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Gasification of Oil Palm Empty Fruit Bunch Fibers in Hot Compressed Water for Synthesis Gas Production

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Abstract: This study on the Hot Compressed Water (HCW) gasification of oil palm Empty Fruit Bunch (EFB) fibers was investigated in a batch system using high-pressure autoclave reactor. Reaction parameters subjected for investigation were solid loading and reaction temperature. Solid particle size, amount of water and reaction time were fixed at $250 < X < 500 \ \mu m$, $300.0 \ g$ water and $30 \ min$, respectively. The optimum reaction conditions found were $5.0 \ g$ solid loading and 380° C which produced gases mainly of CO_2 , CO, H_2 and CH_4 with gasification efficiency of 32.15% and H_2 yield of 7.22%. This study also focused on the role of a homogenous catalyst, K_2CO_3 and its effects towards the reaction. The optimal amounts identified were $3.0 \ wt.\% \ (K_2CO_3)$ with gasification efficiency achieved of 39.04% and H_2 yield 19.03%, respectively. This had proven the important role of homogenous catalyst to increase the hydrogen selectivity of gasification process and thus render the process more feasible for synthesis gas production. The results of these findings were critical towards a more sustainable usage of EFB for renewable energy generation.

Key words: Gasification, empty fruit bunch fibers, hot compressed water, synthesis gas, hydrogen gas

INTRODUCTION

The controversial soaring prices of energy and the destabilizing geopolitical events were a serious reminder of the essential role of affordable energy plays in economic growth and human development and of the vulnerability of the global energy system to supply disruption (UNDP, 2007). There is an urgent need to find and develop new green energy strategies for the sustainable development of the future with minimum impact on the environment. In regards to the environment, this new energy source should able to reduce the negative effects of fossil fuels and the overall emissions from electricity generations, decreases the greenhouse gases and meets the clean energy demand for both industrial and non-industrial applications (Midilli et al., 2005). Currently Malaysia is the second largest producer and exporter of palm oil, producing about 47.0% of the total world supply in 2007 (Basiron and Weng, 2004). This huge amount of biomass is an ideal energy source, which could be tapped for further utilization. However, the energy requirement for the oil palm mills was often much lower compare to the amount of biomass produced and thus forcing the excess to be disposed off separately (Chuah et al., 2006).

Currently, the main method of producing syngas and hydrogen is from fossil fuels through steam reforming method or gasification, which supplies majority of the production in the world. However, apart from its severe dependence on fossil fuels, this non-environmental friendly production is highly endothermic and requires a very high temperature (>800°C). This in turn caused very low in net energy efficiency (Song and Guo, 2005). Biomass has the potential as an alternative to be converted into energy via syngas production. Currently, there are numerous established methods of syngas from biomass, conversion including pyrolysis, liquefaction, combustion, pyrolysis, solar and steam gasification. The major complications faced with these technologies are often associated with its very low energy efficiency, for example, biomass combustion has a net efficiency of about 20-40% (Wang et al., 2008; Caputo et al., 2005). In addition, the current methods of production from biomass are still not economically competitive. A large portion of biomass waste is actually wet biomass containing very high percentage of water, which caused high drying costs when classical gasification process is used (Myreen et al., 2010). Therefore, gasification of biomass in HCW is a promising technology for utilizing high moisture content compounds. Although the prime disadvantage of the HCW (>300°C) gasification is its initial energy requirement for the heating up process, the components of syngas especially H2 and CH4, is substantially high in energy

content which ultimately produced a much higher thermal output. With a comprehensive energy recovery system, it is believed that it will result in high-energy conversion efficiency of the reaction (Van Rossum et al., 2009). In this case, oil palm biomass is the perfect candidate as the feedstocks for the gasification process. Its high moisture content (>60%) and insignificant amount of trace minerals in its compositions are the 2 integral requirements for reactions in HCW medium. The huge amount of biomass readily available in abundance certainly guarantees its sustainable supply allowing continuous operation of the process yearlong. With this realization, this study will focus on investigating various operating parameters in order to optimize two important responses, gasification efficiency and hydrogen yield for the gasification of oil palm EFB in hot compressed water (HCW) for the production of synthesis gas. A homogenous catalyst, K2CO3 was chosen since it was able to lower the onset temperature of cellulose degradation, increasing reaction rate and suppressed formation of tar. Previous study conducted by Kruse and Gawlik (2003) reported 100% conversion of organic compound into H₂ rich gas using the above catalyst.

