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**Deliverable 3.7: Report on the gasification of liquid intermediates to syngas**

Title:	<b>Report on the gasification of liquid intermediates to syngas</b>
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## Introduction

One of the routes to produce advanced biofuels from biomass under investigation in the BECOOL project involves the fast pyrolysis of biomass to a liquid intermediate energy carrier, followed by gasification of the liquid to form syngas from which the biofuel is synthesized. This route gives advantages in solving logistical challenges associated with biofuel production from biomass, as well as providing some technological advantages like separating the inorganic (ash forming) elements prior to the gasification stage and making it easier to enable pressurized operation of the gasifier to avoid syngas compression.

In the BECOOL project, giant reed (*Arundo*), fibre sorghum (*Sorghum*, “Sorgo”) and eucalyptus were converted to fast pyrolysis bio-oil (FPBO) by VTT and samples were sent to BTG for bench scale gasification tests. The production of the FPBO samples is described in deliverable report D3.2.

Various gasification techniques can be applied for the production of syngas, each with specific advantages and disadvantages. Previous work performed by BTG and in cooperation with various institutes includes the conversion of FPBO in an entrained flow gasifier at bench<sup>1</sup> and pilot<sup>2</sup> scale. Entrained flow gasification is widely used to produce syngas from various fossil feedstocks such as coal and natural gas, by reacting the feedstock with pure oxygen to form H<sub>2</sub>, CO, CO<sub>2</sub> and H<sub>2</sub>O. Due to the high operating temperature of typically 1200 °C to 1600 °C, high conversion rates are obtained and the gas quality is generally good with low tar and methane concentrations<sup>3</sup>. For coal gasifiers, part of the inorganic components present in the feedstock form a liquid slag layer inside the gasifier. This slag layer is used to protect the gasifier wall, and is continuously removed from the system. These systems are referred to as slagging entrained flow gasifiers. Feedstocks containing no, or a limited amount of inorganic elements, can be processed in non-slagging gasifiers. Examples include natural gas and residues from a petrochemical refinery, and also FPBO can be converted in a non-slagging gasifier. The main results from the previous experimental work are that; i) syngas can be produced from FPBO under stable operating conditions, ii) methane concentrations are low but do not achieve the thermodynamic equilibrium concentrations, iii) high carbon to gas ratios require efficient atomization of the FPBO to suppress char/soot formation, and iv) a relatively high oxygen to fuel ratio (equivalence ratio, ER ~ 0.4) is required to reach the desired gasification temperature.

In the autothermal catalytic reforming (ACR) system, FPBO is mixed with oxygen, and optionally steam, and partial oxidation reactions will take place and increase the temperature to around 800-900 °C. The gas mixture is then led over a catalyst bed to enhance methane and tar conversion reactions. In practice, autothermal catalytic reforming is applied in particular for natural gas and naphtha. It can be seen as entrained flow system with a downstream catalyst. The advantage is that operation at lower temperature is possible compared to non-catalytic entrained flow systems.

Previous work performed at BTG showed that a virtually tar-free product gas could be produced from FPBO by using dedicated monolithic catalysts<sup>4</sup>. These results were however obtained in an air-blown reformer, which resulted in nitrogen dilution of the syngas. For the BECOOL project, the gasifier is modified to allow the use of pure oxygen as oxidant to avoid dilution of the syngas, while trying to maintain the high overall conversion efficiencies achieved by the catalytic processing. In this deliverable report, the various activities to produce a clean syngas from the FPBO's produced by VTT are described.

## Materials & Methods

### Materials

The FPBO used for gasification tests were produced by VTT (see deliverable report D3.2). The composition of the liquid has been determined by VTT as well, results are included in **Table 1**.

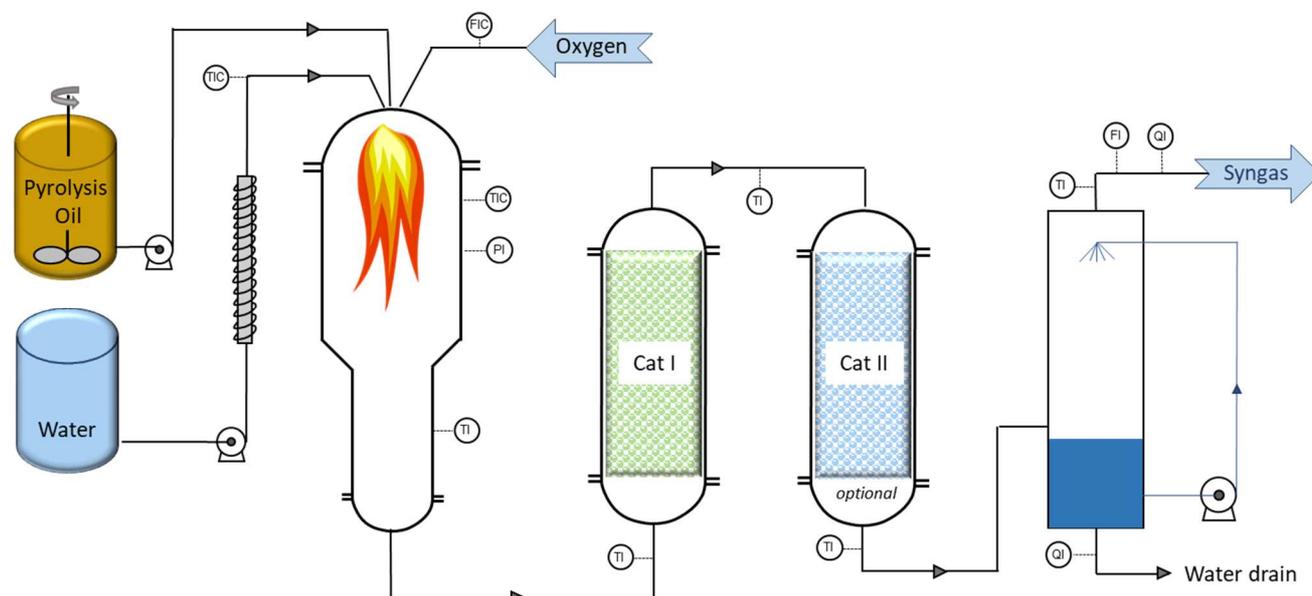
**Table 1:** Detailed analysis results (by VTT) of the FPBO's produced for the gasification work

