasification of a woody biomass and peat mixture

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## ABSTRACT

Finding resilient refractory materials for slagging gasification systems have the potential to reduce costs and improve the overall plant availability by extending the service life. In this study, different refractory materials were evaluated under slagging gasification conditions. Refractory probes were continuously exposed for up to 27 h in an atmospheric, oxygen blown, entrained flow gasifier fired with a mixture of bark and peat powder. Slag infiltration depth and microstructure were studied using SEM EDS. Crystalline phases were identified with powder XRD. Increased levels of Al, originating from refractory materials, were seen in all slags. The fused cast materials were least affected, even though dissolution and slag penetration could still be observed. Thermodynamic equilibrium calculations were done for mixtures of refractory and slag, from which phase assemblages were predicted and viscosities for the liquid parts were estimated.

## 1. Introduction

Biomass gasification can become a part of future energy systems for the production of sustainable transportation fuels, chemicals and power. Among gasification technologies, the entrained flow technology currently under development for biomass produces the highest quality syngas, i.e. tar free syngas mainly composed of CO and H<sub>2</sub>, [1–3]. Furthermore, most industrial coal gasification plants developed after 1950 are of the entrained flow type [1]. Entrained flow gasifiers are generally operated in slagging mode, meaning that the operating temperature is above the ash melting point of the feedstock. At this temperature, tars are destructed and fuel conversion is almost complete. The high operating temperature comes however with the penalty of relatively high oxygen consumption. Nevertheless, different types of reactor walls have been developed for coal gasifiers to protect the reactor shell from the harsh conditions of the reaction zone. The refractory wall is the simplest, most efficient and lowest-cost design [1]. Here, a hot face refractory material, which can withstand the temperature and chemical conditions inside the gasifier, is installed together with one or more insulating layers (back-up layers) inside the reactor. High quality chromium oxide and/or zirconium oxide based refractories are employed in coal gasifiers because of their chemical resistance to the coal ash. Another type of wall is the water-cooled

membrane wall, which during operation is covered by a layer of solid slag over which the liquid slag will flow. This type of wall has the advantage that it is extremely durable. Almost no corrosion will occur because the membrane wall only comes in contact with solidified slag. Drawbacks are, however, high investment cost and higher heat loss (2–4%) compared to refractory walls (1%) [1] which significantly reduces the gasification efficiency.

Despite this, slagging gasification systems employing refractory walls report refractory life-times of only 6–18 months [1] and extensive research has been performed to address material issues in slagging coal gasifiers. The mechanisms for refractory degradation are related to slag-refractory interactions and include chemical dissolution, mechanical erosion, chemical and structural spalling [4–9]. The development of refractories for coal gasifiers continues to be active, and indicates that the development of refractories for entrained flow gasification of woody biomass must be considered as part of the overall development process. This is heightened by the fact that woody biomass is generally enriched in elements such as Ca, K and Mg whereas coal typically has higher contents of Al-, Fe-, Si- and Ti-bearing minerals [10]. Since the ash-forming matter in biomass and coal differ considerably, which thereby also changes the melting and wetting characteristics of the slags [11], refractory materials developed for coal slags are not necessarily resistant to the likely more alkaline woody biomass slags (e.g. [12] and

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references therein). There is a lack of studies concerning the degradation of refractories caused by interaction from ash derived from biomass, while previous experiences from a pilot-scale reactor lined with mullite-based refractory indicated detrimental interactions with woody biomass ash that led to fluxing of the refractory and blockage of the reactor outlet [13].

Additional knowledge is therefore needed towards understanding biomass slag-refractory interactions and to develop resilient refractory materials for slags with origins from biomass. The present study focused on evaluating the degradation of eight different refractory materials after they were exposed to gasification of a woody biomass and peat mixture in a pilot gasifier. The purpose of these exposures was to identify critical refractory properties (e.g., compositions and microstructures) that should be pursued in the development of a refractory for woody biomass gasifiers. The refractories were chosen from commercially available materials, ranging from cheap castables to expensive fused cast materials, and selected based on the project group's previous experiences from gasification of black liquor [14] and stem wood biomass [13]. In this work, we present the results from the evaluation including some conclusions and suggestions on refractory materials for slagging woody biomass gasifiers.

## 2. Experimental

### 2.1. The gasifier

An atmospheric entrained flow pilot gasifier was used for the experiments, see Fig. 1. The gasifier has an inner diameter of 50 cm and a height of approximately 3.9 m. It was previously described in [15], and therefore only a brief overview is given herein. Pulverized fuel was pneumatically transported from a fuel hopper to the burner mounted on top of the gasifier. The fuel feeding rate was controlled by the rotational speed of fuel dosing screws. Fuel entered the gasifier together with transport air through an  $\varnothing$  50 mm central exit of the burner. Oxygen ( $O_2$ ) was controlled by a mass flow controller and injected through four  $\varnothing$  3.5 mm inlets concentrically positioned and evenly distributed  $90^\circ$  apart outside of the fuel exit. The four  $O_2$ -inlets are directed so that the attack angle was  $45^\circ$  towards the central axis. This created a jet flame in the central part of the gasifier. Insulating refractory lining protect the outer steel shell from the hot gasification environment. The refractory hot-face being exposed to the gasification environment was Gouda Vibron 160H (90 mm thick). Temperature monitoring was performed by thermocouples at eight different levels along the reactor, separated approximately 40 cm in height according to Fig. 1. Eight thermocouples were positioned in the gas phase (T1–T8) and eight in the refractory (T9–T16), with the tip approximately 20 mm from the hot face wall. Gas phase thermocouples were protected by ceramic encapsulation ( $\varnothing$  8 mm) and were of type-S (T1–T4) and type-K (T5–T8). Refractory thermocouples were all of type-K.