MATERIALS AND METHODS

Materials: Oil palm EFB fibers were obtained from a local palm oil mill. The fibers were received in sun-dried condition with non-uniformed sized of shredded fibers. Sodium hydroxide (99%) was bought from System Chemicals, Malaysia and potassium carbonate (99.5%) was obtained from R and M Chemicals, UK. All the standard gases employed including carbon dioxide, carbon monoxide, methane, ethane, ethylene and nitrogen were purchased from Mox-Linde Gases Sdn. Bhd.

Preparation of Empty Fruit Bunch (EFB) fibers: The Laboratory Analytical Procedure for Preparation of Samples for Compositional Analysis as provided by NREL (National Renewable Energy Laboratory, USA) was utilized to ensure uniformity of the oil palm EFB fibers solid content in ambient conditions by drying in oven at 45°C for 3 days (Hames *et al.*, 2008). The oven-dried fibers were then grounded using a chopper until the entire sample passes through the 2 mm sieve screens using a vibrator sieve shaker. The grounded fibers were screened again for 15 min through a stack of sieves to obtain the desired solid particle size in the range of 250 < X < 500 μm.

Experimental procedures: The HCW gasification of the samples was investigated using a high-pressure batch reactor (BuchiGlasUster, Model "Limbo Li", Switzerland).

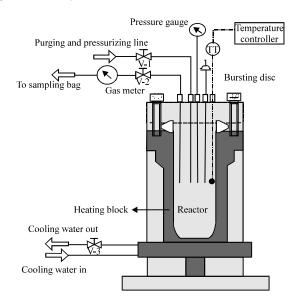


Fig. 1: Schematic diagram of the experimental rig

The schematic diagram of the experimental system is shown in Fig. 1. Pre-determined amount of oil palm EFB fibers were weighted and placed inside the 450 mL reactor together with a known volume of water. For experimental work that required the addition of catalyst, specified amount was also added into the reactor. Nitrogen gas was then used to purge the reactor before pressurized to 1 MPa. The heater was then turned on with the final temperature always reached in the shortest time possible before held for 30 min reaction time after the desired reaction temperature has been attained. At the end of each experimental run, the heating was stopped and the vessel was cooled by cooling water to ambient conditions (34°C). After cooling, the gas was released from the vessel through opening of the gas release valve into the gas meter to determine the volume of the gas produced. The product gas was subsequently collected and sampled for analysis. The liquid solid mixture in the reactor were removed and recovered for separation.

Product analysis: The sampled gases were analyzed using a GC-Gas Chromatography (Perkin Elmer, Clarius 500, USA) with Thermal Conductivity Detector (TCD). A Carboxen-1010 PLOT (porous layer open tubular) Capillary Column (Supelco, USA) with length of 30 m and inner diameter of 0.32 mm was used to analyze the product gases.

Helium gas as supplied by Mox-Linde Gases Sdn. Bhd (MOX, Selangor) was used as the carrier gas with flow rate of 1 mL min⁻¹. The oven temperature programme was 50°C at initial and subsequently ramped to 120°C at 20°C min⁻¹ and held for 15 min at that temperature. The