Feedstock	Eucalyptus	Arundo	Sorghum
Water, wt %	22.8	29.6	26.1
Solids, wt%	0.14	0.25	0.63
MCR, wt%	18.6	16.6	17.5
Ash, wt %	0.03	0.16	0.09
Carbon, wt%	43.0	41.2	41.4
Hydrogen, wt%	7.7	8.0	8.0
Nitrogen, wt %	0.2	0.3	0.9
Chloride, mg/kg	240	260	133
Sulphur, mg/kg	144	338	528
Oxygen (by diff.), wt%	49	50	50
Viscosity (20 °C), cSt	66	34	37
Viscosity (40 °C), cSt	20	11	13
Viscosity (60 °C), cSt	7.1	5.1	5.8
Viscosity (80 °C), cSt	3.8	2.7	3.1
Density (20 °C), kg/dm <sup>3</sup>	1.191	1.162	1.171
Density (40 °C), kg/dm <sup>3</sup>	1.174	1.145	1.154
Density (60 °C), kg/dm <sup>3</sup>	1.156	1.126	1.124
Density (80 °C), kg/dm <sup>3</sup>	1.135	1.106	1.113
HHV, MJ/kg	17.89	17.25	17.63
LHV, MJ/kg	16.21	15.50	15.88
pH	2.8	2.9	3.5
CAN, mg KOH/g	82	88	83
Carbonyl, mmol/g	4.2	3.7	4.2
Stability test 24h 80 °C			
Viscosity increase (80 °C), %	94	143	--
Water increase (80 °C), %	9	2	--
Carbonyl decrease, %	36	32	--

The analysis results of the three FPBO's show that the eucalyptus derived FPBO contains the highest heating value and the lowest ash & solid content. These are positive from a gasification point of view, however the viscosity of this oil is also highest, which will affect the atomization properties of the FPBO. The viscosity of the other two samples is lower, partially caused by a higher water content. From the Eucalyptus, Arundo and Sorghum samples, respectively 6.8, 10.9 and 6.6 kg was supplied by VTT for the gasification tests.

## Methods

For the gasification tests, the Autothermal Catalytic Reformer (ACR) was retrofitted to allow the use of pure oxygen. To ensure safe operation, modifications were also required in the data acquisition and control system of the gasifier. A schematic representation of the ACR system is presented in **Fig. 1**, a photograph of the gasifier in the BTG laboratory is presented in **Fig. 2**.



**Fig. 1:** Schematic representation of the ACR system.

As can be seen in **Fig. 1**, the FPBO is mixed with oxygen and steam upon entering the gasifier. Here the partial oxidation of FPBO takes place and a 'raw' syngas is formed. Fixed bed catalyst(s) can be added to the gasifier for further reforming of the hydrocarbons. The system can run in non-catalytic mode as well, however the maximum temperature is limited to 1000 °C. The gasifier is pre-heated before an experiment using electrical ovens. During the test the ovens are used to compensate the heat loss of the system, to limit the amount of electrical energy added to the process, the oxygen content is adjusted to have a temperature in the partial oxidation zone slightly higher than the oven temperature.

The syngas is cooled down after the gasifier in a water scrubber to allow proper analysis of the volume flow and composition. During an experiment the flow of FPBO, steam and oxygen to the system are continuously recorded, as well as the volume flow and composition of the product gas. An online Synspec GC measures the  $H_2/O_2/N_2/CO/CO_2/CH_4/C_2H_4/C_2H_6/C_3H_6/C_3H_8$  every 10 minutes. A sample of the product gas is injected every 40 minutes in a second GC (Shimadzu) to verify the measurements and provide back-up data in case of problems with the online GC. A hydrogen cell continuously measures the concentration of hydrogen in the syngas, data is logged manually every 10-20 minutes. Before each test the tap water in the scrubber is refreshed and the amount weighed. After each test the scrubber is drained to determine the water accumulation during the test, furthermore the scrubber water is analysed to determine the total organic carbon (TOC) content as well as the solid content.



**Fig. 2:** Photograph of the autothermal catalytic reformer (~2 kg/h) in the BTG laboratory.

The modification towards an oxygen-blown system also resulted in a change in atomization concept. Where the previous, air-blown gasifier utilized the air as atomization medium, the lower gas volume required for oxygen blown gasification prohibits the use of a gas-assisted atomization. Instead, a pressure based atomization system has been fitted to the gasifier. The ‘HA’ range hollow fog nozzles of SprayBest (**Fig. 3**) were selected and nozzles of three different sizes were purchased (HA-0,10-63-G20, HA-0,15-63-G20, HA-0,20-63-G20).

The catalyst used in the gasifier is a commercially available 10-hole spoked wheel natural gas reforming catalyst ‘ReforMax 330’ supplied by Süd-Chemie (now Clariant). The catalyst is a potassium-promoted nickel based catalyst on alumina-oxide carrier material (**Fig. 4**).



**Fig. 3:** Nozzle to atomize FPBO



**Fig. 4:** Steam reforming catalyst used in the gasifier

## Results and discussion

Prior to the gasification experiments, tests were performed to investigate the atomization of FPBO using the HA-fog nozzles, as proper atomization is a crucial aspect to ensure a good carbon-to-gas conversion and prevent contamination and blockage of the catalyst downstream. Results of these investigations are described in the next paragraph, after which the results of the gasification tests are presented.

