



Pressurized oxygen blown entrained flow gasification of a biorefinery lignin residue



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ABSTRACT

Renewable fuels could in the future be produced in a biorefinery which involves highly integrated technologies. It has been reported that thermochemical conversion (gasification) of lignocellulosic biomass has a high potential for end production of renewable biofuels. In this work, lignin residue from biochemical conversion of wheat straw was gasified in an oxygen blown pressurized entrained flow gasifier (PEBG) at 0.25–0.30 MWth, $0.45 < \lambda < 0.5$ and 1 bar (g). A video camera mounted inside the PEBG was used to observe the flame during start up and during operation. Hydrogen (H_2), carbon monoxide (CO) and carbon dioxide (CO_2) were the main gas components with H_2/CO ratios varying during the gasification test (0.54–0.63). The methane (CH_4) concentration also varied slightly and was generally below 1.7% (dry and N_2 free). C_2 -hydrocarbons (<1810 ppm) and benzene (<680 ppm) were also observed together with low concentrations of hydrogen sulfide (H_2S , <352 ppm) and carbonyl sulfide (COS, <131 ppm). The process temperature in the reactor was around 1200 °C. The slag seemed to consist of Cristobalite (SiO_2) and Berlinite ($AlPO_4$) and Na, Ca, Mg, K and Fe in lower concentrations. Cooling of the burner will be necessary for longer tests to avoid safety shut downs due to high burner temperature. The cold gas efficiency and carbon conversion was estimated but more accurate measurements, especially the syngas flow, needs to be determined during a longer test in order to obtain data on the efficiency at optimized operating conditions. The syngas has potential for further upgrading into biofuels, but will need traditional gas cleaning such as acid gas removal and water gas shifting. Also, higher pressures and reducing the amount of N_2 is important in further work.

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1. Introduction

The EU has set ambitious targets to reduce its overall emissions by at least 20% compared to the 1990s levels by 2020 and to have a renewable energy share of 20% by 2020 [1]. Special attention has been given to development of second generation biofuels [2,3]. Studies have shown that production of biofuels (methanol, DME) via thermochemical conversion (gasification) of lignocellulosic biomass has highest well-to-wheel efficiency among different production pathways [4]. Gasification of biomass results in a synthesis gas mixture mainly composed of carbon monoxide (CO) and hydrogen (H_2) that can be further converted into products [5] such as methanol, H_2 , methane (CH_4), gasoline, diesel, dimethyl ether (DME) and ammonia (NH_3). Power production is also possible through the integrated gasification combined cycle (IGCC). Several types of biomass gasifiers exist such as fixed bed reactors, fluidized bed reactors, cyclone and entrained flow gasifiers [6–9]. Entrained flow gasifiers are commercially

used with coal as feedstock. A major difference between coal and biomass is the ash composition, which needs special attention since it can melt and cause slagging, attack the reactor containment material or block the reactor outlet. The entrained flow gasifier is fuel flexible and is targeted for oxygen blown, high pressure, high temperature, large capacities applications and has potential for production of a clean essentially tar free syngas. Durability of the containment material due to the high temperature and possible slag will be important to control. In 2011 an entrained flow oxygen blown pressurized biomass pilot gasifier, here denoted PEBG, was built and operated on wood powder [10]. It was found that process temperatures varied between 1100 and 1300 °C with a H_2/CO ratio from 0.54 to 0.57 in the produced syngas and up to 2% CH_4 was observed in the syngas produced.

Production of a variety of goods from different biomass feedstocks through different technologies may be considered as a biorefinery. The biomass conversion technologies should be highly integrated [11]. Biochemical conversion of lignocellulosic biomass to e.g. ethanol needs a pretreatment step in order to prepare the cellulose to be more accessible for hydrolysis and give high yields in the fermentation step [12]. In this type of processing of the lignocellulosic biomass

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the lignin residue obtained should be further converted to aromatics [11] or used as feedstock to a biomass gasifier [13] to fully utilize the renewable feedstock. Various strategies have been suggested for biomass pretreatment for optimal biomass utilization, but will also result in different degradation products and varying usability of the residues left from the processing [14]. Successful integration of the lignin residue in a biorefinery would also be advantageous for the cellulosic ethanol production cost. Numerous scientific studies in laboratory scale have been reported about lignin gasification [13,15–18]. However, experiences from pilot scale gasification tests of lignin are scarce. In this work we explore the potential for such a lignin residue in the PEBG pilot gasifier. The intended final application of such a process would be production of high value chemicals or biofuels from the bio synthesis gas produced in the gasification process.

2. Experimental

2.1. Lignin preparation

The lignin was produced as a byproduct during biorefinery processing of wheat straw from wheat grown and harvested in Denmark 2011. The initial chemical composition of the wheat straw was 34.5 wt.% lignin, 32.5 wt.% cellulose, 26.4 wt.% hemicellulose, 3.5 wt.% ash and 3.0 wt.% other compounds based on compositional analysis [19]. To prepare the lignin for gasification, the wheat straw was processed by (1) pretreatment, (2) enzymatic hydrolysis and (3) fermentation of free monosaccharides. The pretreatment process was carried out at 163 °C for 15 min in a BioGasol Carbofrac™ 5D at the premises of BioGasol (Ballerup, Denmark) with sulphuric acid (1 vol.% in the fluidization) as catalyst. The pretreatment was used to keep the lignin in the solid fraction throughout the biomass processing [14]. To continue with enzymatic hydrolysis, the pretreated wheat straw was cooled down and water added to give a total solid content of 20 wt.% and pH was adjusted to pH 5.0 with sodium hydroxide. The enzyme complex used was Cellic CTec2 provided by Novozymes A/S (Bagsværd, Denmark) with a concentration of 50 mg enzyme product per g cellulose as recommended by Novozymes A/S. After 120 h the enzymatically hydrolyzed slurry was transferred to a fermentor, nutrients were added and the yeast (*Saccharomyces cerevisiae*) was inoculated. The fermentation took place for 2 days at 35 °C. Subsequent to the fermentation, the fermentation slurry was separated by a decanter centrifuge and the solid fraction was dried and sent to the Energy Technology Centre in Piteå (Sweden) in two batches where the gasification tests were carried out.