injector and detector temperature were held at 230°C. A standard gas mixture with compositions of all types of the product gas (CO₂, CO, CH₄, C₂H₆ and C₂H₄) and N₂ were used for calibration. Identification of each respective component in the product gases mixtures are determined by the peak retention time matching to the standard substances and quantification was performed by constructing a calibration curve. The data elucidated from GC analysis was used to determine the volume percent of each constituents and its mole %. Since the amount of N₂ from initial experimental were known (and does not react during gasification) and the molar ratio of any gas to N₂ can be determined from the GC analysis, therefore the yield of each gas species can be calculated. All this information is used to obtain the desired responses (gas yield, gasification efficiency and hydrogen yield) as given in Eq. 1, 2 and 3:

$$Gas\ yield \left(\frac{mol\ gas}{kg\ biomass}\right) = \frac{Number\ of\ gas\ mole\ (mole)}{Total\ mass\ of\ the\ dry\ feed\ (kg)} \quad \ (1)$$

$$\label{eq:Gasification} \text{Gasification efficiency (\%)} = \frac{\text{Total mass of product gas (kg)}}{\text{Total mass of the dry feed (kg)}} \times 100 \ \ (2)$$

Hydrogen yield (%) =
$$\frac{\text{Total hydrogen in product gas (kg)}}{\text{Total hydrogen in the dry feed (kg)}} \times 100$$
 (3)

RESULT AND DISCUSSION

As described in various other publications, temperature and solid loading ratio were the 2 parameters that largely influenced the gasification reaction (Hao et al., 2003; Yoshida et al., 2004; D'Jesus et al., 2005). The important responses to measure the efficiency of gasification reaction were the product gas compositions, gasification efficiency and hydrogen yield. These effects were rarely considered in the study of HCW gasification with only a few existing publications available (D'Jesus et al., 2005; Lu et al., 2006). Table 1 gives the range of value selected for each parameter used in this experimental work. Preliminary analysis had shown that the gasification reaction will reach its equilibrium within 30 min and further increment will not has significant effect towards the results.

For the industry requirement, it is always desired to process the highest biomass content as possible (Resende *et al.*, 2007). This is justified by reducing the capital and operating costs due to economy of scale. In the experimental study for the effect of biomass loading, the amount of water was always maintained at 300.0 g in excess to ensure complete reaction. Other process parameters were maintained at constant value of 340.0°C,

Table 1: Range of value selected for each parameter

Types of parameter	Range of value
Solid particle size	250 <x<500 th="" μm<=""></x<500>
Reaction time	30.0 min
Solid loading	5.0 - 30.0 g
Temperature	300.0 - 380.0°C
Catalyst	K_2CO_3

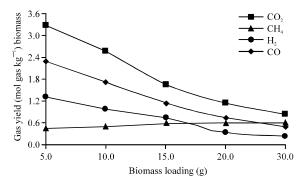


Fig. 2: Effect of biomass loading on product gas compositions for reaction condition: 340.0°C, 300 g water, 30 min and particle size 250<X<500 µm

30.0 min reaction time and particle size 250<X<500 µm. As the solid loading increased, all the product gases exhibited similar decreasing trend except for CH4 which was found to increase as shown in Fig. 2. The highest CO₂ yield was obtained at the lowest biomass loading and consistently decreased. H₂ also displayed a similar decreasing trend with highest yield at 1.31 mol gas kg⁻¹ biomass in comparison to the lowest of only 0.23 mol gas kg⁻¹ biomass. From observation of CO trend, the experimental yield was found to decrease when the biomass loading was increased. The maximum value obtained from the experimental yield was 2.28 mol gas kg⁻¹ biomass and its lowest value was about 0.49 mol gas kg⁻¹ biomass achieved at the maximum solid loading. On the other hand, CH₄ displayed an increasing trend with increasing biomass loading. At 5.0 g biomass loading, the gas yield was only 0.46 mol gas kg⁻¹ biomass and it gradually increased to the highest yield of 0.61 mol gas kg⁻¹ biomass at the highest biomass loading. Same observations were highlighted in few other publications (Yan et al., 2006; Lee et al., 2002; Kruse and Gawlik, 2003). For the lowest biomass loading, the main constituent was CO2 followed by CO, H2 and CH4. Meanwhile at the highest biomass loading it was CO₂, CH₄, CO and H₂.