### Atomization of FPBO

Previous work performed at BTG already showed the necessity to achieve a proper atomization of FPBO to achieve high (>99%) carbon-to-gas ratios<sup>5</sup>. Since no carrier-gas could be used, a pressurized atomization system was selected. To investigate the atomization, a dedicated setup was constructed (see **Fig. 5**) to visually inspect the atomizer performance for the various nozzles.



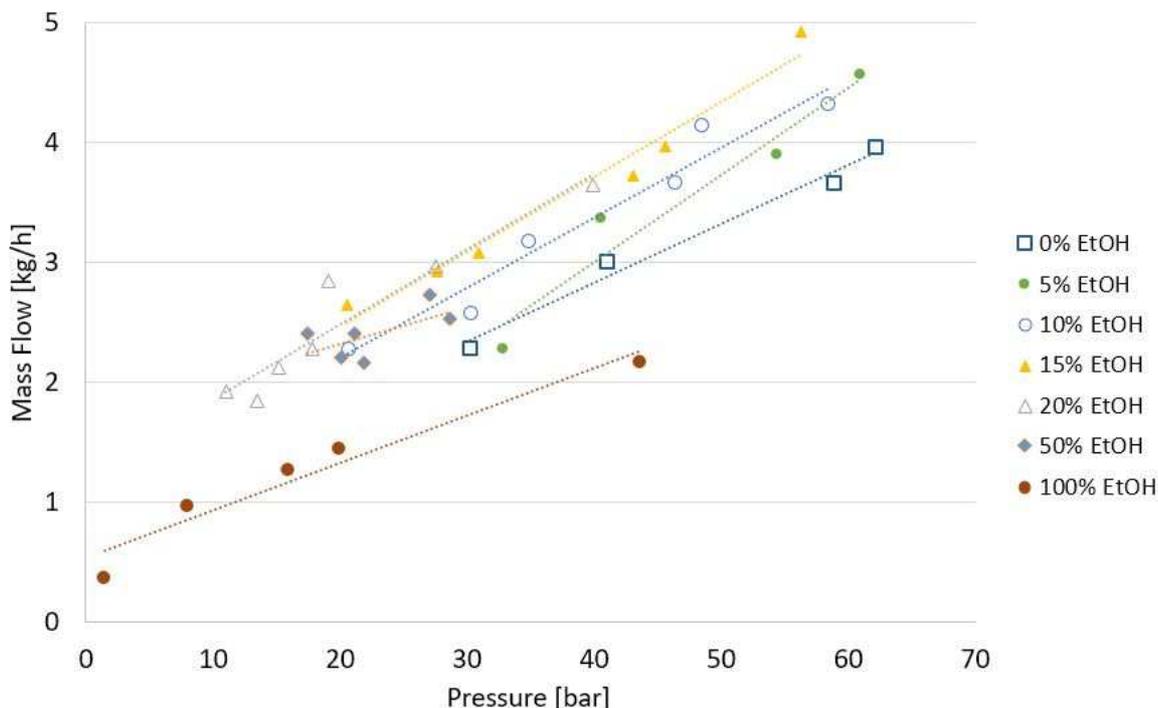
**Fig. 5:** Photograph of the atomization investigation setup.

The liquid pressure supplied to the nozzle (by a high pressure piston pump) is used in the system to control the spray. Generally, a higher liquid pressure increases the atomization performance, but also the flow of the system is affected. A second parameter used in the research is the blending of bioethanol in FPBO to lower the viscosity and surface tension, which also positively affects the atomization.

With the HA-0.10 nozzle the nozzle plugged rapidly even at pressures exceeding 50 bar. No proper atomization could be achieved with bioethanol contents below 50% using the HA-0.10 nozzle (i.e. 50/50 was the lowest possible blend for which proper atomization was achieved). The HA-0.20 nozzle on the other hand did produce

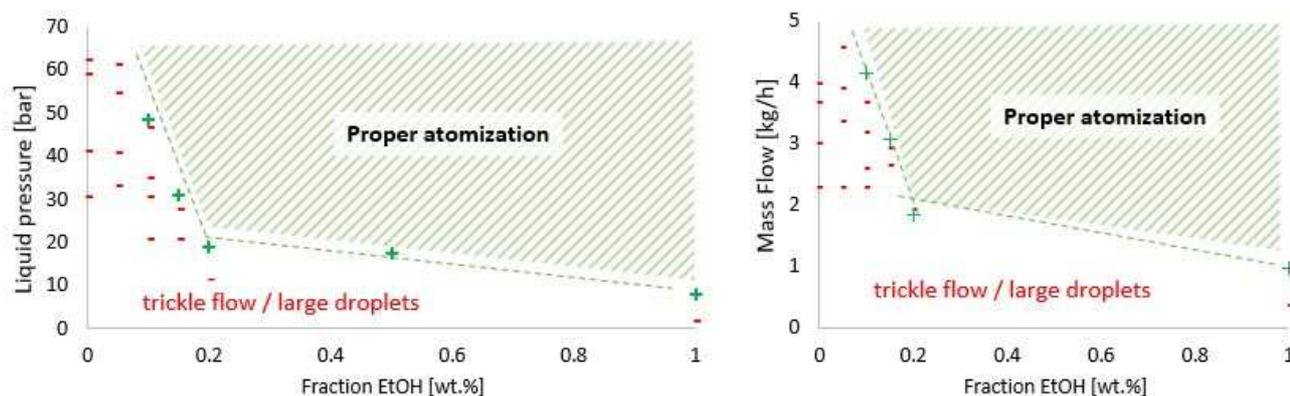
proper atomization with lower EtOH (ethanol) concentrations, however mass flows far above the desired operating conditions (>5 kg/h) were needed to achieve this atomization.

The HA-0.15 nozzle performed the best out of the available nozzles. The influence of the liquid pressure and the bioethanol content on the feed capacity is presented in **Fig. 6**. These results were obtained at a liquid temperature of 40 °C. The data in this graph contains both measurements with proper atomization as well as measurements where the liquid was injected in a single jet or even as large droplets.



