

# A Simple Portable Gas Chromatograph for the Monitoring of Biomass Gasification

Bidhya Kunwar<sup>1</sup>, Hevagamage Wijaylapala<sup>1</sup>, Janet Thieme<sup>2</sup>, Sanjay V. Patel<sup>2</sup> and Todd E. Mlsna<sup>1\*</sup>

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## Abstract

Fossil fuels, coal, oil and natural gas are non-renewable sources of energy that are being consumed by the world at an unsustainable rate. Presented here is the development and testing of a simple autosampling, portable gas chromatograph for the continuous online monitoring of biomass gasification during the production of synthesis gas (primarily CO and H<sub>2</sub>). For improved portability and ease of use, the system was designed with the ability to operate with ambient air as a carrier gas and without the requirement of an oven. For automated operation a six port valve with a gas sampling loop is included. The detector for the chromatograph is a commercial-off-the-shelf metal oxide-based detector and the entire system fits into a portable weatherproof case for field use. Design features, calibration, and results of pilot scale testing are presented.

*Keywords:* Autosampling gas chromatography (GC); Gasification; Biofuel; Online monitoring; Metal oxide semiconductor (MOS)

## 1. Introduction

Fossil fuels, i.e., coal, oil and natural gas, are non-renewable sources of energy that are being consumed by the world at an unsustainable rate (Owen et al. 2010). Considerable effort is currently underway in labs around the world to develop alternative sources of fuel. One source of fuel is derived from the gasification of biomass to produce synthesis gas (syngas) (Maschio et al. 1994). This gas mixture contains varied amounts of CO, H<sub>2</sub>, CH<sub>4</sub>, CO<sub>2</sub> and other chemicals and can be combusted directly to produce energy or catalytically converted into hydrocarbon fuels (Liu et al. 2009). In order to reduce transportation cost, mobile technology is required that is capable of converting biomass to fuel at the source of the biomass. Described here is the development of a simple autosampling, portable gas chromatograph for the continuous online monitoring of biomass gasification during the production of syngas.

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\*Corresponding e-mail: tmlsna@chemistry.msstate.edu

1 Chemistry Department, Mississippi State University, Starkville, MS 39762

2 Seacoast Science, Inc., Carlsbad, CA 92011

### *1.1 Biomass Gasification*

Biomass gasification (Rezaiyan, and Cheremisinoff, 2005) extracts energy from many different types of organic materials. It converts once living lignocellulosic organic materials into carbon monoxide, hydrogen, carbon dioxide and water at high temperatures with controlled amounts of oxygen and/or steam (Equation 1). The resulting gas mixture, known as syngas, can be comprised of varying ratios of hydrogen and carbon monoxide (Gautam et al. 2010). These mixtures vary with feedstock temperature and humidity. Here we describe a real-time monitor of syngas production as the first step in addressing feedstock variability to optimize the fuel production process.



Currently qualitative and quantitative chemical analysis of syngas is typically done using benchtop instruments. In particular, gas chromatography (GC) with a flame ionization detector (Knight, 2000) and GC mass spectrometry (GCMS) (Schmieder et al. 2000) techniques are used. Online monitoring of synthesis gas production has been done using GC with a thermal conductivity detection (TCD) (Koh et al. 2007). These instruments are typically nonportable and require bottled gas. Micro and miniature gas chromatographs have also been used for online monitoring (Terasaki et al., 2009; Kaewluan and Pipatmanomai., 2011); although they are smaller, these systems also require bottled carrier gas for operation and thus limit portability. Other online gas analyzers have been used based on infrared technology for continuous monitoring of CO and CO<sub>2</sub> (Li et al., 2004).

### *1.2 Design Considerations and Requirements*

The components, features and testing of a portable system utilizing an automatic gas sampling loop and a metal oxide detector are detailed below for the monitoring of the concentrations of hydrogen, carbon monoxide and small hydrocarbons produced during the gasification process. Intelligent control is needed to consistently manage the biomass gasification process to yield a syngas composition matching downstream process needs and preventing excessive tar levels and unwanted emissions. Most of the parameters affecting product composition can be readily controlled but the feedstock composition and moisture content are naturally variable and very difficult to measure continuously. Because of the varied composition of biomass, refinery output is highly sensitive to the naturally variable feedstock characteristics. A robust, feed-stock composition sensitive control system would greatly improve biorefinery efficiency.

Specific application requirements including continuous automatic operation, portability, low cost and small size guided the design and choice of components for the autosampling gas chromatograph described here. The prototype monitor was designed and assembled to continuously monitor the presence of combustible gases associated with the production of synthesis gas, specifically carbon monoxide and hydrogen. Noncombustible gases associated with the process such as nitrogen, oxygen and carbon dioxide do not participate in most biofuel production schemes (for example, Fischer Tropsch) and are not monitored with this gas chromatograph. For improved portability and ease of use, the system was designed with the ability to use ambient air as a carrier gas. This feature requires that the sensors and the chromatography column are stable in air for extended periods of time. An automated 6-port valve with a commercial gas sampling loop allows for continuous unmanned operation. Two different transducers were utilized in the prototype chromatograph including a metal oxide semiconductor (MOS) detector (Figaro USA, Arlington Heights, IL) (Gong. 2005) and a MEMS polymer-filled chemicapacitor array

(Seacoast Science, Inc., Carlsbad, CA) (Patel et al., 2008). Thus the assembled system is suitable for monitoring of both combustible gases (MOS) and volatile organic liquids (chemocapacitors). The inclusion of orthogonal transducer technologies expands the system's capabilities. However for the work presented here, only the permanent gas analytes were considered and thus the only data presented is from the metal oxide sensors. Testing and evaluation of the gas chromatograph continues with volatile organic compounds and will be the subject of a future manuscript. MOS detectors have been used with portable GC systems for VOCs detection (Zampolli et al., 2005; Zampolli et al., 2009) but to the best of our knowledge this work represents the first system described for continuous monitoring of hydrogen and carbon monoxide.