The trend obtained could be explained based on the reactions that occur during the gasification. CO, CO₂ and H₂ were produced initially from steam reforming reaction between the intermediate compounds from glucose decomposition and water (Resende *et al.*, 2007). As stated by Le Chatelier's principle, when a system in chemical

equilibrium is disturbed by a change which in this case is the concentration, the equilibrium will shift in a way that tends to counteract this change. In this scenario, the excess water due to low biomass loading shifted the equilibrium of the water gas shift reaction to left, which favored the formation of H_2 and CO_2 while consuming CO. This demands the low content of CO but high content of CO at low biomass loading from theoretical calculation. However, the results from the experimental work were not consistent with the theory.

The high experimental yield of CO indicated that the water gas shift reaction had not gone to completion (Yan et al., 2006). The decrease in CO, CO2 and H2 were attributed to the higher ratio of biomass to water that reduced the equilibrium yield of the water gas shift reaction. Another reason was the effect of heat transfer resistance during the decomposition of cellulose and glucose. When higher biomass loading was used, it caused the increase in heat transfer resistance and hence, the non-isothermal condition in the mixture. This phenomenon severely affected the initial hydrolysis of the biomass solid and affecting its subsequent gasification reaction. Another reason for CO and H2 decreasing trend was attributed to the methanation reaction. It was established from previous work that methanation reaction between CO and H₂ to produce CH₄ are actually competing with the water gas shift reaction (Kruse and Gawlik, 2003). The consumption of CO during the reaction explained its decreasing trend. As stoichiometrically, methanation required 1 mole of CO and 3 mole of H_2 to produce 1 mole of CH_4 and 1 mole of H_2O . The high mole ratio of H₂ required in the reaction explained the significant decrease in H2 trend. The formation of CH₄ from methanation from a stoichiometric point of view did not need any water contribution. Therefore, CH₄ formation was preferred at higher biomass concentration (Kruse and Gawlik, 2003). This explained the increase in CH₄ formation with the biomass loading.

Figure 3 shows the gasification efficiency as a function of biomass loading. The gasification efficiency was found to decrease although the biomass content was increased. The highest efficiency was achieved at 5.0 g biomass loading with 21.76% and the lowest at 6.08% for the highest loading. The decrease in gasification efficiency despite the increase in biomass loading was because of several reasons as explained previously i.e., the roles of each reaction occurring during the course which selectively altered the product compositions due to the shifting of the equilibrium in addition to other isolated reaction due to the lignin content in the reactant. This in turn will affect the overall efficiency of the gasification. Figure 4 exhibits the effect of solid loading on the H₂

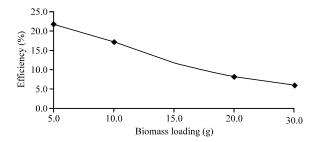


Fig. 3: Effect of biomass loading on the gasification efficiency for reaction condition: 340.0°C, 300g water, 30 min and particle size 250<X<500 μm

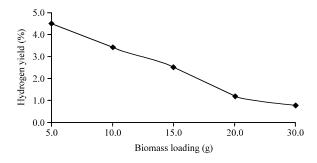


Fig. 4: Effect of biomass loading on the hydrogen yield for reaction condition: 340.0°C, 300g water, 30 min and particle size 250<X<500 μm</p>

yield. It was found that the highest H₂ yield was 4.52 % (5.0 g) before decreasing to the lowest at 0.79% (30.0 g). As mentioned previously, the trend shown was due to several reasons including the decrement of the water gas shift equilibrium yield, the effect of heat transfer resistance towards the reaction due to the higher concentration of solid and hydrogenation of lignin, which consumed H₂. Temperature also played a vital role in influencing the gasification efficiency of biomass in HCW. Water should ideally be at a temperature where it can enhance its chemical and physical properties to become an efficient processing medium. The temperature chosen must be high enough so that the desired reactions can take place at a significant rate (Jr et al., 1993).