**Fig. 6:** Influence of liquid pressure on the mass flow through the HA-0.15 atomizer for various FPBO:EtOH blends

A linear relation exists between pressure and capacity for the various blends. Pressures above 20 bar were required to pump the liquid through the nozzle (i.e. lower pressure did not give any flow) for most blends. The data points in **Fig. 6** however do not all represent proper atomization. Based on the visual inspection the operating window was determined for proper atomization as function of EtOH concentration and liquid pressure. The results of the visual inspection are presented in **Fig. 7**. Here, the atomization ‘quality’ is presented as function of the EtOH content and pressure (left). The resulting mass flow is very important for the applicability of the conditions in the gasifier, and is presented in the second figure (right).



**Fig. 7:** Atomization ‘quality’ for various FPBO:EtOH blends as function of liquid pressure (left) and the resulting mass flow (right) using the HA-0.15 nozzle.

To achieve a proper atomisation, while still keeping the mass flow limited to the specific system conditions, about 20 wt.% of EtOH needs to be added to the FPBO. It’s impossible to operate at the target capacity of 1 kg/h with FPBO, as compromise a 20 wt% EtOH blend is used at a capacity of 2-3 kg/h. For larger scale systems where higher mass flows are desired, pure FPBO can be used and EtOH blending is not necessary.

### Gasification of FPBO

Before performing the gasification tests using the FPBOs received from VTT, the system was commissioned using FPBO derived from clean wood. FPBO from clean wood is available in virtually unlimited supply.

### Commissioning of the setup

The FPBO from clean wood was used both to commission the setup, as well as to investigate the impact of the EtOH blended in with FPBO to improve the atomization. Based on the available thermodynamic gasification model<sup>6</sup>, the impact of EtOH in the fuel can be predicted as well. Ethanol has a higher heating value than (most) FPBOs, and also the hydrogen content is higher, these properties are expected to lead to a higher possible syngas production and a higher H<sub>2</sub> concentration in the syngas.

The FPBO mixtures with EtOH, as fed to the gasifier, were analysed by BTG as well. The analysis results are presented in **Table 2**. The name of the FPBO refers to the original biomass feedstock from which it was produced, the number in subscript refers to the amount of ethanol added to the FPBO (in wt.%).

**Table 2:** detailed analysis results on as received (ar) basis of the FPBO’s used in the gasification tests

Fuel	Arundo <sub>20</sub>	Eucalyptus <sub>20</sub>	Sorghum <sub>20</sub>	Wood <sub>20</sub>	Wood <sub>50</sub>	Wood <sub>80</sub>
Moisture, wt.%	22.9	19.8	25.6	18.7	14.5	6.1
MCRT, wt.%	12.6	14.8	12.7	13.0	11.0	4.0
Viscosity @ 40 °C, cSt	6.8	11.7	7.5	16.2	4.9	1.7
Carbon, wt.%	43.4	45.8	42.9	46.3	44.7	49.3
Hydrogen, wt.%	8.9	8.5	8.4	8.6	9.2	12.0
Nitrogen, wt.%	0.4	0.3	0.7	0.3	0.3	0.2
Oxygen (by diff), wt.%	47.3	45.4	48.0	44.8	45.8	38.6
HHVar, MJ/kg	19.9	20.5	18.9	21.0	21.3	27.8
LHVar, MJ/kg	18.0	18.7	17.1	19.1	19.2	25.2

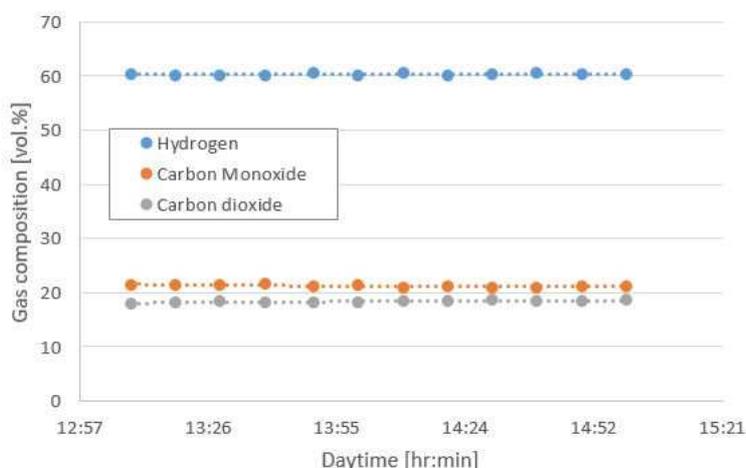
### Operating conditions

The operating conditions for the gasifier were fixed as much as possible to be able to directly compare the influence of the fuel on the process performance. A gasification temperature slightly above 900 °C in the partial oxidation zone was targeted. The gasifier was pre-heated to 900°C using electrical ovens. During operation the oxygen content to the gasifier was adjusted to reach a temperature just above 900 °C to minimize the influence of electrical heating (used as heat loss compensation) on the process performance. Typically ER values of around 0.28 were used to achieve this temperature. The steam added to the system was targeted at a S/C ratio of 1.4 mol steam / mol carbon, and also affected the gasification temperature. The catalyst used in the system accelerated the endothermic hydrocarbon reforming reactions, which resulted in a temperature drop of typically 100 °C resulting in a catalyst bed temperature around 800 °C.

The actual operating conditions (fuel input, O<sub>2</sub> input, steam input and their temperatures) achieved during the experiments were used as input values in a thermodynamic model<sup>6</sup>, which allowed the calculation of the theoretical temperature in the system as well as the gas composition at thermodynamic equilibrium.

