

Research paper

Pilot Study of Hydrogen-Rich Syngas Production from Ma Bamboo (*Dendrocalamus latiflorus*) by Plasma Reforming

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【 Summary 】

Major components of bamboo are solid hydrocarbons in the forms of cellulose, hemicellulose, lignins, and pentosan. The abundance of hydrocarbons indicates the potential of bamboo to be an excellent raw material to produce hydrogen. In this study, 1-yr-old ma bamboo (*Dendrocalamus latiflorus*) was liquefied with polyethylene glycol and then treated with plasma steam reforming to produce hydrogen. Productivity levels of hydrogen at different bamboo concentrations and pyrolysis temperatures were experimentally and theoretically studied. The liquefaction and pyrolysis apparatuses were hence optimized. A maximum productivity occurred at a weight ratio of 1: 10 of ma bamboo to water and a pyrolysis temperature of 800°C by plasma treatment.

Key words: ma bamboo, liquefaction, hydrogen production, thermal plasma steam reforming.

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研究報告

麻竹電漿重組產氫之初步研究

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摘要

竹材主要由纖維素、半纖維素、木質素、聚戊醣組成的固體型態碳氫化合物，使用藥劑進行液化後便能做為熱電漿重組產氫之原料，進而降低產氫造成的碳外循環排放。本研究係以聚乙二醇將一年生麻竹液化後，做為熱電漿重組產氫的原料，同時探討實驗值與理論值中，不同重量比及不同溫度下產氫率，進而建立最佳化之液化與產氫系統。實驗結果得知，在能源效率及產氫效率觀點下，實驗值中，在800°C下，液化麻竹/水重量比為1:10時，產生之合成氣為最佳，且證明以液化麻竹為原料進行產氫是非常可行的，此結果將為林業界及再生性氫能源開啟更多應用方向。在即將來臨的氫能經濟時代，將為我國在能源多元化政策上注入一針強心劑。

關鍵詞：麻竹、液化、產氫、熱電漿重組。

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INTRODUCTION

Humankind began to heavily rely on fossil fuels in the industrial revolution in the 1850s. Since then, civilization has dramatically progressed because of bulk fossil fuel consumption. The use of fossil fuels emits greenhouse gases into the atmosphere, which are believed to be the cause of global warming and climate change. To reduce emissions of greenhouse gases, a series of meetings have been held by the United Nations Framework Convention on Climate Change (UNFCCC) committee since 1992. Afterwards, many countries began to seek alternative, renewable energy resources such as hydrogen, biomass, solar energy, wind power, hydroelectric power, and geothermal energy (Wu and Huang 2006). Among them, hydrogen has the greatest diversity of applications as it can be used for the generation of electricity and engine fuel with zero pollution. Therefore, many countries have spent considerable ef-

forts in developing hydrogen production as an alternative energy source (Chen et al. 2011).

The thermal plasma steam reforming technique for hydrogen production has several advantages such as a simple system configuration, no need for catalysts, a high conversion efficiency, and low production costs (Bromberg et al. 2000). It pyrolytically converts fossil fuels into hydrogen with carbon black as the by-product (Cho et al. 2004). Any hydrocarbon material can be a feedstock to a thermal plasma reformer, but liquid fossil fuels are the most practical (Krashennikov et al. 1997; Petitpas et al. 2007). However, the use of fossil fuels violates the objective of reducing greenhouse gas emissions. Hence the use of biomass for hydrogen production using plasma reforming has become a trend. Tsai et al. (2007) demonstrated that vegetable oils could be a good feedstock. The conversion rate was 75% at a pyrolytic temperature

of 750°C. Chen et al. (2008) converted 60% of glycerol into hydrogen at a pyrolytic temperature of 800°C. Those 2 studies indicated the feasibility of using non-fossil, organic materials for hydrogen production by thermal plasma reforming.

