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Characterization of the Effect of Reaction Residence Time on CO_2 Gasification of Empty Fruit Bunch (EFB)

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ABSTRACT

Empty Fruit Bunch (EFB) is a lignocellulosic biomass residue from extraction and processing of palm oil, which is rich in lignin, cellulose and hemicellulose that makes it suitable to be used to produce syngas through CO_2 gasification process. The reaction residence time of EFB biomass species in gasification reactions critically influences the CO_2 conversion, quality of syngas composition, the carbon efficiency of overall cold gas and tar formation. This paper investigates the effect of reaction residence time on CO_2 gasification of EFB biomass using a 2-inch quartz tube furnace reactor. Three different weights: 20g, 30g and 40g of EFB biomass samples equivalent to reactor bed heights of 9, 14 and 19cm were gasified at temperatures of 700, 800 and 900°C. A constant flowrate of CO_2 of 50ml/min was passed through the three different bed heights, resulting in an increase in residence time from 229s to 459s. This study demonstrated the influence of residence times over CO_2 conversion using variation of bed temperatures and feedstock reactor bed heights on the quality of the syngas obtained. The results showed that higher resident times and temperatures enhanced the reverse Boudouard gasification reaction and the formation of CO from 20 to 93% and H_2 from 50 to 64% in syngas, whereby the material reactivity increased from 0.0968g/min to 0.369g/min. Therefore, the increase in the reaction residence time and temperature enhanced CO_2 gasification considerably and promoted syngas production quality with LHV increase from 3.607MJ/m^3 to 14.106MJ/m^3.

1. Introduction

Thermochemical conversion process is one of the most efficient lignocellulosic biomass valorisations to produce energy, biofuels or chemicals. So far, several thermochemical conversion methods have been used, such as incineration, torrefaction, pyrolysis, hydrothermal liquefaction and

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gasification [1]. Globally, the utilization of highly distributed, low-value, and diverse, lignocellulosic biomass such as Empty fruit bunch (EFB) have been readily converted through CO_2 gasification to produce syngas fuel and chemical feedstock. However, more research is needed to improve syngas quality for its commercial uses in high energy-efficient heat and power generators such as gas turbines or fuel cells, and the production of liquid fuels and H_2 [2]. The study on performance of an effectively integrated biomass multi-stage gasification system shows that up to 13% increment of the gasifier system energy efficiency is observed and fuel switching results in 10% lower flue gas loss and improved furnace efficiency [3]. Another study on the application of the developed installation for plasma gasification of organic waste led to the formation of high calorific-value gases [4].

The gasification process and syngas composition are strongly influenced by the reaction residence time (RTD) which generally increases hydrogen yield, carbon conversion, and overall gasification efficiency, while shorter times favour incomplete reactions and higher tar content. An experimental demonstration of RTD diagnostics on a photochemical reactor is performed to identify the most practical locations for the inlet/outlet pipes (axial or radial) and the photochemical reactor's ideal working posture (horizontal, vertical, or inclined) and to understand the level of mixing and determine the fluid flow defects [5]. As such, a one-dimensional kinetic model was developed to simulate the effects of residence time and heating rate on the gasification characteristics and results showed that gasification under higher residence times and heating rate improves the hydrogen yield and carbon conversion efficiency [6]. Another study on kinetic modelling and analysis of CaO enabled biomass gasification was performed to analyse the effect of steam to residence time, sorbent addition and biomass ratio on syngas composition in MATLAB platform, which shows that the developed model can be used to predict the effect of residence time on syngas composition for air–steam biomass gasification [7]. A comprehensive review of biomass gasification characteristics in circulating fluidized bed (CFB) reactors reveals also that particle residence time is closely related to carbon conversion rate and ash residue. As such, too short of a residence time prevents biomass coke from fully participating in the gasification reaction and thus resulting in a decrease in carbon conversion rate, while too long of a residence time may cause ash melting and slagging, destroying fluidization stability. Therefore, optimizing the above flow parameters is the key to improving CFB gasification performance, and multi-scale regulation is required to achieve gas–solid synergy [8]. Similarly, another study presented a method for obtaining the residence time required for complete conversion of biomass in a bubbling fluidized bed using the time-variation of temperature and pressure in the bed [9]. The pyrolysis of woody biomass waste in a continuous screw reactor shows that the higher the peak temperature and the residence time, the higher the CO and H_2 content and the lower the CO_2 content. This makes them valuable for energy production, like hydrogen sources, synthesis gas or reducer making the production of quality charcoal from biomass more profitable from the economic point of view and environmentally more sustainable [10]. Furthermore, an investigation on the pyrolysis characteristics of EFB was achieved by analysing product properties based on study reaction temperature and heating rate [11]. In another study, the residence time of the hydrothermal carbonization process was successfully investigated but it did not significantly affect the physical and chemical properties of hydro-char considering the fuel properties of hydro-char [12].