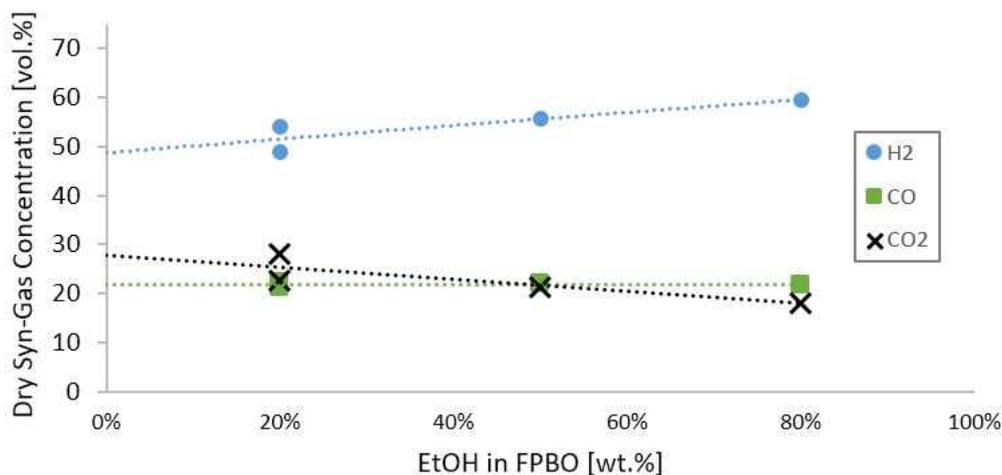
### Gasification of FPBO-EtOH blends

To investigate the impact of EtOH in the process, four gasification tests were performed with variable FPBO/EtOH blends. Each of the tests consisted of a 2 hour run at stable operating conditions. After each test the system was cooled down and the fuel injector (atomizer) was removed from the system and replaced with a new (unused) one. The gas composition was very stable during the run, indicating stable atomization performance. In **Fig. 8** the gas composition for the experiment with Wood<sub>80</sub> fuel is presented. Here, the hydrogen production is stable at 60 vol.%, with CO and CO<sub>2</sub> at 22 resp. 18 vol.%.



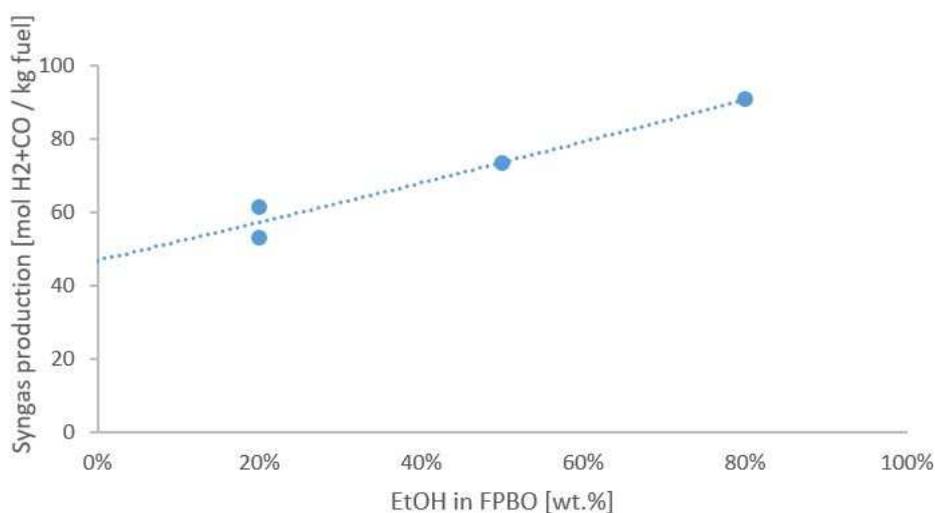
**Fig. 8:** Dry syngas composition leaving the gasifier in the Wood<sub>80</sub> experiment

The syngas composition as function of the EtOH concentration is presented in **Fig. 9**. As can be seen from the figure, the hydrogen content increases with increasing ethanol content, while the CO<sub>2</sub> content decreases. This is a direct result of the increased heating value and hydrogen content in the fuel (see **Table 2**).



**Fig. 8:** Syngas composition as function of the ethanol content in FPBO.

Depending on the desired product to be synthesized from the syngas, the gas can be shifted to change the H<sub>2</sub>/CO ratio using conventional water-gas-shift catalysts. To evaluate the results, as well as for process modelling, a useful parameter is to express the production of the system in ‘mol syngas / kg FPBO’. The production of syngas as function of the ethanol content is presented in **Fig. 9**.



**Fig. 9:** Syngas production as function of the ethanol content in FPBO.

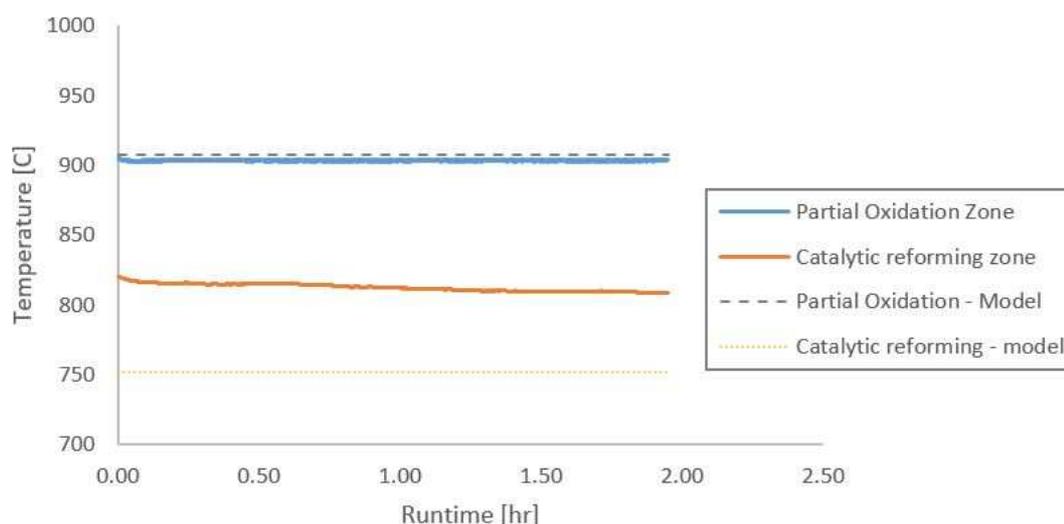
From **Fig. 9** it can be seen that higher EtOH concentrations leads to an increase in the total syngas production. This is the result of a higher hydrogen content in the fuel as well as a higher heating value. The higher heating value of the fuel results in a lower amount of fuel needed to be combusted (oxydized) to achieve the desired gasification temperature, which in turn has a beneficial effect on the overall efficiency. The total syngas production extrapolated to 0% EtOH (i.e. pure FPBO) is 47 mol syngas / kg FPBO, which corresponds well with the theoretical optimum of 48 mol syngas / kg FPBO<sup>6</sup>.