Biomass is a renewable fuel from the land. In Taiwan, 58.53% of the land is covered by forests, of which 4.24% is covered by bamboo (Taiwan Forestry Bureau, 2006). Bamboo has a fast growth rate and a short cultivation span. It could serve as a good fuel source because of its high reproducibility and as a feedstock for hydrogen production because of its abundance of hydrocarbons. The hydrocarbons are in the forms of cellulose, hemicellulose, lignins, and pentosan (Li et al. 1995). The fibrous forms of the hydrocarbons impair its utilization in hydrogen production using thermal plasma reforming, since the reformer accepts only liquid feedstocks.

In this study, ma bamboo *Dendrocalamus latiflorus* was used as a feedstock for hydrogen production by thermal plasma steam reforming. Solid ma bamboo was liquefied and then mixed with water for plasma reforming. Bamboo solutions of different concentrations were processed at different reforming temperatures. Optimal process parameters were determined in terms of the maximum hydrogen yield, maximum biogas yield, minimum carbon dioxide production, and minimum energy consumption.

MATERIALS AND METHODS

Pretreatment of bamboo materials

One-year-old ma bamboo *D. latiflorus* was harvested from compartment 16 of the Suili Tract located in the National Taiwan Univ. Experimental Forest, Nantou, Taiwan. The bamboo was crushed and ground to a powder of 40~60 mesh. The powder was

dried in an oven at 105°C for 12 h and kept in a desiccator at room temperature before being used.

Composition analysis of ma bamboo

The physical and chemical properties of ma bamboo were analyzed using the following codes/methods: moisture content followed ASTM D1576-90; ethanol-toluene extractives followed ASTM D1107-96; holocellulose was analyzed by the chloride method (Wise and Karl 1962); acid-insoluble lignin followed ASTM D1106-96; acid-soluble lignin was analyzed by the UV absorptivity method (Sluiter et al. 2008); and ash followed ASTM D1102-84.

Liquefaction of ma bamboo

Liquefaction was carried out in a flask equipped with a stirrer and reflux condenser. Polyethylene glycol (PEG, molecular weight 200) and glycerin at a weight ratio of 8: 1 was used as the liquefying reagent. The liquefying reagent was mixed and reacted with the dried bamboo powder at a weight ratio of 1.2: 1. Sulfuric acid (3 wt% of 97%) in the liquefying reagent was placed in a flask. The flask was immersed in an oil bath and preheated to 150°C which initiated the reaction. After 60 min, the flask was immersed in cold water to quench the reaction product. Then, the liquefied product of ma bamboo was obtained.

The following analyses were further performed on the ma bamboo liquid.

1. Elemental Analysis

Ma bamboo liquid was dried in an oven at 105°C for 24 h. The solid residue that weighed 5 ± 0.2 mg was enclosed in a tin container and placed inside an autosampler drum for elemental analysis of C, H, N and S using an Elemental Vario CHNS/O analyzer (Germany).

2. Thermogravimetric Analysis

Thermogravimetric analyses were carried out with a PerkinElmer Pyris1 thermogravimetric analyzer (USA) in an N₂ atmosphere at 20 ml/min and at a temperature that ranged 50–850°C. The heating rate was 20°C/min, and the weight of the initial sample was 5–10 mg. After initiating heating, weight losses of the sample and carbon residue were measured.

The thermal plasma H₂ reformer

The thermal plasma reforming system (TPRS) used in this study can perform non-combustion pyrolysis, non-equilibrium thermal plasma chemistry, and gas-phase electrolysis of superheated vapor (Wang 2006, Wang et al. 2007, Tsai et al. 2008). The TPRS reactor uses electricity to preheat the feedstock and generate plasma. It accepts only fluid feedstocks, as an interesting limitation to plasma reforming.

Figure 1 shows a flowchart and framework of the thermal plasma H₂ reformer used in this study. The liquid feedstock was preheated using waste heat from the heat exchanger, and this was then fed to the sprayer/evaporator unit. The sprayer atomizes the

solution to fine droplets, and the droplets are vaporized in the hot atmosphere of the evaporator. The temperature inside the evaporator is a compromise between energy saving and carbon residue. The vapor enters the plasma reformer for a pyrolytic reaction. Finally, resultant syngases are collected for subsequent analysis or utilization.