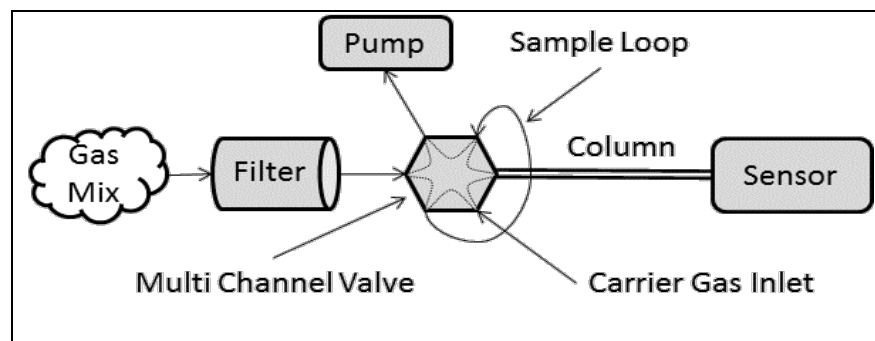
Metal oxide-based detectors were chosen as transducers for the gases because they operate well in ambient air, are a mature technology (Mlsna et al., 2010), and are commercially available. A MOS detector is generally comprised of a semiconducting sensitive layer with an integrated heater. An electrical connection is used to measure the resistance of that layer at elevated temperatures. When the surface is exposed to the exhaust of the biomass gasifier, oxidizable gases such as hydrogen, methane and carbon monoxide lower the surface potential, and the conductivity measurably increases. As the concentration of the gas increases so does the change in resistance; thus, these devices can be used to quantify chemicals. MOS sensors have the added advantage of being small and low cost, and having low power requirements.

The syngas monitoring system was designed to produce baseline separation of the target analytes without heating or cooling. Time was also considered to be a factor with a goal of approximately 10 chromatographs per hour or operation on a 6-minute cycle. Eliminating the requirements of an oven or cooling hardware resulted in the significant size, power and cost advantages and reduced cycle times.

## **2. Experimental**

### *2.1 Chromatograph system design*

As shown in the Fig. 1 block diagram, the synthesis gas monitoring system consists of a gas inlet with filter, a multi-position valve with a gas sampling loop, a packed chromatographic column and a detector. Not shown are the electronics and laptop computer for system control and display. This prototype GC requires a pressurized carrier gas input (40 psi resulting in a flow rate of  $\approx 10\text{mL}/\text{min}$ ) to carry the analytes through the system. This can be accomplished with an internal gas pump or portable gas compressor. Thus, the system is designed to pull in a gas sample with a pump for analysis and accept a pressurized carrier gas input. The MOS responds to combustible gases like carbon monoxide, hydrogen and methane. The multi-position micro volume gas sampling valve allows for the collection of analyte gas in a 1 or  $10\mu\text{L}$  sample loop. Included is a ceramic filter for removal of tars and other particles from the sample gas to protect the column and sensors. All components were assembled in a weather-proof polyethylene Pelican™ case as shown in Fig. 2.



**Fig 1.** Schematic illustration of the autosampling gas chromatograph



**Fig 2.** GC in a weather-proof case (13.5" X 7.5" X 12").

## 2.2. Components for system fabrication

The electrically actuated multi-position gas sampling valve (part #EHC6WE) was purchased from Vici Valco Instruments Co, Inc. (Houston, TX). The precise 1  $\mu$ L or 10  $\mu$ L volume gas collection sample loop (part #SL10NW) was also purchased from Vici Valco. The miniature gas pump (part #A18418A) used to fill the gas sampling loop was purchased from Koge Electronics. Restek (Bellefonte, PA, USA) provided the 100/120 mesh packed 2 m long chromatographic ShinCarbon ST Column. The MOS detector was purchased from Figaro (part # TGS2620) (Arlington Heights, IL, USA). The MOS detector is housed in a small volume (approximately 0.2 ml) gas tight housing attached to the exhaust of the chromatographic column. Ceramic filters were purchased from Anver Corp of Hudson, MA (FLTP-1/8F). The weather-proof Polyethylene case was purchased from Pelican Products Inc. A LabVIEW (National Instruments, Inc., Austin, TX) software application was developed to control the gas chromatograph components. Seacoast Science designed and fabricated a custom control circuit to manage the various systems and collect data.