Syngas was continuously sampled from the bottom part of the gasifier as indicated in Fig. 1. The resulting syngas composition was monitored by a Micro-GC (Varian 490 GC) with molecular sieve 5A and Poraplot U columns followed by TCD (thermal conductivity detector) detectors for detection of  $H_2$ ,  $N_2$ ,  $O_2$ , CO,  $CO_2$ ,  $CH_4$ ,  $C_2H_2$ ,  $C_2H_4$  and  $C_2H_6$ .

### 2.2. Fuel and experimental conditions

The feedstock was prepared by mixing a bark fuel from Glommersträsk, Sweden, with peat from Norrheden, Sweden. This fuel mixture was chosen based on a previous study that showed that bark fuel alone would not form a flowing slag at typical wall temperatures of  $1200$ – $1250$  °C [16]. Estimations indicated a flowing slag could be formed under the mixing proportion of 70 wt% bark and 30 wt% peat. The fuel mixture was milled in a hammer mill with sieve size of 1.25 mm directly after blending. The individual fuel compositions are

shown in Table 1 together with the calculated composition of the fuel mixture. The pulverized fuel mixture was collected in big-bags, where it was stored awaiting the gasification experiments. Fuel powder was pneumatically transported from the big-bags to the receiving fuel hopper. This experimental campaign included 42 h of gasifier operation. Refueling of the hopper was repeated every 12 h. During these time periods, the gasification process was paused by introducing a small purge flow of  $N_2$  through the gasifier while fuel and  $O_2$  feeding was stopped.

Prior to campaign startup, the gasifier was preheated over night with a  $\sim$ 100 kW oil burner firing conventional diesel fuel. Additional heating was accomplished by combusting the pulverized fuel mixture until the refractory temperature in the gasifier reached close to  $1200$  °C. Once this temperature was reached, the probes holding the ceramic samples were installed in the gasifier directly followed by reducing the  $O_2$  feeding rate in order to switch the operation to gasification. Fuel powder was fed to the gasifier using constant mass flow of transportation air corresponding to  $220 \pm 10$  NL/min (average  $\pm$  standard deviation). Fuel feeding rate was  $25 \pm 3$  kg/h, whereas the  $O_2$  feeding rate ( $174 \pm 8$  NL/min) was used to control the process temperature and maintain a temperature of approximately  $1100$ – $1140$  °C on the refractory thermocouples that were positioned closest to the sample probes, i.e. T13 in Fig. 1. Pilot gasifiers generally have higher proportional heat losses compared to industrial full-scale gasifiers. In order to achieve a certain gasification temperature, pilot gasifiers must therefore be operated with relatively higher oxygen feed. Thus, the gasifier used in this work was operated at an oxygen stoichiometric ratio,  $\lambda$ , of  $0.47 \pm 0.02$  during the gasification experiment. The resulting process temperatures and syngas composition are found in Tables 2 and 3, respectively.

### 2.3. Exposure of ceramics

Eight different materials, that are commercially available and used in different refractory applications, were chosen for this study. Material specifications can be found in Table 4.

Refractory samples were cut as cuboids with dimensions  $13 \times 13 \times 110$  mm. Each sample was fitted with boiler cement into a rectangular  $Al_2O_3$  tube ( $20 \times 20$  mm outer dimensions and 3 mm wall thickness) that were mounted at the probe tip, see Fig. 2. The purpose of the rectangular tube was to reduce the conductive cooling effect from the probe itself. Eight water cooled sample probes were used simultaneously during the experiment. Fig. 3 is a photo taken from the top of the gasifier showing the probes installed inside the reactor during gasification. The locations of the probes were chosen as approximately representative of the average conditions inside the gasification zone. Two of the probes were equipped with type-S thermocouples for temperature measurement at the rear end of the cuboid sample (Fig. 2). Average temperatures measured by these thermocouples are also shown in Table 2. Probe temperatures were generally lower, and showed greater variation than surrounding refractory thermocouples. This was most probably an effect of the water cooling of the probes.

This investigation included two sample pieces of each refractory material. The first sample piece of each refractory material was exposed to slagging gasification during 6 h, whereas the second round of samples, except graphite and top piece of HB-sample, were installed in the gasifier for 26 h. This included approximately 2.5 h of paused operation during hopper refueling and approximately 1 h in combustion mode during heat-up after refueling. The second graphite sample was heavily affected and therefore removed in conjunction with refueling already after 7 h of exposure. Once a graphite sample was removed from the hot reactor, it was allowed to cool down in a nitrogen purged sample holder in order to avoid further combustion in the surrounding air. The top piece of the second HB-sample broke and fell down into the boiler part of the plant after being exposed under gasification conditions for 11 h and 45 min. All other samples were just removed from the gasifier and

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