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Hydrogen production from catalytic gasification of cellulose in supercritical water

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Abstract

Interests in large-scale use of biomass for energy and in hydrogen are motivated largely by global environmental issues. Cellulose and sawdust were gasified in supercritical water to produce hydrogen-rich gas in this paper, and Ru/C, Pd/C, CeO_2 paticles, nano-CeO_2 and nano-$(CeZr)_xO_2$ were selected as catalysts. The experimental results showed that the catalytic activities were Ru/C > Pd/C > nano-$(CeZr)_xO_2$ > nano-CeO_2 > CeO_2 particle in turn. Low-concentration sodium carboxymethylcellulose (CMC) (2–3 wt.%) was mixed with particulate biomass and water to form a uniform and stable viscous paste which can be efficiently gasified. The 10 wt.% cellulose or sawdust with CMC can be gasified near completely with Ru/C catalyst to produce 2–4 g hydrogen yield and 11–15 g potential hydrogen yield per 100 g feedstock at the condition of 500 °C, 27 MPa, 20 min residence time in supercritical water.

Keywords: Cellulose; Supercritical water; Gasification; Hydrogen production

1. Introduction

Interests in large-scale use of biomass for energy and in hydrogen are motivated largely by global environmental issues. If grown and used renewably, biomass would make little or no net contribution to atmospheric greenhouse gas concentrations. Of the options for making hydrogen from renewable sources, biomass appears to be the lowest cost one for the near- and mid-term. Wet biomass can be directly dealt with by supercritical water gasification (SCWG) so as to avoid the drying process with high energy-consumption in conventional thermo-chemical gasification. A number of researchers have investigated hydrogen production by SCWG of whole biomass.

The earliest report on supercritical gasification of wood is that of Modell [1,2]. In 1978, he issued a patent and reported the gasification of glucose and maple sawdust with a catalyst composition that promotes hydrogenation, reforming and cracking in water in the vicinity of its critical state. No solid residue or char was produced. In 1982, Elliott and co-workers [3–5] converted wet biomass to methane-rich gas using a reduced metal catalyst selected from the group consisting of ruthenium, rhodium, osmium, iridium or mixtures thereof. Reaction conditions from about 300–450 °C and, at least, 13 MPa pressure were covered. In 1996, Minowa et al. [6] found hydrogen-rich gas could be obtained in hot-compressed water (350 °C, 17 MPa) from biomass with reduced nickel catalyst and sodium carbonate. In 1993, Antal et al. [7,8] presented the first studies of glucose complete gasification at 600 °C, 34.5 MPa and a 30 s residence time. Following this work newly discovered carbon-based catalysts were used in high-concentration biomass supercritical water gasification for high efficiencies, and particulate biomass could be mixed with a cornstarch gel to form a viscous paste that can be delivered with a cement pump. In 2000, Schmieder et al. [9] found that wet biomass and organic wastes were completely gasified by addition of KOH or K_2CO_3 at 600 °C and 25 MPa, forming a H_2 rich gas containing CO_2 as the main carbon compound.

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Concentrations of CO, CH_4 and C_2–C_4 hydrocarbons were low in the product gas (<1, ∼3 and <1 vol.%, respectively). In 2001, Lin et al. [10,11] proposed a new H_2 production process named the HyPr-RING process, which used calcium oxide (CaO) or/and calcium hydroxide $Ca(OH)_2$ as the adsorbent of carbon dioxide to improve the hydrogen fraction in product gas. In 2002, Watanabe et al. [12] reported that zirconia (ZrO_2) was also effective for H_2 production from biomass in supercritical water. In 2002, CMC [13,14] was reported to mixed with particulate biomass and water to form a uniform and stable viscous paste, which could be pumped to the continuous flow tubular reactor.

A successful catalytic process depends on the optimized combination of catalyst (components, manufacturing process, and morphology), reactants, reaction environment, process parameters, and reactor configuration [15]. Catalysts must be more durable as compared to catalysts used in typical gaseous phase operations, due to water adsorption, sintering, and dissolution of catalyst components. Each of these interrelationships must be understood in order to develop a successful catalytic supercritical water gasification process.

Elliott et al. [16,17] demonstrated that Ru, Rh and Ni had significant activity for the conversion of *p*-cresol, Pt, Pd and Cu was reported lacking of activity, and α-alumina, ZrO_2 and carbon was identified as the stable supports. Usui et al. [18] presented Pd supported on Al_2O_3 particularly with highest catalytic activity for cellulose gasification among a supported Ni, Pd or Pt. Osada et al. [19] reported the catalytic conversion of biomass with a Ru catalyst supported on TiO_2 in supercritical water would be an effective method for biomass gasification at low temperatures as 400 °C. CeO_2 particles are used as high-temperature oxidation catalyst in Elementar High TOC II analyzer. In this paper, Ru/C, Pd/C, CeO_2 paticles, nano-CeO_2 and nano-$(CeZr)_xO_2$ were selected as catalysts of biomass SCWG in the batch reactor. Cellulose, a major component of woody biomass, was used as the starting material to study the effect of catalyst and reaction time. Then the catalytic gasification result of cellulose was compared with sawdust.