EFB is the major most efficient lignocellulosic biomass by-product from the palm oil industry and has great potential as primary feedstock used for production of alternative energy fuels such as bio-syngas, bio-oil and char [13,14]. Additionally, it is established that the catalytic gasification of EFB can be utilized to produce environmentally friendly fuel synthetic gas as a source of energy was tested with wide range of catalytic materials such as natural bentonite [15], iron and aluminium metal pillared bentonite[16] catalysts. The simulation study of gasification of EFB for electricity generation using super-pro designer simulation software shows that the biomass feed stream of zero-point five metric tons of air, two metric tons per hour of EFB and one metric ton per hour of steam feed were enough to generate about 8246 kWh of electricity [17]. In similar study, co-gasification of EFB was performed to optimize syngas production and is receiving researchers attention in recent times, in this regard, steady state modelling simulation using Aspen Plus for the gasification of palm oil EFB in pilot plant downdraft reactor suggests that co-gasification of feedstock have a significant potential to overcome the problem of disrupted feedstock supply in gasification [18]. Additionally, EFB waste was successfully utilized through a two-step process (pelletization and microwave pyrolysis) to produce biochar as an alternative renewable energy approaching commercial standards [19].

Thermal gasification remains a well-established and reliable technique in production of syngas fuel and chemical feedstock; however, challenges such as uneven heat distribution and tar formation require careful process control and parameter optimization. In addition, the characterization of residence time effects in CO_2 gasification of EFB remains underexplored in peer-reviewed literature. Therefore, the current study will look at the influence of several gasification conditions and parameter variations of temperatures, reactor bed heights along their corresponding resident times to investigate CO_2 gasification process of EFB. This study is a step forward towards achieving production of high yield syngas from EFB biomass residue extraction and processing of palm oil.

2. Materials and Methods

2.1 Materials

Empty fruit bunch is the biomass feedstock used for this work and was collected from United Oil Palm Industries Sdn. Bhd, located in Nibong Tebal, Penang, Malaysia. To reduce the moisture content of EFB, pre-processing through washing was carried out with tap water to remove undesired compounds, then a further drying process was performed using natural sunlight. The EFB was spread out evenly on a clean surface and exposed to direct sunlight for at least 24 hours and this facilitates reducing the moisture content through a simple and energy-efficient manner before the biomass was subjected to controlled drying in an oven 100°C to ensure that the moisture content is reduced and consistent. The tools needed in this research are CO_2 tank, a digital flowrate controller, a monitoring unit, a vertical furnace oven, 2-inch quarts reactor tube, a multistage filtering unit and portable gas analyser.

2.2 Experimental Set Up

The gasifier utilized for this experimental work is a vertical fixed bed type furnace as shown in **Figure 1.** The gasifier is insulated to avoid heat loss and is provided with inlet part of 2" quartz reactor tube that permits EFB and the CO_2 as gasification agent, while the output is connected through multistage filtering unit to the TY-6030P NDIR Portable Syngas Analyzer for composition of gas products.