After preheating, the reactor produces plasma to heat up the feedstock and maintains the high internal temperature with heat energy from non-combustion pyrolysis of the feedstock. The reactor is designed with thermal radiation enhancement, energy trapping techniques, and high-density insulation to minimize heat losses during operation. Feedstock circulating in the reactor follows electromagnetic hydrodynamics (EMHD). These designed hydrodynamics ensure non-equilibrium chemical reactions for minimum unexpected chemicals and hence a maximum conversion (reforming) rate. Unlike other plasma reformers (Levent et al. 2003), the reactor does not explicitly need a chemical catalyst but is designed with an inherent catalytic function by releasing intensive, activating ions and electrons (Ahmed et al. 1999, Palm et al. 2002, Lutz et al. 2003).

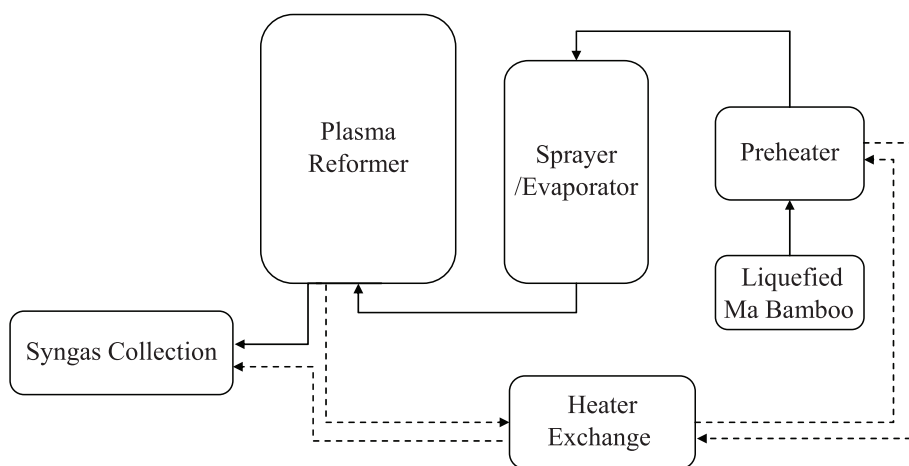


Fig. 1. Thermal plasma hydrogen production system.

Liquefied ma bamboo and water as feedstock

Ma bamboo liquid was diluted with water to three weight ratios of 1: 6, 1: 8, and 1: 10. Diluting with water reduced the viscosity of the liquefied ma bamboo. At the same time, it provided water requirements for plasma reforming. The plasma reformer was set to 3 operating temperatures of 750, 800, and 850°C. Solutions of the same concentration were reformed at the 3 different pyrolysis temperatures. This arrangement gave 9 types of experiment.

Resultant syngases from the reformer were subjected to a gas composition analysis (5890, Hewlett-Packard, USA). The Hewlett-Packard gas chromatograph was equipped with a thermal conductivity detector (TCD) and a Supelco Carboxen 1010 capillary column (0.53 mm × 30 m). The operation conditions were an injector temperature of 180°C; a detector temperature of 240°C; He carrier gas at 3 ml/min; an initial oven temperature of 35°C; an initial holding time of 6 min; a heating rate of 15°C/min; a final temperature of 240°C; and a final holding time of 4 min. The analyzer analyzed hydrogen (H₂), carbon monoxide (CO), carbon dioxide (CO₂), and trace hydrocarbons such as CH₄, C₂H₂, and C₂H₄.

Thermodynamic and chemical equilibrium analyses

Thermodynamic properties and chemical equilibrium were estimated using chemical reaction and equilibrium software (HSC Chemistry for Windows, ChemSW, Outotec, Finland). The feedstock solutions and reactant syngases were assumed to have been completely oxidized, i.e., CO₂ and H₂O were the only reactants. The estimation gave the enthalpy (H), entropy (S), heat capacity (C), and product compositions of possible reactants from ma bamboo liquids by pyrolysis.