2. Apparatus and experimental procedure

The gasification of cellulose and sawdust was carried out in a 140 ml, high-pressure autoclave. Fig. 1 shows the autoclave schematic and Fig. 2 shows the profile of temperature measure point. The axial distributing H1–H7 and circumambient distributing HB1-4 are fixed on the reactor outer wall and H4 and HB1 is the same one measure point. And axial distributing HI1–HI4 is fit in the furnace. This is an axial symmetry autoclave and furnace, so the temperature of H4 and HB3 is very close. The autoclave is fabricated from 316L stainless steal and the lines of purging and sampling are constructed of 1Cr18Ni9Ti stainless steal. The system was operated at temperatures up to 650 °C and pressures up to 35 MPa. The

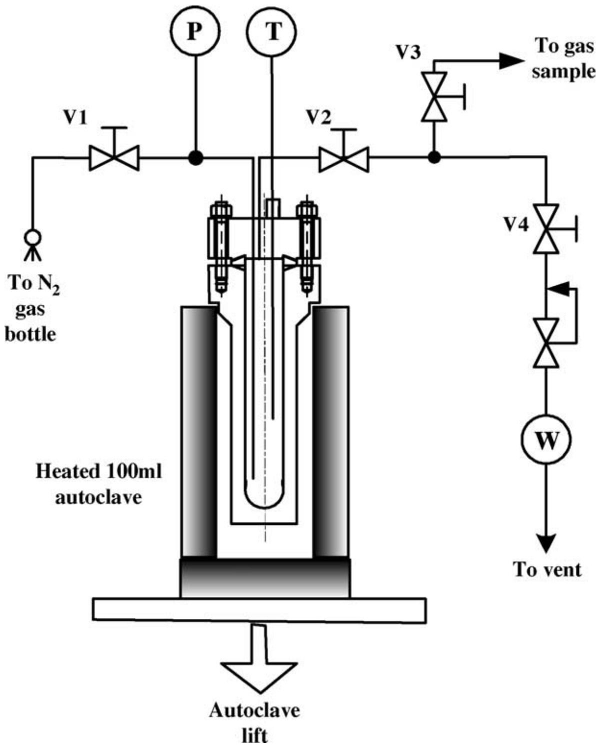

Fig. 1. The autoclave schematic.

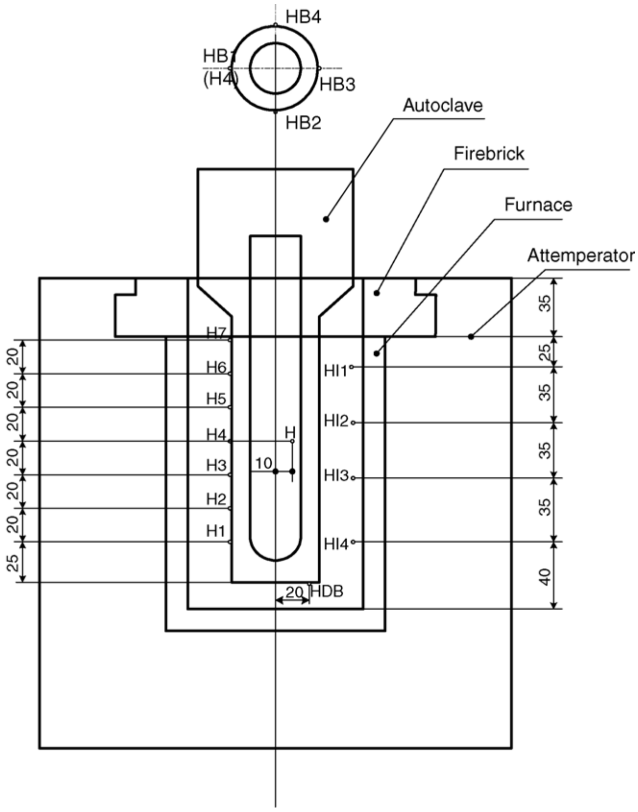

Fig. 2. The profile of measure point.