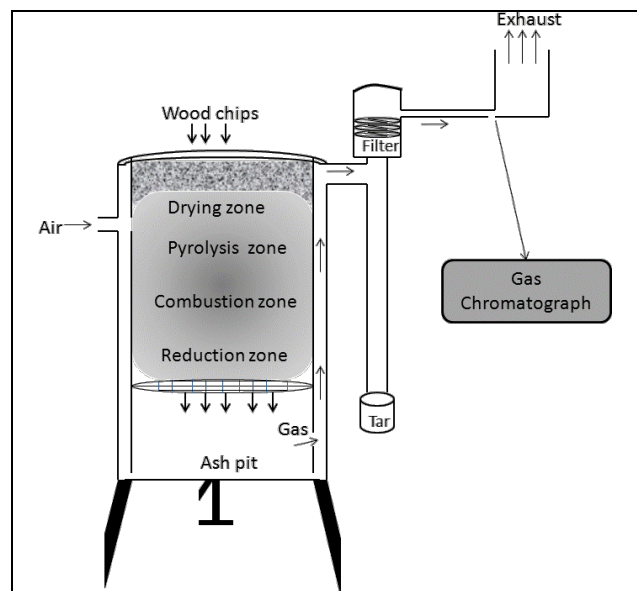
### 2.3. Test gases

#### 2.3.1 Lab Tests

Pure gases (hydrogen, methane, carbon monoxide, carbon dioxide and nitrogen), and various mixtures of carbon monoxide, carbon dioxide, hydrogen, methane and nitrogen were provided by Airgas Company. Other custom mixtures (0.8% hydrogen and 1.5% methane in nitrogen, 0.8% hydrogen and 3.5% methane in nitrogen) including various concentration of hydrogen in a balance of nitrogen for calibration were prepared by the authors using pressurized gas transfer lines. One liter Tedlar bags with 2-in-1 polypropylene fittings were purchased from Cel Scientific Company (Santa Fe Springs, CA) filled with the pure gases or gas mixtures and attached to the mini gas chromatograph for analysis.

#### 2.3.2 Pilot Scale Tests

The downdraft gasifier used in this work was a GEK Gasifier purchased from All Powers Labs, Berkeley, CA. While the gasifier operated, the GC collected samples off of a steady stream of syngas mixture (Fig. 3). The biomass used in this program was dried pine woodchips supplied by Mississippi State University.



**Fig 3.** GC sampling from the field gasifier.

### 2.4 Software Systems

Custom software to operate the mini gas chromatograph was developed using LabVIEW software. The developed software controls GC operation, including the operation sequence and collects sensor output and displays the results. The software also controls the electrically actuated gas sampling valve allowing for automated unmanned operation.

### 2.5 System Operation

As displayed in Fig. 1, the two-position six-port low volume valve is used to collect and inject a sample into the GC. The normally open position of the valve allowed the sample to be pulled into

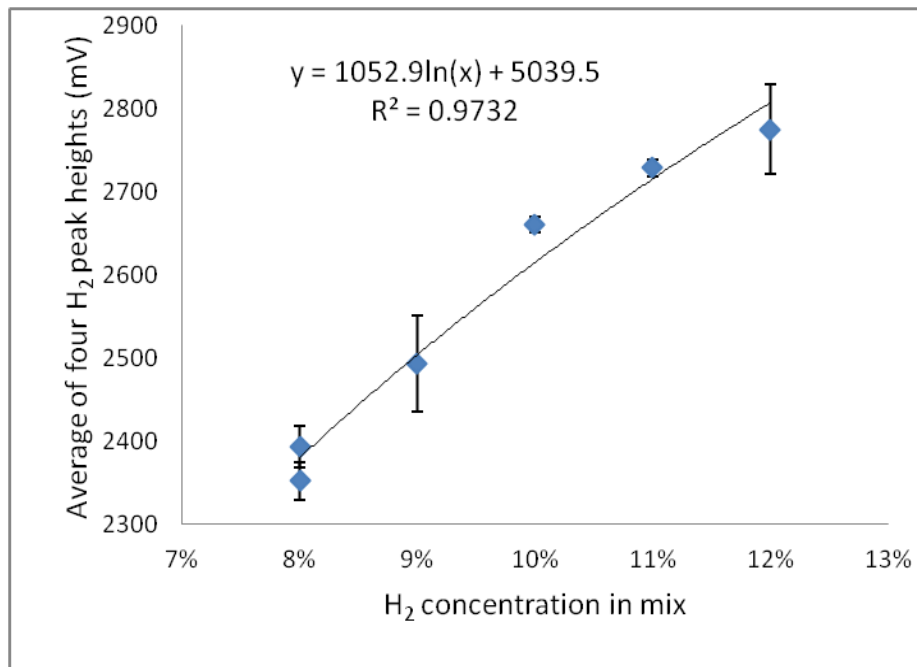
the 10  $\mu\text{L}$  sample loop by the vacuum pump through a filter while at the same time the carrier gas (compressed air) is pushed through the valve onto the column. When the valve is switched, whatever gas that had been collected in the sample loop is pushed on the column in approximately 1 second by the clean air stream and the vacuum pump pulls the gas mix through the filter directly to exhaust, bypassing the sample loop. The valve quickly repositions and the process is repeated at a programmed time interval.