On the other hand, conducting reactions in extreme temperature without a legitimate necessity caused complications in heat management and inflations in processing cost. In this study, effects of temperature (300.0-380.0°C) were studied with other parameters kept constant at the value of 5.0 g solid/300.0 g water, 30.0 min and particle size 250<X<500 μm . In Figure 5, it was observed that the order of the gases yield was different for the highest and lowest temperature. The order at 300°C was CO₂ > CO > CH₄ > H₂ and at 380.0°C it was CO₂ > CO > H₄. The lowest CO yield obtained from experiment

was 0.99 at 300.0°C. The highest yield was obtained at 380.0°C with 2.69 mol gas kg⁻¹ biomass. The similar observation with effect of solid loading strongly indicated that the experimental reaction was far from equilibrium. This observation was consistent with literature in which they concluded that the high experimental yield of CO was caused by the water gas shift reaction that had not gone to completion (Yan et al., 2006). However, the increasing trend of gas yield for CO2, H2 and the decreasing trend for CH₄ was in accordance with the equilibrium theory. CO₂ exhibited a significant increase with temperature with the lowest yield of 1.55 mol gas kg⁻¹ biomass (300.0°C) and highest of 5.21 mol gas kg⁻¹ biomass (380.0°C). The increase in H2 yield was consistent with the decrease in CH₄, which indicated the importance of methanation in influencing the gas compositions. Each of the trends could be explained by examining the reactions that occurred during the period.

The formation of gaseous products from biomass in HCW was from the decomposition of cellulose to glucose and subsequently steam reforming of the intermediates compounds to form CO₂, H₂ and CO. The increasing trend for all 3 types suggested that the rate of both reactions were accelerated with the increase of temperature. The theory behind the acceleration of the solid decomposition rate was supported by other literature (Shoji *et al.*, 2005). It was concluded from the literature that the decomposition rate of the wood biomass increased with the reaction temperature.

Theoretically, the steam reforming of the intermediate compounds produced CO, CO2 and H2 As observed from Fig. 5, unlike CO₂ which exhibited a significant increase in yield with temperature, for H₂ and CO, the increase were not significant hence suggesting another competing reaction i.e., methanation, which consumed both. These observations indicated that both water gas shift and methanation played vital role in determining the product gas compositions with manipulation of the reaction temperature. The gasification efficiency of the reaction as function of temperature is shown below in Fig. 6. The highest gasification efficiency achieved was 32.15% at 380.0°C a far increase from 21.76% obtained at reaction temperature of 340.0°C earlier for the solid loading effect. The highest yield obtained at the maximum temperature of reaction employed in this work further established the importance of decomposition rate for the intermediates compound. Figure 7 exhibited the hydrogen yield as function of temperature. As observed, the hydrogen yield increased from 0.87% (300.0°C) to 7.22% (380.0°C). Therefore, it was concluded that higher temperature caused a higher yield of H2 since it accelerated the decomposition of biomass constituents and subsequently

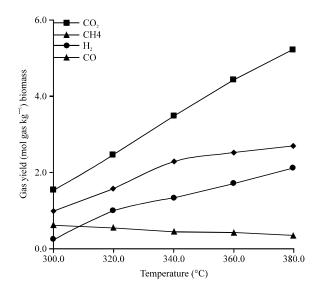


Fig. 5: Effect of temperature on the product gas compositions for reaction condition: 5.0 g solid/300g water, 30 min, and particle size 250<X<500 μm</p>

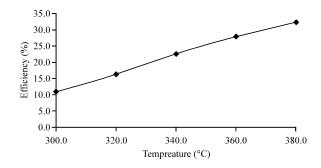


Fig. 6: Effect of temperature on the gasification efficiency for reaction condition: 5.0g solid/300g water, 30 min and particle size 250<X<500 μm</p>

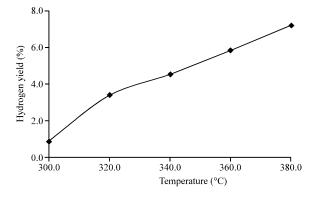


Fig. 7: Effect of temperature on hydrogen yield for reaction condition: 5.0 g solid/300g water, 30 min, and particle size 250<X<500 μm

the steam reforming reactions which were the precursor for gas formations.