### Gasification of FPBOs produced by VTT

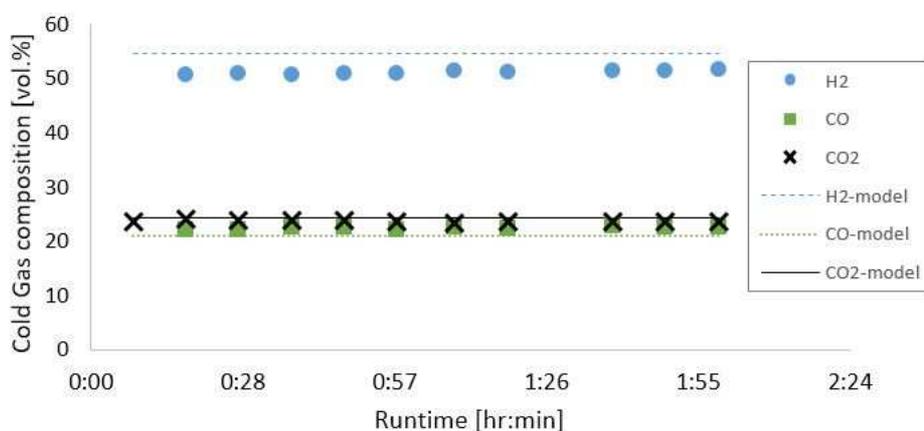
The three FPBOs were gasified attempting to use similar conditions as the wood-derived FPBO/EtOH mixtures described in the previous paragraph.

#### Gasification of Eucalyptus derived FPBO

A slightly higher fuel flow (2.9 kg/h) was required to achieve proper atomization with the Eucalyptus because of the higher viscosity. 1.23 kg/h of O<sub>2</sub> was added, and 2.40 kg/h of steam. The temperature in the gasifier is presented in **Fig. 10**, together with the temperature predicted by the model. The syngas production is presented in **Fig. 11**. For both the temperature as well as the gas composition, the experimental results are close to the thermodynamic equilibrium. The main difference lies in the methane steam reforming, which does not achieve its equilibrium value. The syngas still contained ~2.5 vol.% of methane, which results in a lower hydrogen production and a higher temperature (because methane steam reforming is endothermic). The total syngas production with Eucalyptus<sub>20</sub> was 62 mol/kg, which would correspond to approximately 52 mol/kg Eucalyptus derived FPBO when no ethanol is used in the blend.



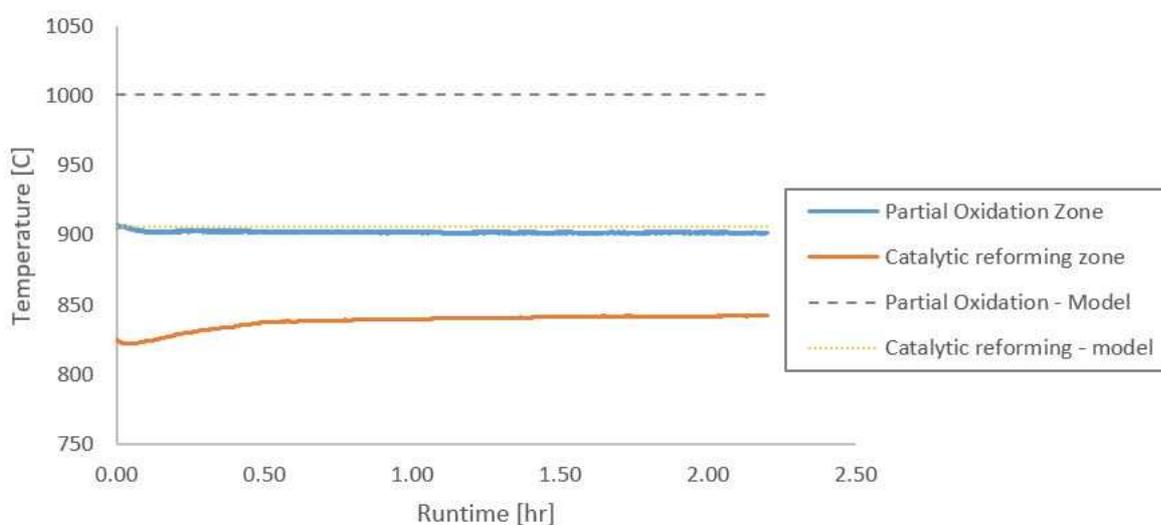
**Fig. 10:** Temperature in the gasifier for Eucalyptus derived FPBO, compared to the model results.