### 3. Results and discussion

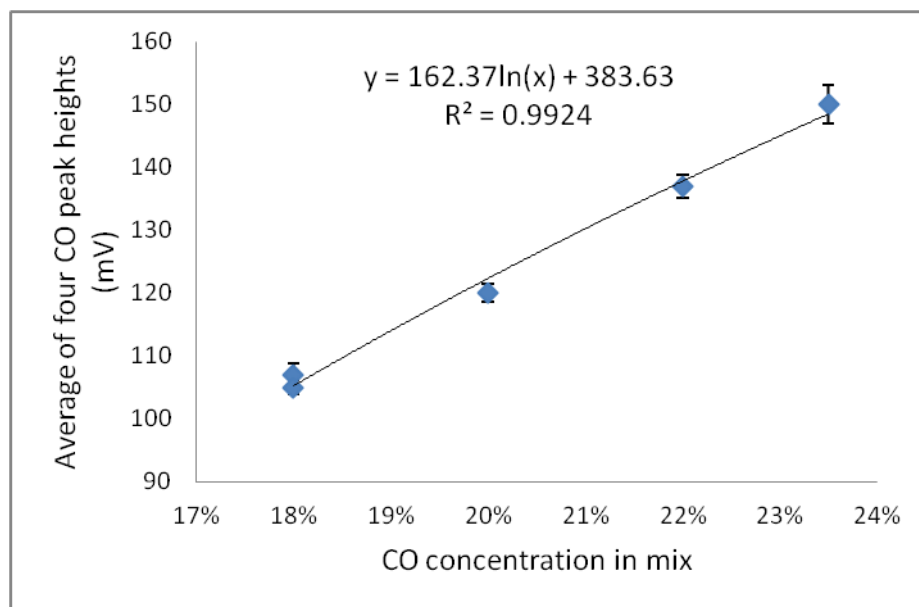
#### 3.1 Laboratory Tests Standards

Laboratory testing was completed in order to evaluate the system response to gases alone and in combinations. In a typical test the mini gas chromatograph was set up to go through 6 repeated analysis cycles. MOS sensors measure the change in resistance when an analyte interacts with the detector. The DC resistance of the MOS detector was measured using the recommended circuit in the Figaro specification sheet (<http://www.figarosensor.com/products/2620pdf.pdf>). The voltage drop across a resistor in series with the detector was used to measure the current through the detector and the voltage was measured by the microprocessor. A constant 5V DC was used across both resistors before being recorded and displayed.

#### 3.1.1 System calibration



(a)



(b)

**Fig 4.** Calibration curves for hydrogen (a) and carbon monoxide (b) using peak height

The gas chromatograph was calibrated using the automatic gas sampling loop (1.0  $\mu\text{L}$  or 10  $\mu\text{L}$ ) to collect and inject a range of concentrations of hydrogen, carbon monoxide and methane diluted in nitrogen. The results indicated that peak height (Figure set 4) or areas (Figure 5) obtained from these chromatographs could be used to obtain calibration curves. The calibration curves for the three test gases fit a logarithmic trend line best. The  $R^2$  values included with the graphs indicate a good fit for the data with the peak area providing the best fit. Each data point represents the average peak height over four consecutive samples at each concentration. The error bars on each data point represent the standard deviation of the numerous samples.

The calibration curve fits for Figures 4 and 5 demonstrate that the prototype gas chromatograph can be used for quantification. Similar to most analytical instruments, for best results, the gas chromatograph should be calibrated approximately once a week or when test conditions and analytes change. For the following data, the systems were optimized for each particular experiment to achieve baseline separation and recalibrated when required for quantification.