The effect of K2CO3 on the gasification was studied in the range of 1.0 to 5.0 wt% with other reaction conditions fixed at 380.0°C, 5.0 wt% solid, 30.0 min, 300 g water and particle size 250<X<500 μm. Figure 8 exhibits the yield of product gas as a function of catalyst loading. The order of the gas compositions at zero catalyst loading was $CO_2 > CO > H_2 > CH_4$ while the order at the highest catalyst loading was CO₂ > H₂ > CH₄ > CO. CO₂ and H₂ initially showed a significant increase in its yield with the addition of K2CO3 until it reached 3.0 wt% of catalyst loading. However, the yield only increased slightly afterwards despite higher catalyst loading was added into the reaction. Highest value of CO yield is at 6.82 mol gas kg⁻¹ biomass (3.0 wt% K₂CO₃) while for H₂, its highest yield was 5.75 mol gas kg⁻¹ biomass (3.0 wt% K₂CO₃). It was also observed that the yield remained constant despite the increment of K2CO3 loading. For CH₄, there was also an increase in its yield although it was not as significant as to CO₂ and H₂. The highest yield of CH₄ was 2.38 mol gas kg⁻¹ biomass (5.0 wt% K₂CO₃) compared to 0.41 mol gas kg⁻¹ biomass (0 wt% K₂CO₃). On the other hand, CO exhibited opposite trend when compared to the other product gases. CO yield decreased from 1.49 mol gas kg⁻¹ biomass (0 wt% K₂CO₃) to only 0.53 mol gas kg⁻¹ biomass (5.0 wt% K2CO3). The trend of the product gas compositions strongly indicated that the addition of K₂CO₃ catalyzed gasification with formation of more H₂. CO₂ and lesser CO. One of the reasons for the increase was attributed to the function of K₂CO₃ in lowering the decomposition temperature of cellulose from 180°C to about 120-150°C (Minowa and Inoue, 1999). With the decrease in the decomposition temperature, more aqueous intermediate compounds were produced which enhanced the gasification pathways. Another reason was attributed to the catalyst influence towards the equilibrium of the reactions schemes that occurred during the gasification. K₂CO₃ accelerated the formation of CO₂ and H₂ by shifting the equilibrium of the water gas shift reaction through formation of intermediates (Sinag et al., 2003). When K₂CO₃ were added into the mixtures (solid biomass and H₂O), it reacted with water to produce intermediates of potassium bicarbonate (KHCO₃) and potassium hydroxide (KOH). KOH subsequently reacted with CO, which were formed from the initial steam reforming reaction of intermediate compounds from cellulose and glucose decomposition.

This reaction produced another intermediate, potassium formate (HCOOK). The consumption of CO during this reaction explained the decreasing trend in its

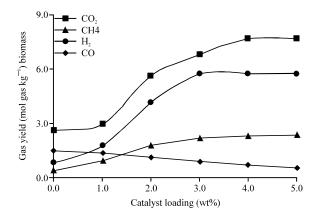


Fig. 8: Effect of K_2CO_3 loading on the product gas compositions for reaction condition: 380.0°C, 5.0 wt% solid, 30 min reaction time, 300g water and particle size 250<X<500 μm