RESULTS AND DISCUSSION

Composition of ma bamboo

Table 1 summarizes the chemical composition of ma bamboo. The ethanol-toluene extractives are a lumped measure of waxes, fats, resins, oils, tannins, and certain other ether-insoluble components of the bamboo, which were about 11.61%. It is worth noting that ma bamboo consists of 85.32% cellulose and lignins. These 2 components can be easily liquefied with polyethylene glycol. The ash content of the bamboo was made up of inorganic minerals, of about 3.07%. Yamada et al. (2001) previously reported that the lignin in lignocellulosic materials condenses with itself under acidic conditions. In this study, it comprised about 13.67% of the residue. By excluding the 3.07% of ash, 10% residue from lignin condensation may occur during liquefaction.

Determining the evaporating temperature

The optimum operating temperature of the evaporator was determined by a thermogravimetric analysis for maximum efficiency and minimum char residue.

Figure 2 shows residues of the ma bamboo solutions in response to temperature variations. Increasing the water content decreased the content of char residue. An evaporating temperature of 130°C produced a quite good residual reduction rate for all solutions. There

Table 1. Composition of ma bamboo

Composition	Weight percent (wt %)
Ethanol-toluene extractives	11.61 ± 0.71
Holocellulose	62.76 ± 0.05
Acid-insoluble lignins	20.47 ± 0.33
Acid-soluble lignins	2.09 ± 0.08
Ash	3.07 ± 0.12

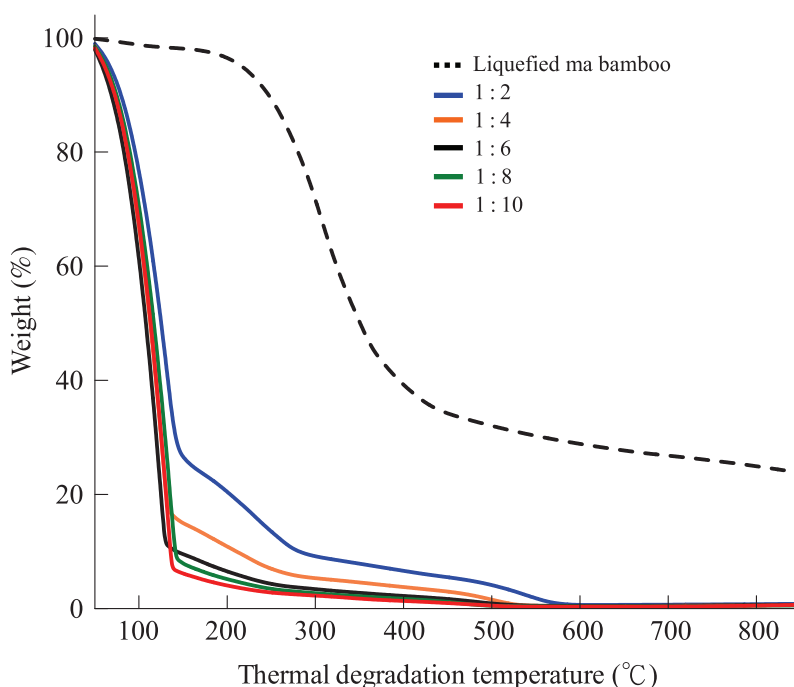


Fig. 2. Thermogravimetry curves at different weight ratios of liquefied ma bamboo to water.

was no significant difference in the char residue when the water content was increased from 1:10 to 1:6. At an evaporating temperature of 400°C, char contents from solutions with bamboo/water ratios of 1: 6, 1: 8 and 1: 10 were $\leq 2.5\%$. Further raising the evaporating temperature reduced the char residue sluggishly. Hence, as a compromise between energy consumption and char residue, the evaporating temperature was set to 400°C for all subsequent operations. Thus, during the experiment, the evaporator was set at a maximum temperature of 400°C, and samples were liquefied at ma bamboo product/water ratios of 1: 6, 1: 8, and 1: 10.

Elemental analysis

Table 2 indicates that elements C and O accounted for 88.25% of the liquefied ma bamboo products. These are the 2 major compositions of liquefied ma bamboo. The chemical formula of the liquefied ma bamboo was

Table 2. Elemental analysis of liquefied ma bamboo products

Elemental composition	Weight percent (wt %)
N	2.41
C	47.19
S	0.99
H	8.34
O	41.07

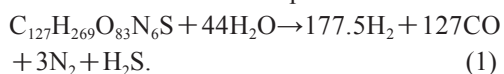
determined to be $C_{127}H_{269}O_{83}N_6S$. Compositions of syngases from the plasma reformer could hence be estimated using thermal and chemical equilibrium calculations with the above-derived chemical formula.