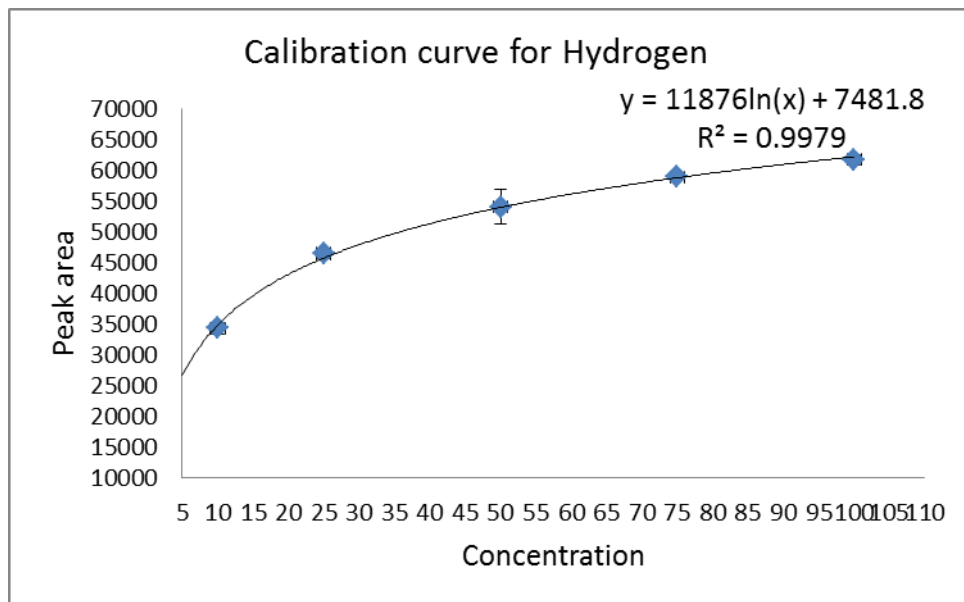


Fig 5. Calibration curve for hydrogen using peak area

### 3.1.2 Mixtures of gases

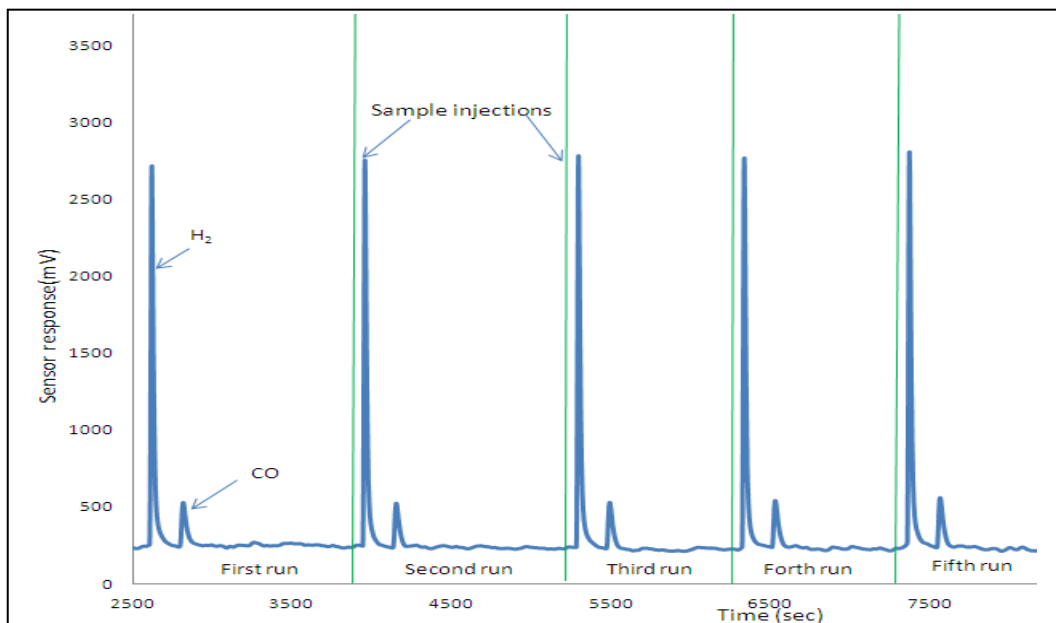
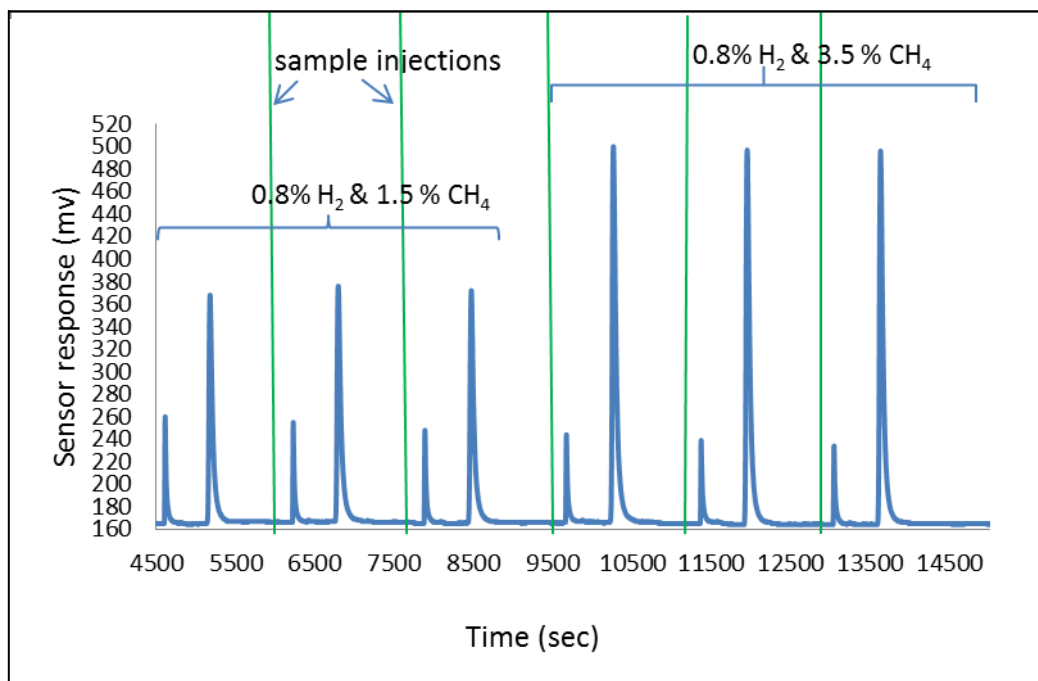


Fig 6a. Hydrogen/ Carbon monoxide/ nitrogen (48%/48%/4%) mixture sampled using the 1  $\mu$ L injection loop.