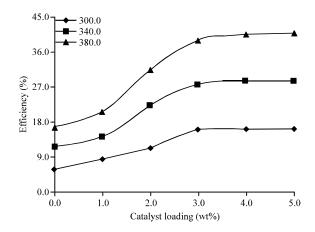


Fig. 9: Effect of K₂CO₃ loading for different reaction temperature on the gasification efficiency for condition: 5.0 wt% solid, 30 min reaction time, 300g water and particle size 250<X<500 μm</p>

yield with the increase of catalyst loading. HCOOK subsequently reacted with the excess water in the mixture to obtain H_2 and $KHCO_3$. This explained the significant increase in H_2 yield obtained from this particular reaction in addition from the original steam reforming reaction. The catalytic reaction came to a full circle with the dissolution of $KHCO_3$ to produce additional CO_2 and H_2O . The additional CO_2 produced in addition to those obtained earlier from steam reforming reaction contributed to the significant increase as observed from Fig. 8. The increase in CH_4 with the addition of catalyst loading was due to the methanation reaction. However, the increase was not significant due to the low partial pressure of CO in the mixture, although the other reactant, H_2 was in excess. Figure 9 shows the gasification efficiency at different

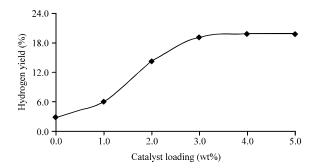


Fig. 10: Effect of K_2CO_3 loading on hydrogen yield for reaction condition: 380.0°C, 5.0 wt% solid, 30 min reaction time, 300g water and particle size 250<X<500 µm

temperature (300.0, 340.0 and 380.0°C) as a function of K₂CO₃ loading. As observed, the gasification efficiency increased significantly with addition of K₂CO₃. Highest gasification efficiency achieved at 380.0°C was 40.68% (5.0 wt% K₂CO₃), a significant increase from 16.61% (0 wt% K2CO3). It was also observed that the yield showed only a slight increase once it surpassed 3.0 wt% of K₂CO₃. The increase was attributed to 2 factors i.e., lower decomposition temperature and the shift in equilibrium for various reactions both of which influenced significantly on the gasification efficiency. From observation of Fig. 9, the optimal amount of K2CO3 was 3.0 wt%. Figure 10 exhibited the effect on hydrogen yield with the addition of different K2CO3 loading. The significant increase in the yield proved that K₂CO₃ enhanced the formation of H2. H2 yield increased significantly from 2.81% (0 wt% K₂CO₃) to 19.03% (3.0 wt% K₂CO₃) and subsequently showed only a small increase to 19.80% (4.0 wt% of K₂CO₃) and 19.72% (5.0 wt% of K₂CO₃). The significant increase in its yield was attributed to the enhancement of steam reforming reactions due to lower decomposition temperature of the solid biomass.

Apart from that, H_2 was also produced from the reaction between H_2O and HCOOK (intermediate formed from K_2CO_3). These observations proved the advantages of K_2CO_3 not only in enhancing the gasification but also in influencing the H_2 selectivity.

CONCLUSION

In conclusion, it was established that increasing the solid loading influenced all the 3 responses. Furthermore, changing the solid loading would affect the selectivity of the product gas differently. Therefore, this could be used to change the selectivity of the different products

according to desired applications. It was also observed that temperature played a vital role in influencing the outcome of this reaction. From the experimental work, it was concluded that higher temperature was favorable for gaseous product reactions although its compositions were significantly influence not only by temperature but also by others as determined in this study. In retrospect, there is a need to find a right balance between temperature and other parameters that could give the highest possible conversion to gaseous products but still able to maintain an acceptable heat efficiency to ensure the feasibility of this process. For the effect of homogenous catalyst, it was proven that K₂CO₃ can influence the outcome of the reactions. Catalytic gasification has several advantages due to its superiority in enhancing the reaction hence better possibility of obtaining high efficiency without the necessity to use very high temperature as previously thought. This is encouraging since the ultimate the aim of this process is to obtain maximum gasification efficiency using minimum energy as favorable from the economic and engineering viewpoint. When sufficient catalysts are added, the unique chemistry of this reaction medium can be further enhanced with respect to both the rate of conversion and the selectivity of products.

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