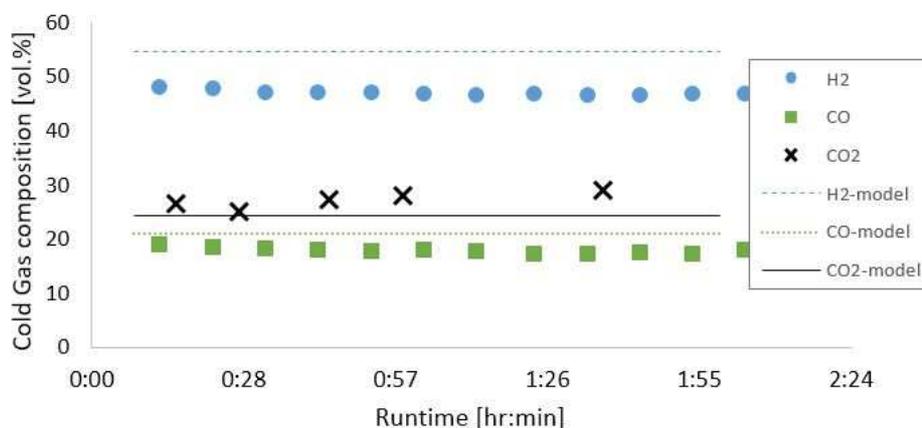
**Fig. 11:** Syngas production from Eucalyptus derived FPBO, compared to the thermodynamic equilibrium values.

*Gasification of Arundo derived FPBO*

The Arundo derived FPBO was gasified at a capacity of 2.4 kg/h, with 1.18 kg/h O<sub>2</sub> and 2.63 kg/h steam. The fuel flow was slightly lower than anticipated, while the steam supply was a bit higher. As a result, the S/C ratio was higher at 1.7 mol steam / mol carbon. Consequently, a higher O<sub>2</sub> supply was required to achieve a temperature slightly above 900 °C. The temperature in the gasifier is presented in **Fig. 12**, together with the temperature predicted by the model. The syngas production is presented in **Fig. 13**. The temperature predicted by the model is significantly higher than the temperature measured in the system. The hydrogen production is a bit lower than predicted by the model, which can be explained by the incomplete conversion of CH<sub>4</sub> in the system. However the lower CH<sub>4</sub> conversion would result in a higher than expected temperature rather than lower. The reason for the deviation in temperature is not clear at this point. The total syngas production with Arundo<sub>20</sub> was 56 mol/kg, which would correspond to approximately 46 mol/kg Arundo derived FPBO when no ethanol is used in the blend.



**Fig. 12:** Temperature in the gasifier for Arundo derived FPBO, compared to the model results.

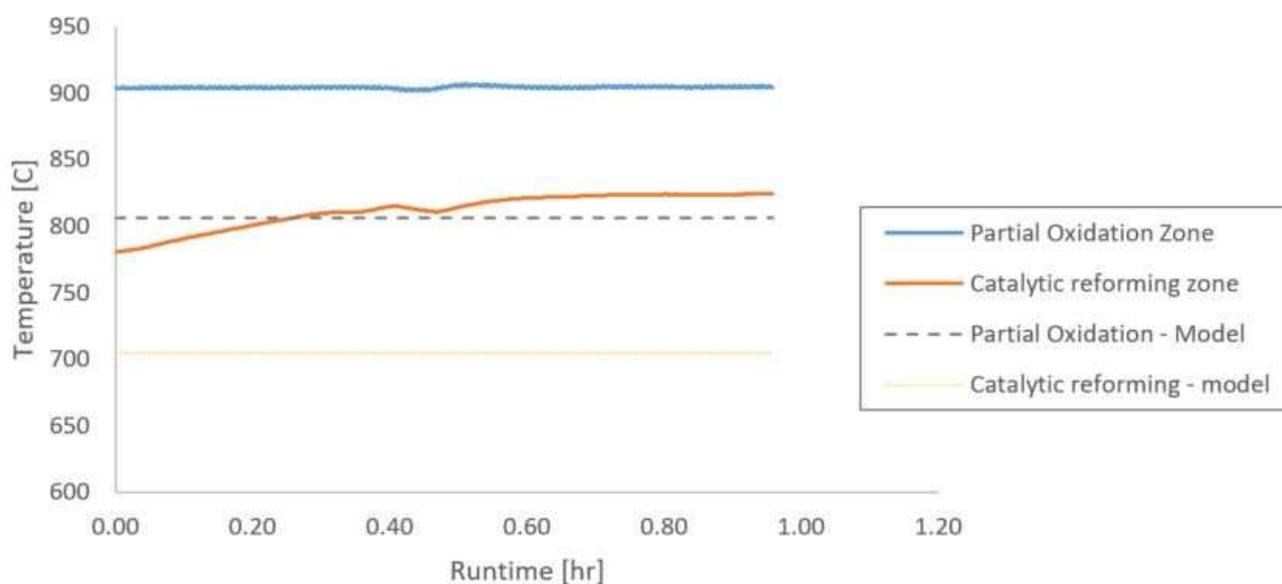


**Fig. 13:** Syngas production from Arundo derived FPBO, compared to the thermodynamic equilibrium values.

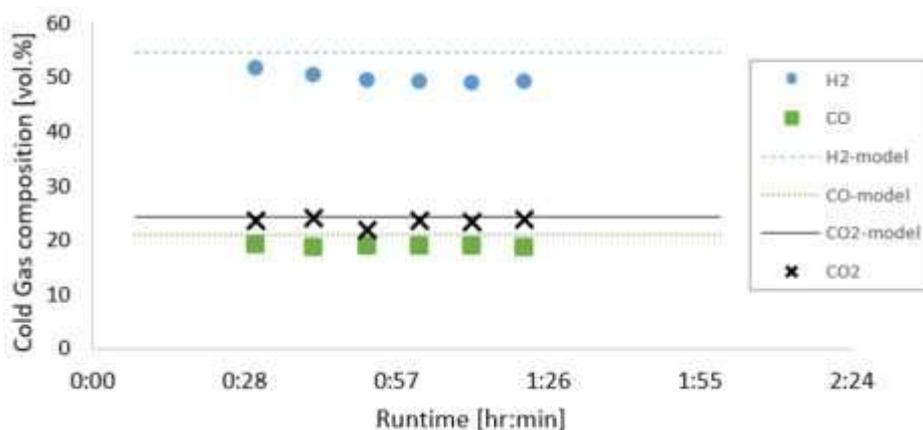
*Gasification of Sorghum derived FPBO*

The gasification of Sorghum derived FPBO was performed in two tests, during the first experiment a trip in the data acquisition system resulted in an emergency shut-down of the system. After this shut-down the nozzle was blocked and the system had to be cooled down. The nozzle was replaced and in a second experiment all the remaining FPBO was gasified. The results from the latter run are presented in this paragraph, however a similar gas composition was found in the first test with Sorghum derived FPBO.