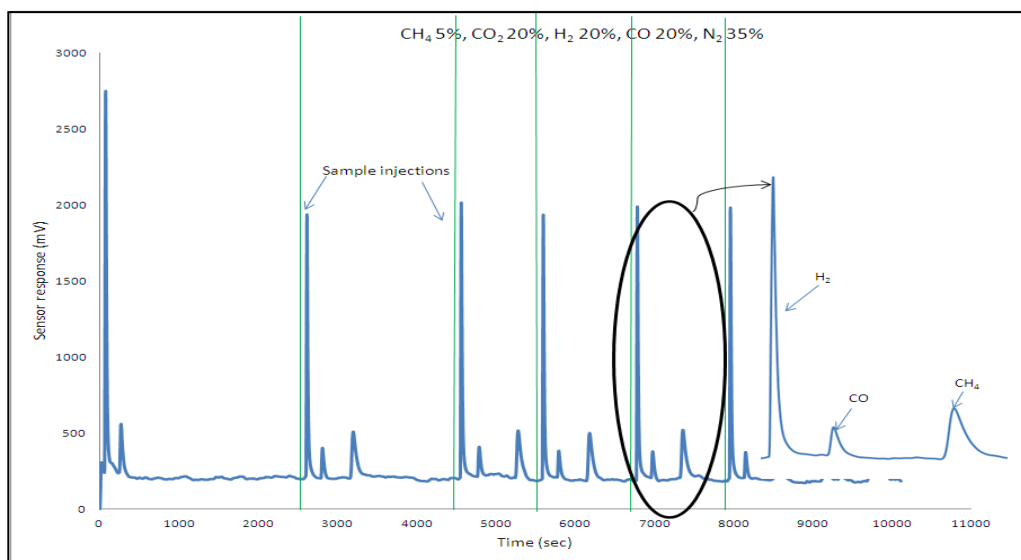


**Fig 6b.** Hydrogen and methane mixture in nitrogen sampled using the 10  $\mu$ L injection loop.

A series of high concentration gas mixtures were tested (Fig. 6a) using a 1  $\mu$ L sampling loop while the lower concentration tests and the calibration using the standards were performed with a larger 10  $\mu$ L loop. Figure set 6 shows the gas chromatograph's response to varied mixtures containing combinations of hydrogen, carbon monoxide and methane. Figure 6a is a chromatogram containing a series of six consecutive injections and repeated chromatographic cycles from a mixture of 48% hydrogen, 48% carbon monoxide in nitrogen. Due to the high concentrations used in this mixture, a more linear response was obtained using the 1  $\mu$ L volume injection. This one to one mixture spectrum exhibits good system and sensor reproducibility, and the detector's significant selectivity for hydrogen over carbon monoxide, which is consistent with this class of metal oxide detectors. This selectivity to hydrogen gas is due in part to the partially oxidized state of carbon monoxide and to the relatively narrow width of the eluting hydrogen gas.

In Figure 6b the concentrations of individual gases within the mix were varied. Six consecutive samples of a mix of 8% H<sub>2</sub>, 18% CO and CH<sub>4</sub> in nitrogen were injected. The first three samples have 1% CH<sub>4</sub> and the last three have 3.5% CH<sub>4</sub>. The result shows clear separation, precise elution times, reproducible peak heights, and a clear increase in response magnitude with increased CH<sub>4</sub> concentration. The response of the GC's MOS detector to the methane is consistent at each concentration and an increase in peak height is clearly observed as the concentration is increased. This test was run with nitrogen as the balance of the sample gas and with 40 psi air as the carrier resulting in a flow of approximately 10ml/min.

Figure 6c shows several chromatograms for a mixture consisting of 20% hydrogen, 20% carbon monoxide, 5% methane, 20% carbon dioxide and 35% nitrogen.



**Fig 6c.** MOS output for Hydrogen, carbon monoxide and methane mixture in nitrogen sampled using the 10  $\mu$ L injection loop.

There is no sensor response to carbon dioxide. The retention times and standard deviation of the hydrogen, carbon monoxide and methane are given in Table 1 and exhibit good reproducibility with standard deviations of less than 1 second. The chemicals elute in order of boiling point (hydrogen - 253°C, carbon monoxide -191.5 C and methane -187°C).

### 3.1.3 Results of laboratory calibrations

Traditional analysis of the chromatograms (Skoog, 2007; Harris, 2010) was done to determine the quality of the chromatographic separations for qualitative and quantitative analysis using the unusual combination of the MOS detector and air as the mobile phase. Calculations were done to determine peak separation and resolution and column plates and plate height.

#### *Unadjusted relative retention*

A closer look at the three detectable gases used in the mixed gas experiments demonstrates the utility of the system when operated under the above conditions. Adequate separation was achieved as is evident from the unadjusted relative retention ( $\gamma$ ) calculated using Equation 2 where  $t_1$  and  $t_2$  are the retention times for the neighboring eluting chemicals.

$$\gamma = t_2/t_1 \quad (2)$$

Using the data from Table 1 in Equation 2 results in  $\gamma$  values of 3.6 for CO/H<sub>2</sub> and 2.4 for CH<sub>4</sub>/CO. This differs from results obtained with helium as the mobile phase (6.5 for CO/H<sub>2</sub> and 1.5 for CH<sub>4</sub>/CO) but still allows for baseline separation and analyte quantification within our 6 minute cycle target. Elution time differences and peak width are two important factors that contribute to how well peaks can be used for quantification. Resolution combines peak width and retention to quantify chromatographic quality.

### Resolution

The resolution of neighboring peaks was calculated using Equation 3. Where  $\Delta t_r$  is the separation in seconds between the neighboring peaks and  $w_{av}$  is the average width in seconds at the baseline between tangents drawn to the steepest parts of the peaks.

$$\text{Resolution} = \Delta t_r / w_{av} \quad (3)$$

**Table 1** Retention Times of a synthesis gas mix.

injection	H <sub>2</sub> (s)	CO (s)	CH <sub>4</sub> (s)
1	39	131	286
2	40	133	287
3	40	131	287
4	38	132	284
5	38	132	284
6	39	132	286
Average	39	131.8	285.7
St. dev	0.9	0.8	1.4

The resolution values for hydrogen and carbon monoxide were 8.4 and 9.1 for carbon monoxide and methane using air as the mobile phase. Improved resolution values for hydrogen and carbon monoxide of 13.3 were obtained using the 8610C SRI GC (20720 Earl St., Torrance, CA 90503, USA) with the same packed column operating with helium as the carrier. Both instruments fall well within the acceptable range where a value greater than 1.5 is considered highly desirable (Skoog, 2007) for quantitative analysis.

### Number of Plates and Plate Height

Although using ambient air as a carrier gas significantly improves portability it is expected to detrimentally affect the column efficiency compared to helium due to increased molecular size and effort required for the analyte to diffuse through the mobile phase (Whiting et al. 2001). The efficiency of the column was evaluated by determining the theoretical number of plates (N), using Equation 4 and the plate height (H), using Equation 5. Where  $t_r$  is the unadjusted retention time for the eluting chemical,  $w$  is the width measured at half height (seconds) of a peak and  $L$  is the column length. The results from these calculations using both the autosampling GC and SRI GC can be found in Tables 2 and 3, respectively.

$$N = 16(t_r)^2/w^2 \quad (4)$$

$$H = L/N \quad (5)$$

Narrower eluting bands and longer eluting times correspond to greater number of theoretical plates and smaller plate heights. Plate height varies with several factors including instrument design, mobile phase and temperature profile and is approximately the length of column required for one equilibration of solute between the mobile and stationary phases. The plate height test results using the portable system did show an increase compared to results from a tradition gas chromatograph using helium as a carrier with an FID detector. For example, tests for methane

resulted in an  $H = 0.45$  (air carrier and MOS detector) compared to 0.02 (helium carrier and FID detector).