2.2. Characterization and preparation of the lignin for gasification

One sample from each lignin batch was analyzed by external laboratories (ALS Global, Sweden and Belab, Sweden) for ultimate and proximate analysis as well as elemental composition. The material had been thoroughly mixed before sampling. The two lignin batches were milled with a hammer mill (MAFA EU-4B) using a sieve of 0.75 mm. The resulting powders were then characterized for particle size distribution by a Fritsch Analysette 3 sieve instrument and visually observed in a Zeiss Stemi 2000-C optical microscope. The dry content of the mixed lignin powder after milling was 89.5% as measured by a moisture balance (Mettler Toledo HB43 moisture analyzer). Slag samples obtained after the gasification test were analyzed by scanning electron microscopy (SEM) in a Zeiss Gemini Merlin at an accelerating voltage of 20 kV with a thermal field emission source. Energy dispersive spectroscopy (EDS) was carried out with an Oxford Instruments X-Max detector attached to the SEM. A PANalytical Empyrean X-ray Diffractometer equipped with an Empyrean Cu LFF HR X-ray tube, a graphite monochromator and a PIXcel3D detector was used for identification of the slag sample after the gasification test. The slag was crushed in a mortar into a powder before analysis. The

HighScore Plus evaluation software and the ICDD PDF database were used for identification of the crystalline phases.

2.3. Gasification of lignin

2.3.1. Description of the pressurized entrained flow biomass gasifier (PEBG)

The PEBG is described in detail elsewhere [10] and only a brief description is presented here. A schematic drawing of the pilot gasification plant is given in Fig. 1. The fuel powder was pneumatically transported from the hammer mill to one of the two hopper storage tanks ($2 \times 1 \text{ m}^3$) in the PEBG plant. The PEBG pilot plant, built by Infjärdens Värme AB (IVAB, Sweden), was designed for high process temperatures (1200–1500 °C) and with a thermal throughput of maximum 1 MWth and pressures up to 10 bar (g). The dimension of the PEBG gasifier was 0.52 m (inner diameter) with a length of 1.67 m with a conical shaped outlet. The reactor ceramics, mainly Al_2O_3 (63 wt.%) and SiO_2 (31 wt.%), were slowly heated up (below 100 °C/h in order to avoid thermal stress) to 1000 °C by an electrical heater (Kanthal Tubothal 27 kW) vertically mounted in the centre of the reactor. Thermocouples (Type S, with protective ceramic encapsulation) measured the process temperature at the top, at the middle circumference (120° apart) and at the lower part of the vertical reactor wall according to Fig. 1. The thermocouple tips were mounted coincident with the reactor wall. Note that the temperature in this work is defined as the process temperature measured by the thermocouples and not necessary the real gas temperature [10]. The synthesis gas and solids were cooled and separated in a water quench. The system pressure was controlled by a regulating valve on the syngas outlet pipe after the quench. A water cooled nitrogen purged video camera was used to visualize the flame and the interior of the reactor during the gasification test. After gas sampling, the main syngas stream was sent to a flare on top of the building where an LPG (Liquefied Petroleum Gas) pilot flame was used as a continuous ignition source.

2.3.2. Experimental conditions

Gasification of lignin was targeted at 1 bar (g) with a fuel feeding rate of 55 kg/h, i.e. 0.25–0.30 MWth. The oxygen equivalence ratio, or lambda (λ), defined as the ratio between the supplied oxygen (kg/s) and the stoichiometric oxygen demand (kg/s) for complete combustion, was targeted at 0.45 with an oxygen concentration of 70% of the inlet gas (i.e. 30% nitrogen). The water level in the quench was set to 30%. These parameters were not constant but had to be adjusted slightly during plant operation, which is normal in a pilot plant. In total, 160 kg of lignin was available for the gasification test.

2.4. Gas sampling and analysis

The gas composition, in mol% or mol ppm, was analyzed with respect to CO, H_2 , carbon dioxide (CO_2), nitrogen (N_2), oxygen (O_2), CH_4 and ethylene (C_2H_4) using a micro-gas chromatograph, Varian 490-GC (Micro-GC), with two thermal conductivity detectors (TCD). The Micro-GC was connected to the gasifier after the quench and had a cycle time of 3.5 min. Gas samples were withdrawn from the gasifier, using a suction pump. Before entering the chromatograph the gas was filtered using a heated absolute filter to remove particles and cooled through an indirect heat exchanger and filtered through glass wool. Two samples were also collected in 10 dm³ foil gas sample bags with a single polypropylene septum fitting (SKC). The sample bags were rinsed with N_2 3 times before sampling. The gas samples were analyzed immediately after sampling by a CP-3800 (Varian Inc.) gas chromatograph equipped with two thermal conductivity detectors (TCD) for detection of H_2 , CO, CO_2 , O_2 , C_2H_4 , acetylene (C_2H_2) and H_2S . A PFPD (pulsed flame photometric) detector was used for detection of COS and a FID (flame ionization) detector was used for hydrocarbons.

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