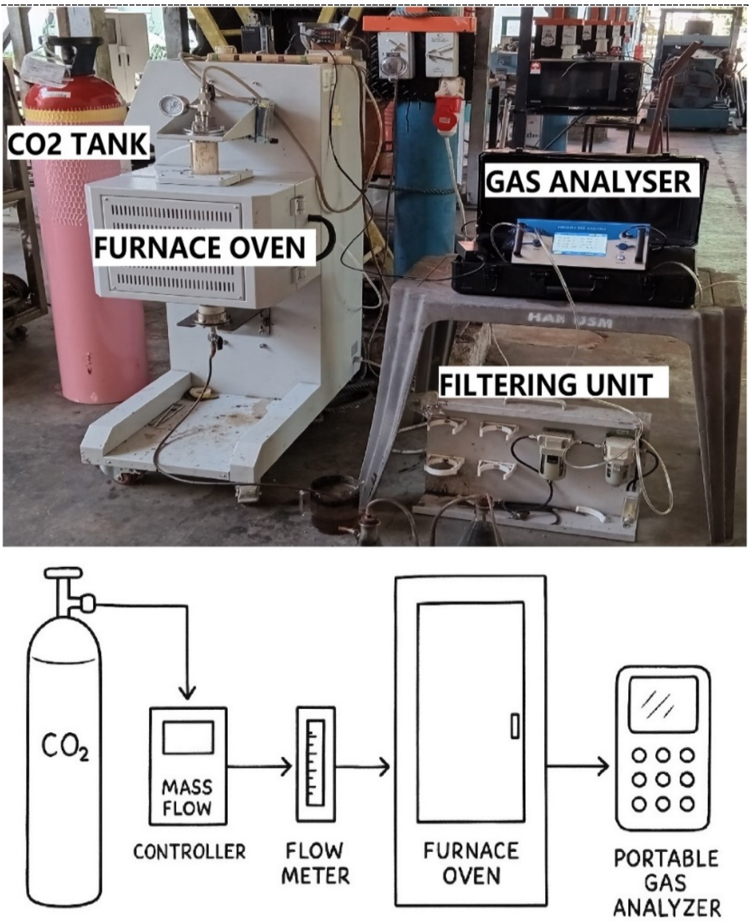

Fig. 1. The laboratory experimental set up and schematic diagram of the setup

The palm oil residue EFB sample was set at three different weights: 20g, 30g and 40g which is equivalent to reactor bed heights of 9, 14 and 19cm to permit the investigation of variations of reaction residence time of EFB biomass species in gasification reactions as shown in **Figure 2.** The gasification temperatures were set at 700, 800 and 900°C with a constant CO_2 flowrate of 50ml/min. A mass flow controller was installed to measure and control the flow rate of the gasification agent CO_2, while the pressure of gasification process is atmospheric. In this gasification process, the experimental parameters are, gasification temperatures and three different bed heights. Gasification temperatures were set at 700°C, 800°C and 900°C and the resulting syngas were analysed for the content of H_2, CO, CH_4 and CO_2 gases, and other hydrocarbon organic compounds.