**Table 2** Plate height and theoretical plates from portable system

	H <sub>2</sub>	CO	CH <sub>4</sub>
Retention time (s)	40	132	287
Width (s)	16.5	27	25
N	94	382	435
L (cm)	200	200	200
H (cm)	2.1	0.52	0.45

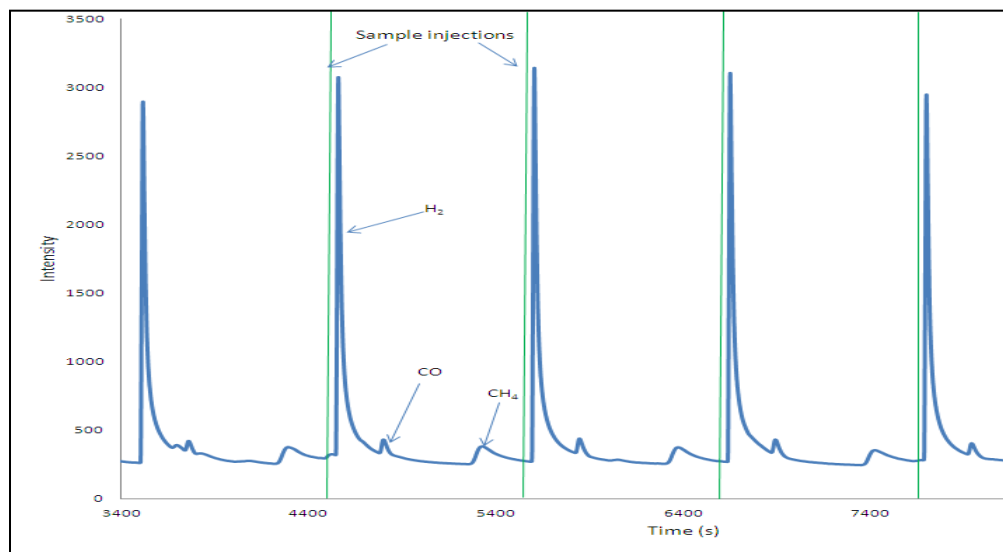
**Table 3** Plate height and theoretical plates obtained from benchtop GC

	H <sub>2</sub>	CO
Retention time (s)	48.6	116.4
Width (s)	5.5	4.7
N	1249	9940
L (cm)	200	200
H (cm)	0.16	0.020

As demonstrated here, this difference was not enough to preclude analyte quantification with the portable. Again we see improvements with the commercial bench-top instruments but the described portable system performs well enough for online process monitoring during bio syngas production.

### 3.2 Pilot-scale Tests

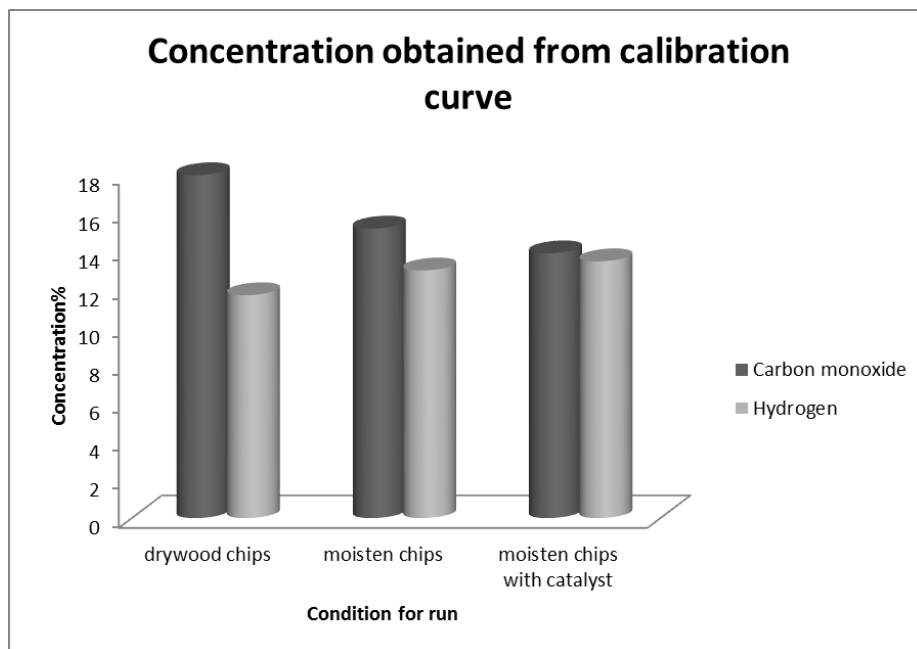
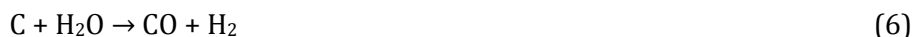
#### 3.2.1 Changing moisture content and the water gas shift reaction



**Fig 7.** Typical chromatograph showing 5 cycles from pilot gasifier

Pilot testing was done by producing syngas using the GEK gasifier described above and monitoring of the production gas composition with the portable GC in order to determine if changes in the gasifier operation could be determined in real-time. Dried pine woodchips were used with moisture content levels ranging from 7.9% to 10.3% by mass. The gasifier was operated under various conditions in order to change and monitor the composition of the synthesis gas produced. A typical run can be seen in Figure 7, which shows 5 cycles of sample loop collection, injection and analysis.