Thermodynamics and chemical equilibrium of the liquefied ma bamboo

With this element composition or chemical formula (see section 3), the hydrogen-rich syngas compositions produced from the plasma reformer could be predicted via ther-

mal and chemical equilibrium calculations. There are 4 possible chemical reactions in a complete reforming process.

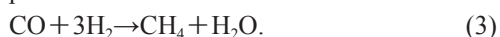
Steam reforming occurs in the plasma reformer as a result of pyrolysis. The ma bamboo solution is decomposed into:



The carbon monoxide reacts with the hot water steam. From the reactions listed above, the water content will increase the production of H_2 and CO_2 . This reaction is called a water shift and has the form:



The hydrogen may react with carbon monoxide to produce methane and water products:



In CO_2 reforming, different from the water shift, the deposited carbon is formed by CO disproportionation:



The above 4 reactions take energy which comes from the plasma reformer. Hence plasma reforming for hydrogen production requires a bulk energy supply.

Theoretical syngas compositions from ma bamboo using thermal plasma steam reforming technology

Figure 3 shows the calculated molar fractions of each syngas composition produced from a 1: 6 ma bamboo solution using thermal plasma steam reforming at different pyrolysis temperatures. The results indicated that the productivity of hydrogen was 66.98~67.59% in molar fraction. Carbon dioxide and methane decreased in response to an increase in temperature. Carbon monoxide increased as the temperature increased.

To explore the optimal conditions of hydrogen production, ma bamboo solutions of different concentrations were processed with plasma reforming at 800°C and 1 atm.

Figure 4 shows the results. The results were identical to the prediction shown in Fig. 3. Only a trivial amount of methane was produced. Carbon dioxide increased and carbon monoxide decreased with an increasing water content, both as a result of a sufficient water supply in the water shift procedure. CO was reduced, CO_2 increased, and H_2 increased as shown in Eq. (2). The highest hydrogen

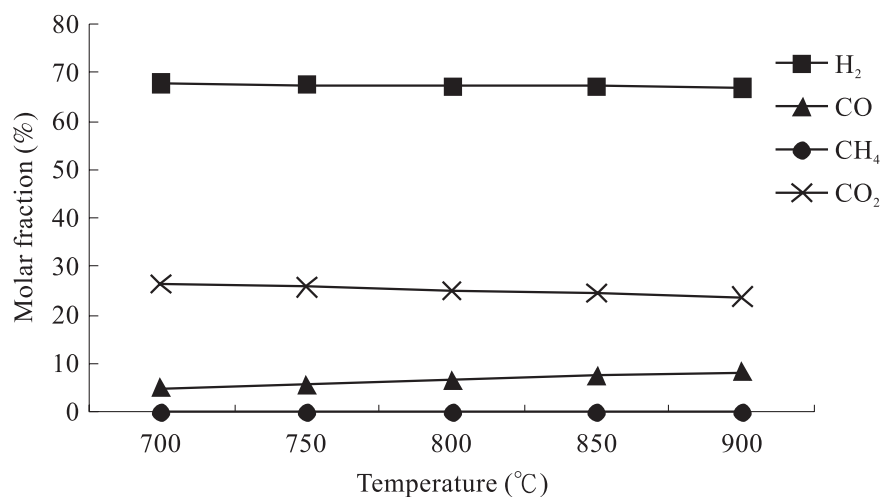


Fig. 3. Theoretical H_2 -rich syngas compositions (molar fractions) at a liquefied ma bamboo products/water weight ratio of 1: 6 and different temperatures.