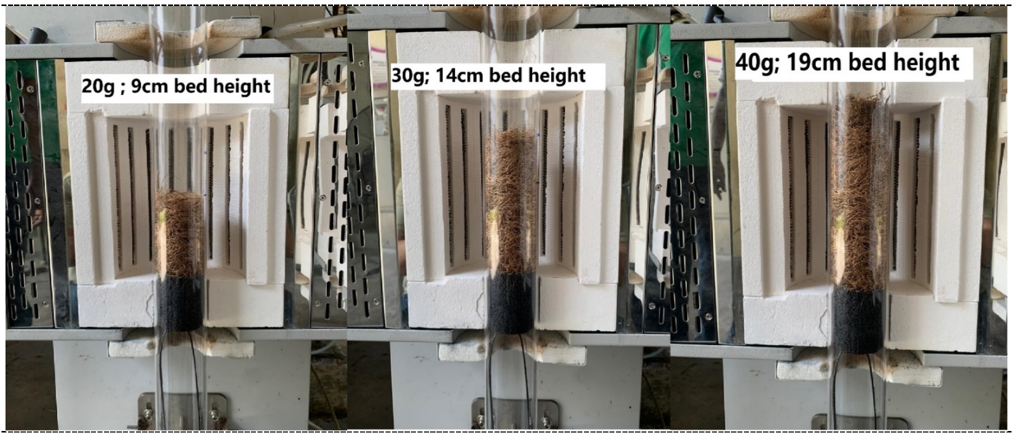

Fig. 2. Different EFB biomass feedstock masses and corresponding bed heights

2.3 Gasification Process

A prepared sample of 20g, 30g and 40 g dried EFB was loaded at separate experimental runs into the 2-inch quartz tube for each experiment making total of 9 runs. To maintain the stability and position of the sample during the gasification process, porous ceramic corks were placed at both the upper and lower ends of the biomass bed, which is to ensure the sample remained stationary even under gas flow and reduce heat losses from the feedstock top and bottom. The quartz tube was then carefully inserted into the furnace reactor chamber. A CO_2 gas cylinder was connected to the system, and gas flow was controlled using a calibrated digital mass flow controller. The gas is then inserted into the reactor through the top flange where it interacts with the EFB biomass during the gasification process. The outlet of the quartz tube was connected to a multi-stage filtration system that removes moisture, particulates, and Tar from the gas stream. The clean syngas was then directed to the in-line infrared TY-6030P NDIR Portable Syngas Analyzer for continuous composition analysis.

2.4 Characterization and Analysis of EFB

The elementary/ultimate analysis involves the determination of carbon, hydrogen, nitrogen, and Sulphur (CHNS) percentage composition in EFB samples. The Elementary analysis of EFB was performed using a CHNS/O Analyzer at the analytical lab, school of chemical engineering, engineering campus, Universiti Sains Malaysia. To determine its moisture content (MC) (dry basis), volatile matter (VM), fixed carbon (FC) and ash content, the proximate analysis of the sample was conducted using a thermo gravimetric analyzer (TGA) (model Perkin elmer, TGA pyris 1), at the school of mechanical engineering, engineering campus, Universiti Sains Malaysia. Approximately, 10mg of dried EFB was placed in a small alumina crucible and weighed while, 25°C and 1000°C were programmed as minimum and maximum oven temperatures respectively. The 30ml/min flow rate of N_2 purging was used at heating rate of 100°C /min. In addition, adiabatic YOSHIKA type bomb calorimeter, NENKEN 1013-B model was used to determine calorific value (heat of combustion) of the fuel sample at heat transfer lab, school of mechanical engineering, engineering campus, Universiti Sains Malaysia. The key measured properties of EFB, for this research is shown in **Table 1.**

Table 1
The properties of EFB

Calorific value (MJ/kg^{-1})	**Proximate analysis (%)**	**Ultimate analysis (%)**
15.732	MC = 9.93	C = 47.72
	FC = 25.73	H_2 = 5.89
	VM = 61.93	N = 0.43
	Ash = 2.42	S = 0.00

2.5 The Reactivity of the EFB Biomass

The reactivity of EFB biomass was determined by analysing the mass conversion rate of the biomass in each experimental run and it was quantified based on the rate at which the biomass was consumed, using the difference in mass before and after gasification, relative to the reaction duration. The net mass loss of the biomass during each run was calculated using **Equation 1** below:

$$\text{Net Mass Loss}, \Delta W = W_{\text{initial}} - W_{\text{residue}} \tag{1}$$

Where,

W_{initial}= Mass of EFB biomass before the run (gram)

W_{residue}= Mass of ash/char remaining after the run (gram)

To determine the reactivity in terms of how quickly the biomass reacted during gasification, the net mass loss was divided by the reaction time. This gives a direct measure of the biomass reactivity rate for each test as shown in **Equation 2**. The rate of thermal decomposition and gasification of the EFB under various combinations of the oven temperatures and CO_2 flow rate is represented by this reactivity value.

$$\text{Reactivity}\left(\frac{\text{g}}{\text{min}}\right)=\frac{\Delta\text{W}}{\text{t}} \tag{2}$$

Where t = total reaction time (minutes).