Two different approaches were used to vary the synthesis gas composition in order to evaluate how the gas chromatograph would respond. In one set of experiments the aim was to increase the production of hydrogen through the addition of water by soaking the wood chips in water before gasification. Addition of water can affect hydrogen production in two different ways. As water travels through the reduction zone in the gasifier it reacts with carbon at elevated temperatures to produce carbon monoxide and hydrogen (Equation 6). Water can also react with carbon monoxide producing carbon dioxide and hydrogen, which is known as the water gas shift reaction (Equation 7). Promotion of the water gas shift reaction can be accomplished through the addition of a catalyst.

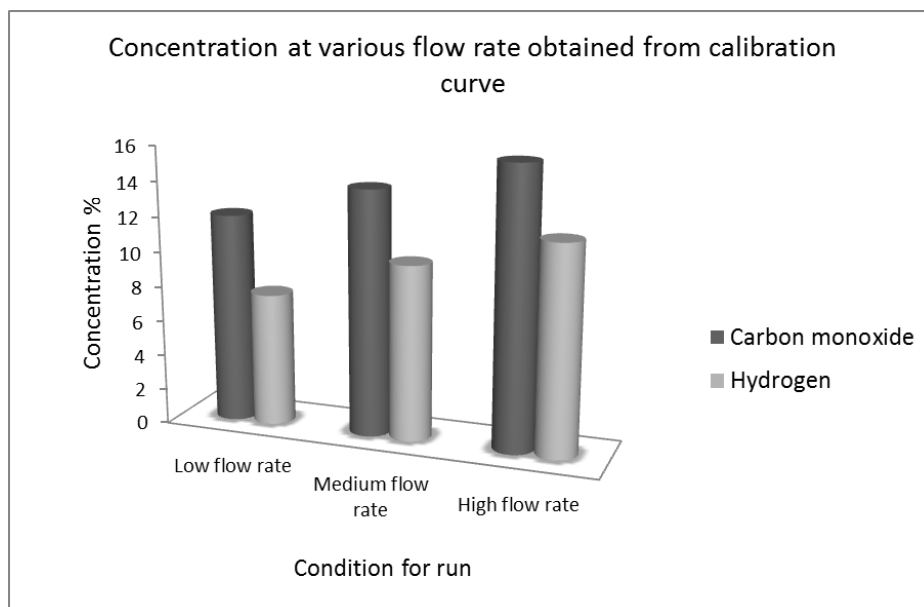


**Fig 8.** Peak area percentage of hydrogen relative to carbon monoxide from gasification and standard sample

A water gas shift reaction catalyst (Hifuel W220 copper-based water gas shift reaction catalyst from Alfa Aesar) was used in the gasifier during biomass gasification. To promote this reaction, 30 g of catalyst was placed in a reduction zone of the gasifier. Three related experiments were conducted detecting the effluent from the gasifier. The first used dry wood chips, the second used wood chips soaked in water and the third used wet chips and a water gas shift catalyst. The addition of water and the addition of the water gas shift catalyst caused an increase in hydrogen gas and a decrease in carbon monoxide production (Figure 8). It is observed that the hydrogen to carbon monoxide ratio increases from dry wood chips to wet wood chips to wet wood chips with the water gas shift catalyst. The concentration ratios in Figure 8 were calculated from the calibration curves (Figure 4)

### 3.2.2 Modifying reactor flow

Air flow rates also affect the production of fuel gas and was monitored during the gasification process. Woodchips, containing 10.3% moisture, were used for this set of tests. Three different flow rates were maintained for three batch gasification runs (Figure 9). The low flow rate was maintained between 1-2 L/min, medium flow rate was between 10-12 L/min and the high flow rate was between 27.5-29.5 L/min. The ratio of hydrogen to carbon monoxide did not significantly change with varied gasifier flow. However, the concentrations of both hydrogen and carbon monoxide increased with flow relative to the other gases that comprise syngas.



**Fig 9.** The ratio of carbon monoxide to hydrogen did not significantly change with gasifier flow rate but the concentration of both increases with increasing flow.

## 4. Conclusion

Here we described the assembly and evaluation of a simple gas chromatograph for the inline monitoring of biomass gasification. The system was calibrated for the gases of interest including

hydrogen, carbon monoxide and methane. Results indicated a good logarithmic calibration fit for the quantification of hydrogen, methane and carbon monoxide using either peak area or peak height. Repeated injections show a standard deviation in retention times of near one second demonstrating good system reproducibility.

Comparisons were done with a benchtop chromatograph using helium as the mobile phase. Result show that even though chromatographic factors were somewhat reduced in quality the described system with air as a mobile phase provided acceptable results. The cycle times of approximately five minutes were adequate for relative retentions over 2.4 and peak resolution values over 8. The system demonstrated that the number of theoretical plate and plate heights were sufficient under the operating conditions used in this study for repeated analyte quantification using the 2 meter packed column. Pilot plant testing demonstrated that the system could be operated outdoors and could track variations in the syngas mixture due to changes in feedstock moisture content and in gasifier flow rate.

The simple design described here performed well in a laboratory environment over hundreds of cycles and continued to operate well during several months of pilot plant testing proving to be a very robust system. The only maintenance required was that the 1  $\mu$ L sample loop would occasionally become blocked required cleaning. The 10  $\mu$ L loop, which was predominantly utilized, did not suffer from clogging. Future work will focus on integrating the gas chromatograph with gasification control hardware to automate the production of syngas.

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