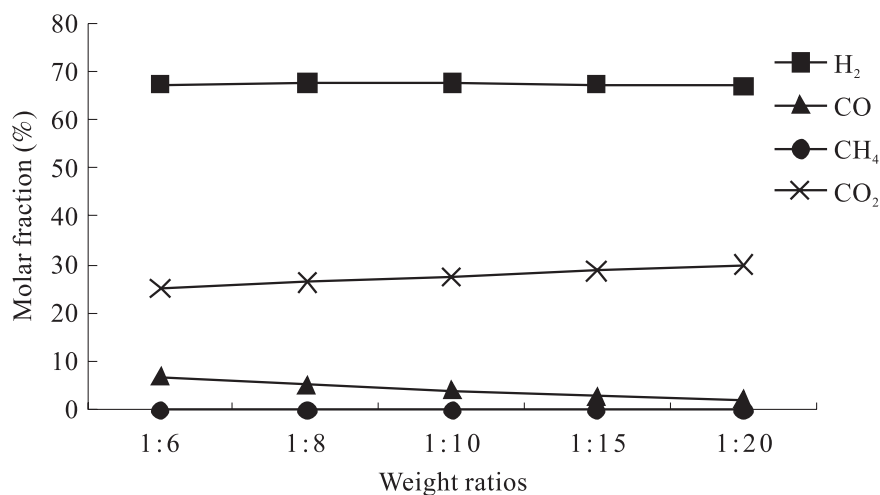


Fig. 4. Theoretical H₂-rich syngas compositions (molar fractions) at different liquefied ma bamboo product/water weight ratios and a temperature of 800°C.

content was at 1: 10 at about 67.59%. The result also shows that an optimal weight ratio of ma bamboo liquefaction to water was 1: 10.

Experimental hydrogen production from ma bamboo using thermal plasma steam reforming technology

An experimental system was constructed, and experimental testing was performed at different operational conditions based on theoretical calculations. Water was added to the liquefied ma bamboo. After mixing, the mixture entered the experimental system for hydrogen-rich syngas production. The hydrogen-rich syngas was sampled and analyzed by gas chromatography (GC). In order to minimize the number of the parameters that affected the results, the pressure and ma bamboo product/water mixture flow rate were fixed in the present study. However, the operational temperature and liquefied ma bamboo weight ratio were varied during the experiment. Test results are shown in Figs. 5 and 6.

Figure 5 shows that at a liquefied ma bamboo to water weight ratio of 1: 6, the molar fraction of produced hydrogen content was

around 34.9% at 700°C; this hydrogen content increased to 49.5% at 750°C, but slightly dropped at 800°C. The experiment also found that CO increased at increasing temperatures, but CO₂ had a reverse trend. The trend was similar to that reported in the literature (Van Oost et al. 2008, Ouni et al. 2009).

Figure 6 also shows that when the experiment was performed at 800°C, as the water content increased, the molar fractions of the hydrogen and carbon dioxide slightly dropped; carbon monoxide increased; but methane increased at the ratios of 1: 6~1: 8 and then decreased at the ratios of 1: 8~1: 10. However, from the point of view of energy efficiency and hydrogen yield, a tradeoff should be made at a liquefied ma bamboo to water weight ratio of 1: 10 (also confirmed in Fig. 4).

Experimental results are shown in Fig. 4 but do not have a good agreement with those shown in Figs. 5 and 6. The theoretically calculated hydrogen molar fraction can reach 65%; however, the experimental values were in a range of 33~50%. The possible reasons include the following.