2.6 The Reaction Residence Time of EFB

Residence time in a tubular reactor is the time a gas parcel spends in the hot reaction zone. For the gasification of EFB, its calculations depend on reactor type (entrained flow, bubbling/circulating fluidized bed, fixed bed), geometry, operating conditions, and flow regime. In this work, the reaction residence time gasification of EFB has been studied and estimated using 2-inch quartz tube furnace reactor gasification systems to produce syngas, which informs typical temperatures, particle sizes, and flow regimes used for calculations of residence time. Thus, this EFB gasification operates at high temperatures of 700–900°C for entrained flow to ensure short gas/particle residence times and rapid conversion. Therefore, for a 2-inch quartz tube furnace, the residence time calculation is done based on geometry and the actual gas flow at reactor conditions as shown in **Equations 3 to 5** below

The residence time; (RT); $RT=\frac{V_{\text{void}}}{Q_{\text{actual}}}$ (3)

Where: $V_{\text{void}}=$ Volume of Void = $V_{\text{void}}=\pi\left(\frac{D}{2}\right)^2 L_{\text{void}}$ (4)

where $D=$ the inner diameter of the reactor quartz tube and

L_{void}= the effective heated length (uniform-temperature zone).

And $Q_{\text{actual}}=$ The actual volumetric flow in the hot zone = $Q_{\text{actual}}=Q_{\text{std}}\,\frac{T_{\text{hot}}}{T_{\text{std}}}\,\frac{P_{\text{std}}}{P_{\text{hot}}}$ (5)

Where $T_{\text{hot}}, P_{\text{hot}}$ are the gas temperature and pressure within the hot zone.

3. Results and Discussion

3.1 Effects of Temperature, and EFB Bed Heights on Syngas Composition

This section provides an in-depth discussion on the influence of several gasification conditions and parameter variations of temperatures, reactor bed heights along their corresponding resident times that affects the production and quality of syngas during CO_2 gasification of EFB biomass. The analysis focuses on key syngas parameters which are CO%, H_2%, CH_4%, and the Low Heating Value (LHV), observed across constant CO_2 flow rate of 50ml/min and three temperatures of 700°C, 800°C, and 900°C respectively. The results are interpreted based on plotted scatter diagrams and recorded data over time.

3.1.1 The effects on composition of CO%

The CO% concentration showed a clear increasing trend with rising temperature across the parameter variables of temperatures of 700°C, 800°C, and 900°C and corresponding reactor bed heights and EFB masses of 9, 14 and 19cm and 20g, 30g and 40g respectively. It is therefore shown that at higher resident times and temperatures, CO production increases from 20 to 93% as shown in **Figure 3**. This combination of high temperature and resident times resulted in the most reactive gasification environment among all tested conditions.

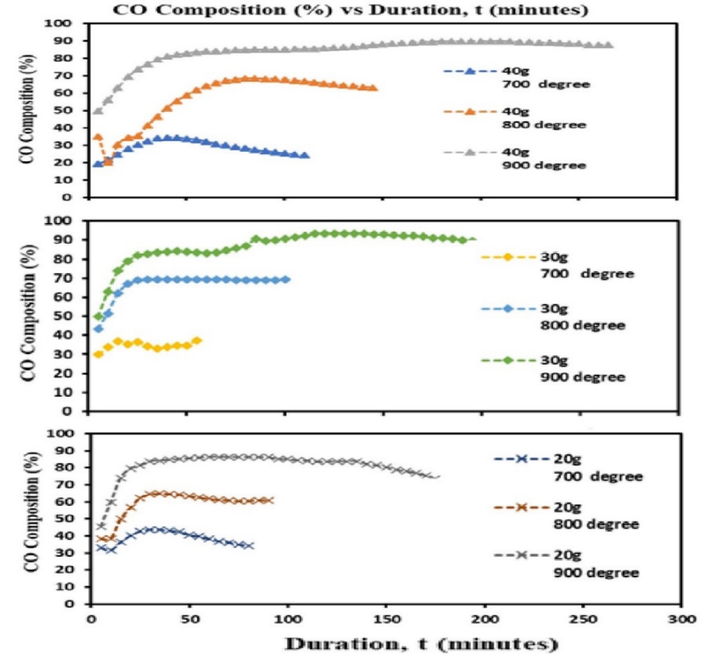

Fig. 3. CO% composition against duration of time (s)