The Sorghum derived FPBO was gasified at a capacity of 3.2 kg/h, with 1.27 kg/h O<sub>2</sub> and 2.74 kg/h steam. The S/C ration (1.3) and ER (0.29) were close to the target values. The temperature in the system (**Fig. 14**) was a bit higher than predicted by the equilibrium model, again this is likely caused by incomplete methane conversion. The syngas composition was quite similar to the composition obtained with the other FPBOs, and is presented in **Fig. 15**. The total syngas production with Sorgho<sub>20</sub> was 60 mol/kg, which would correspond to approximately 50 mol/kg Sorghum derived FPBO when no ethanol is used in the blend.



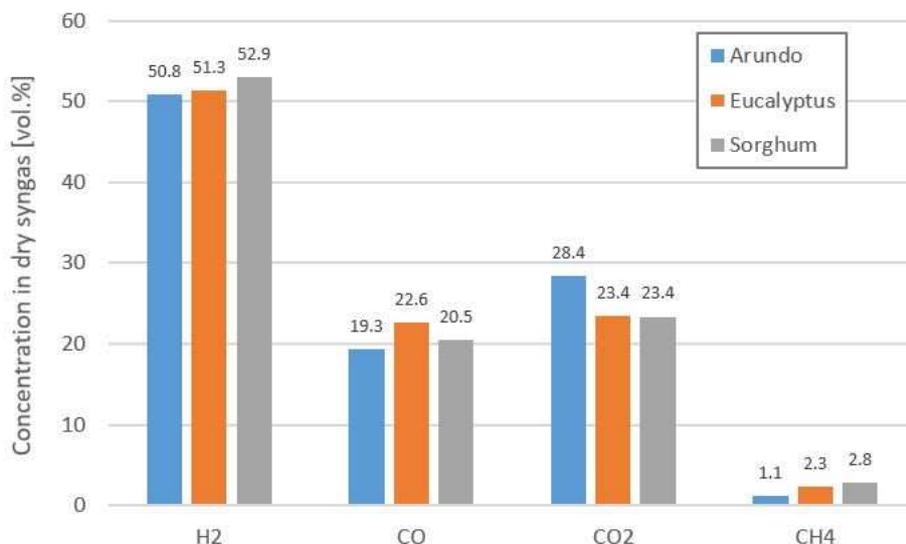
**Fig. 14:** Temperature in the gasifier for Sorghum derived FPBO, compared to the model results.



**Fig. 15:** Syngas production from Sorghum derived FPBO, compared to the thermodynamic equilibrium values.

*Summary of the gasification results with project feedstocks*

Three FPBOs produced from Arundo, Eucalyptus and Sorghum by VTT were gasified in the autothermal catalytic reformer. The temperature in the partial oxidation zone was around 900 °C and around 800 °C in the catalytic reforming zone. For each of the feedstocks, a good quality syngas was produced, containing roughly 50 vol.% H<sub>2</sub>, and the remainder almost equally distributed between CO and CO<sub>2</sub>. (see Fig. 16). The H<sub>2</sub>/CO ratio is near 2, which is good for subsequent FT synthesis. Some CH<sub>4</sub> is still present in the syngas, indicating the thermodynamic equilibrium is not yet achieved. A further increase in syngas production can be achieved by either improving the catalyst or increasing the amount of catalyst (space velocity). Long duration tests are required to determine the stability of the catalyst in time.



**Fig. 16:** Syngas composition produced from the project feedstocks.

These results show that the proposed value chain is feedstock flexible, the gas composition is almost independent of the original biomass used.

The thermodynamic equilibrium model shows well predictions of the experimental results and can be used to provide additional input to the modelling of the value chains.

*Carbon conversion*

Another important parameter to determine the performance of a gasification system is the carbon conversion, often expressed as carbon-to-gas (CtG) ratio. For each of the project feedstocks, the total amount of carbon in the product gas (mainly CO, CO<sub>2</sub> and a little CH<sub>4</sub>) slightly exceeded the amount of carbon fed to the gasifier in the fuel (carbon balance closure 101-103%). Instead it was chosen to calculate the carbon to gas ratio based on the amount of carbon recovered from the water scrubber (both the dissolved organic carbon and the solid carbon residue). The CtG for each of the tests was > 99.5%, indicating a very good gasifier performance.

## Conclusions

The proposed value chain in which various biomass materials are first converted to FPBO and subsequently gasified in an oxygen blown autothermal catalytic reformer is demonstrated to be feasible and feedstock flexible. A good quality syngas could be obtained from all the different feedstocks, with only minor variations between the different feedstocks. In general, a syngas with approximately 50 vol.% H<sub>2</sub> could be produced, with the remainder of the gas almost equally distributed between CO and CO<sub>2</sub> (H<sub>2</sub>/CO ratio ~2). A lower moisture content in the FPBO leads to a higher heating value of the FPBO, which in turn leads to a slightly higher CO production and a lower CO<sub>2</sub> production. For FPBO's with higher moisture content the opposite behaviour is observed. The average syngas production is around 50 mol (H<sub>2</sub>+CO) / kg FPBO, which corresponds well with the theoretical maximum.

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- <sup>6</sup> Biomass gasification via fast pyrolysis, chapter 7. E.J. Leijenhurst, PhD thesis – University of Ghent (2016).