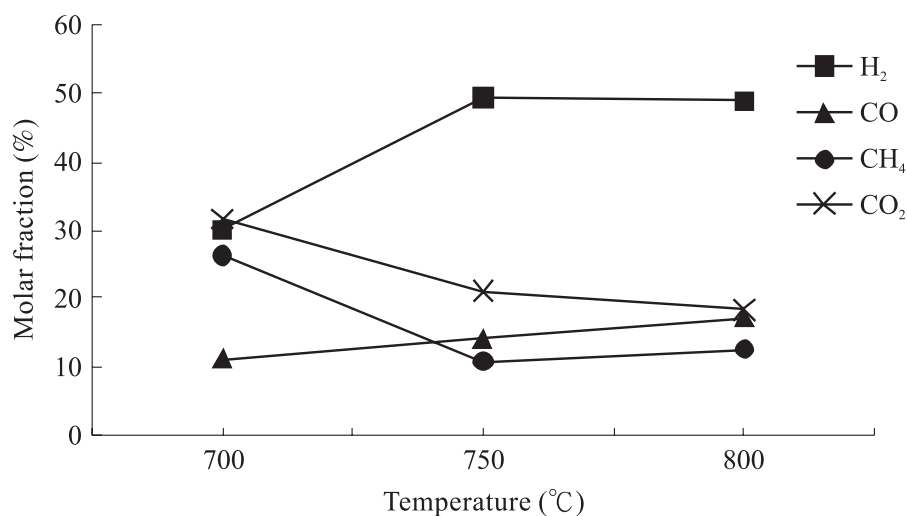


Fig. 5. Experimental results of H₂-rich syngas compositions (molar fractions) at a liquefied ma bamboo product/water weight ratio of 1: 6 and different temperatures.

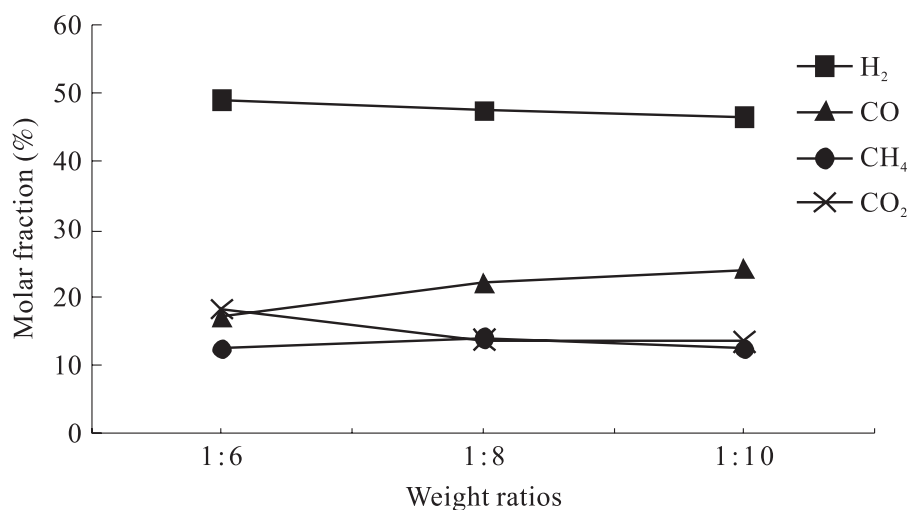


Fig. 6. Experimental H₂-rich syngas compositions (molar fractions) at different liquefied ma bamboo product/water weight ratios and a temperature of 800 °C.

1. The theoretical calculations were based on ideal thermal and chemical equilibriums in a steady state. A steady state only arises at very high temperatures and/or very long-time durations. However, at the test conditions we used, an equilibrium state was not reached.
2. The boiling point of ma bamboo can be high and can vary because of its physical

structure and composition; this was validated by Fig. 2. This wide range of boiling points limits the evaporation efficiency of the evaporator. This was confirmed by the residue or carbon formation inside the evaporator and reformer after the experiment. That consequently affected the results of H₂-rich syngas production.

CONCLUSIONS

The theoretical and experimental studies showed that it is feasible to produce hydrogen from ma bamboo using thermal plasma reforming technology. The amount of carbon residue in the atomizer/evaporator unit should be minimized. As a compromise between energy efficiency and hydrogen yield, the optimal operating conditions for hydrogen-rich syngas production were a liquefied ma bamboo to water weight ratio of 1: 10 and a reforming temperature of 800°C. There was a big discrepancy between the theoretical hydrogen molar fraction (65%) and the experimental result (33~50%). This is the first study on hydrogen production from liquefied ma bamboo and by thermal plasma reforming. Significant efforts are still required to optimize system operation. Future work should mainly focus on equipment modifications for complete evaporation of liquefied ma bamboo and changes in the ma bamboo liquefaction process to minimize its molecular size and lower its boiling point.

Although this study is considered preliminary, it opens up new possibilities for renewable energy sources by the forestry industry, especially for bamboo farming in Taiwan. In the upcoming hydrogen economy, this can provide wider information on energy diversity for national policymakers.

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