3.1.1 *The effects on composition of H_2%*

H_2 highest production of 64% occurred within the first 50min of duration time at 30g;700°C, and the lowest at 50% at 20g;800°C before gradually declining to below 5%;900°C above 200 min duration time as shown in **Figure 4**, and this suggests that, occurrence of secondary reactions such as the water-gas shift or methanation were consuming available hydrogen at elevated temperatures and resident times.

Generally, at 900°C, the H_2 concentration remained relatively low as compared with the other two temperatures among all the tested conditions, and this trend provides the idea that higher temperatures and resident times speed up reactions that use H_2, which lowers the amount of free hydrogen in the gas that is produced. Additionally, the sharp drop in H_2 across all temperatures at this flow rate of 50ml/min suggests rapid conversion or dilution effects under higher gas velocities.

3.1.2 *The effects on composition of CH_4%*

Figure 5 shows that the CH_4% concentration is at low value of 8.53% at bed height of 19cm and increases to 16% maximum at bed height of 9cm, which occurs at low gasification temperature of 700°C, and hereby, suggests the promotion of high methanation and hydrocarbon cracking processes. Therefore, the production of CH_4 across the three bed heights shows that increase in temperatures and resident times produces decrease formation of methane at constant CO_2 flow rate.

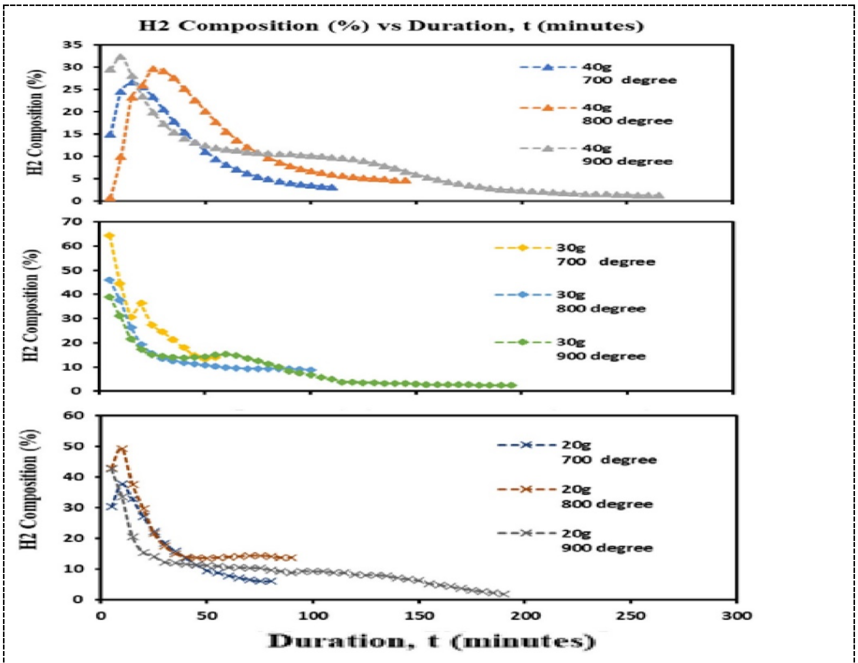

Fig. 4. H_2% composition against duration of time (s)

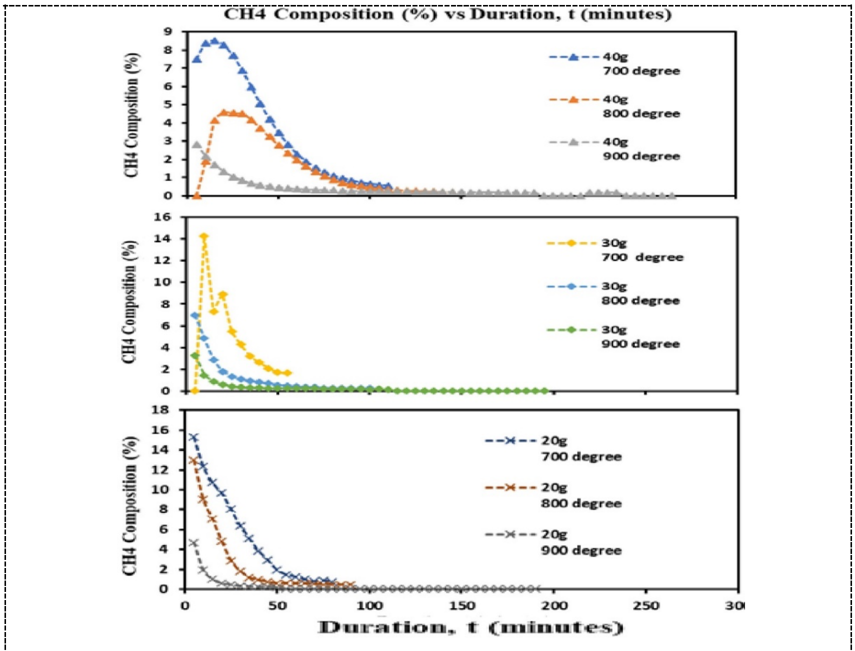

Fig. 5. CH_4% composition against duration of time (s)

3.1.2 The effects on LHV

The formation of LHV for all bed heights showed a clear increasing trend with rising temperature across the parameter variables of temperatures of 700°C, 800°C, and 900°C. However, this trend is not observed for bed heights of 9 and 14cm within the first 50min of duration time. It is therefore generally shown that at higher resident times and temperatures, the heating values increases from 3.607MJ/m^3 to 14.106MJ/m^3 as shown in **Figure 6**, and this suggests that more energy-dense syngas were produced at higher temperatures.

In addition, a steady-state plateau was observed at 800°C and 900°C after 50min duration time and these results indicate that high temperatures and resident times offer a good balance between fuel composition and heating value and demonstrate the importance of both factors in the energy output.

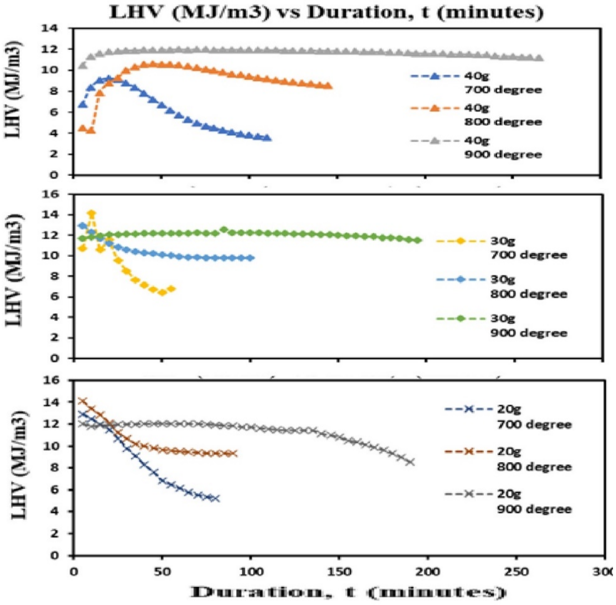

Fig. 6. of LHV (MJ/m³) against duration time (s)

3.2 Reactivity for Each Experiment

The EFB biomass reactivity was determined by dividing the initial mass of the feedstock before the experiment with the mass of its residual remaining after the experiment and multiplying the result with reaction time from the gasification process. The data generated for the calculation of reactivity of EFB is thereby shown in **Table 2.**

The highest and lowest values of 0.36927 and 0.0968g/min reactivity rates were achieved at temperatures of 700°C ;30g and 20g mass of EFB respectively, thus it reveals how fast the rate of reaction of EFB is at lower temperature and mass of EFB.

Table 2
LHV value for each variation of temperature and bed heights

Test run	Temp (°C)	$W_{Initial}$ (g)	$W_{Residue}$ (g)	ΔW (g)	Reaction Time (min)	Reactivity (g/min)
1.	700	40	9.01	30.99	110	0.28173
2.	800	40	7.89	32.11	145	0.22145
3.	900	40	3.67	36.33	265	0.13709
4.	700	30	9.69	20.31	55	0.36927
5.	800	30	6.58	23.42	100	0.23400
6.	900	30	1.79	28.21	195	0.14467
7.	700	20	5.45	14.55	80	0.18190
8.	800	20	4.47	15.53	90	0.17300
9.	900	20	0.64	19.36	200	0.09680

2.1 Estimation of Reaction Residence Time

The reaction residence time is estimated by dividing the volume of void inside the reactor tube by volumetric flowrate from the calculations using **Equations 3 to 5**. It can also be estimated by dividing the heights of bed by the actual velocity as shown in **Table 3** below.

The highest and lowest values from 459 to 229s reaction residence times were achieved at bed heights and EFB masses of 0.19 to 0.095m and 0.04 to 0.02kg of EFB respectively, thus it reveals the time gas parcel of EFB spends in the hot reaction zone at given temperatures.

Table 3
Calculation of reaction residence time

Wt. of EFB (kg)	True density (kg/m^3)	Diameter of bed (m)	Height of bed (m)	Gross density (kg/m^3)	Volume flowrate (m^3/s)	Void volume (m^3)	Percentage Void (%)	Actual Velocity (m/s)	Residence time (s)
0.04	760	0.054	0.190	91.97064	8E-07	0.00038	87.899	0.000414	459
0.03	760	0.054	0.1425	91.97064	8E-07	0.00029	87.899	0.000414	344
0.02	760	0.054	0.095	91.97064	8E-07	0.00019	87.899	0.000414	229

3. Conclusion

The investigation on the effect of reaction residence time on CO_2 gasification of EFB biomass using a 2-inch quartz tube furnace reactor was successfully performed. The experimental results from several gasification conditions and parameter variations of temperatures, reactor bed heights along their corresponding resident times to investigate CO_2 gasification process of EFB are therefore summarized as follows.

- At higher resident times and temperatures, CO production increases from 20 to 93% and this combination of high temperature and resident times resulted in the most reactive gasification environment among all tested conditions.
- H_2 highest production of 64% occurred within the first 50min of duration time at 30g;700°C, and the lowest of 50% at 20g;800°C before gradually declining to below 5%;900°C above 200 min duration time and this suggests that, occurrence of secondary reactions such as the water-gas shift or methanation were consuming available hydrogen at elevated temperatures and resident times.
- The CH_4% concentration is at low value of 8.53% at bed height of 19cm and increases to 16% maximum at bed height of 9cm, occurring at low gasification temperature of 700°C, which suggests that it promotes high methanation and hydrocarbon cracking processes.
- The formation of LHV for all bed heights showed a clear increasing trend with rising temperature across the parameter variables of temperatures of 700°C, 800°C, and 900°C, and is generally found out that at higher resident times and temperatures, the heating values increases from 3.607MJ/m^3 to 14.106MJ/m.^{3}
- The highest and lowest values of 0.36927 and 0.0968g/min reactivity rates were achieved at temperatures of 700°C ;30g and 20g mass of EFB respectively, thus it reveals how fast the rate of reaction of Empty Fruit Bunches (EFB) is at lower temperature and mass of EFB.
- The highest and lowest values from 459 to 229s reaction residence times were achieved at bed heights and EFB masses of 0.19 to 0.095m and 0.04 to 0.02kg of EFB respectively, thus it reveals the time gas parcel of EFB spends in the hot reaction zone at given temperatures.

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