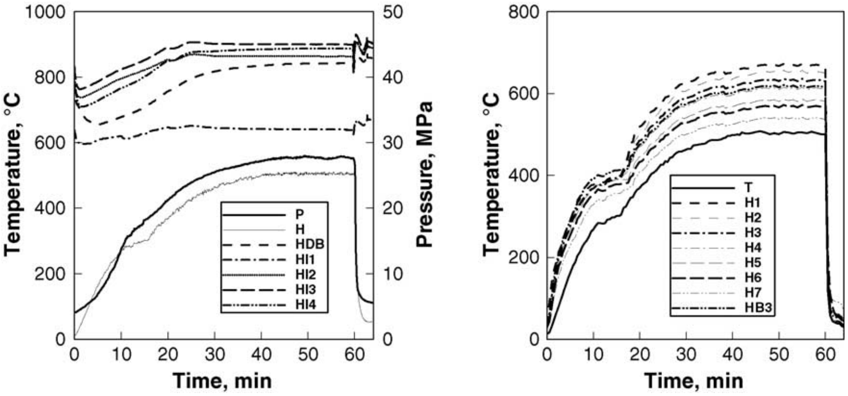

Fig. 3. The temperature profile of autoclave heated when the Ini.p is 4 MPa.

pressure were monitored by means of pressure transducer system in the line of N_2 purging. The temperature inside the reactor was monitored by type K thermocouple. The autoclave was heated by 1.2 kW temperature controlled electric furnace and cooled by water. After cooling down to room temperature, the product gas is sampled and measured using a wet test meter. Then the autoclave is demounted, and the reaction mixture is recovered for separation. The aqueous phase is separated by washing the reaction mixture with water and by filtration. The fraction insoluble on the filter is dried at 70 °C to obtain the residue. The carbon element compositions of the solution and residue are analyzed using an Elementar High TOC II analyzer. Analysis of the gaseous products is accomplished by use of a Hewlett-Packard model 6890 gas chromatograph (GC) equipped with thermal conductivity detectors.

Fig. 3 shows the temperature profile of autoclave heated when the initial pressure (Ini.p) is 4 MPa. The temperature can be heated to 500 °C in 40 min. The pressure is increased as the temperature is increased, when the temperature remains steady, the pressure in the autoclave remains steady too. The temperature of reactor wall is on the range of 10% with the average wall temperature. The temperature inside the reactor can be cooled to below 200 °C in 1 min and below 100 °C in 2.5 min.

Cellulose microcrystalline $[(C_6H_{10}O_5)_n]$ is water insoluble particle with white color. CMC $[(C_8H_{11}NaO_7)_n]$ is straw yellow powder with 6.5–8.5 wt.% Na. Sawdust $[(CH_{1.35}O_{0.62})_n]$ was collected in sawmill, and crushed to 200 meshes. The 5.0 wt.% Pd on activated carbon, 5.0 wt.% Ru on activated carbon, nano-CeO_2 with purity of 99.9 wt.% (dry basis), nano-$(CeZr)_xO_2$ with purity of 99.9 wt.% (dry basis) and CeO_2 particle produced by Elementar Company were used as catalysts.

3. Properties of catalysts

The isothermal absorption and desorption characteristics, area surface and pole volume were measured by Beckman Coulter Co. SA3100TM surface area analyzer.

3.1. *The isothermal absorption and desorption characteristics*

Fig. 4 shows the isothermal absorption and desorption characteristics of catalysts. Liquid N_2 was isothermally absorbed and desorbed by the catalysts mentioned above. The isothermal absorption curves of Ru/C and Pd/C are shown as Langmuir-type mono-molecule absorption. The isothermal absorption curves of nano-CeO_2 and nano-$(CeZr)_xO_2$ are shown as BET-type multi-layer absorption. The isothermal absorption curves of CeO_2 particle is shown as Langmuir-type mono-molecule absorption at the less relative pressure and BET-type multi-layer absorption at the improved relative pressure. The capillary agglomeration of nano-CeO_2 and nano-$(CeZr)_xO_2$ appears when the relative pressure is near 1.0.

3.2. *The profile of pore diameter*

Fig. 5 shows the profile of pore diameter curves of the catalysts. The pore diameters of nano-CeO_2 and nano-$(CeZr)_xO_2$ have wide range from several to several hundred nanometer. The state of saturation absorption is not appeared. The Ru/C and Pd/C catalysts have micro-pore structure, its pore diameter is distributed below 5 nm. When the micropore is filled up, the amount of absorption is not increased with the increasing of relative pressure. The state of saturation absorption appears. The pore diameter of the CeO_2 particle is distributed below 50 nm.

3.3. *The surface area of catalyst*

The surface area of catalyst is calculated by BET formula, and the value is shown in Table 1. The surface area has wide range from 0.276 m^2/g (CeO_2 particle) to 1196.86 m^2/g (Ru/C).

4. Results and discussion

The mass fraction of hydrogen in cellulose is 6.17%, and hydrogen theoretical yield of cellulose gasification is 14.8%

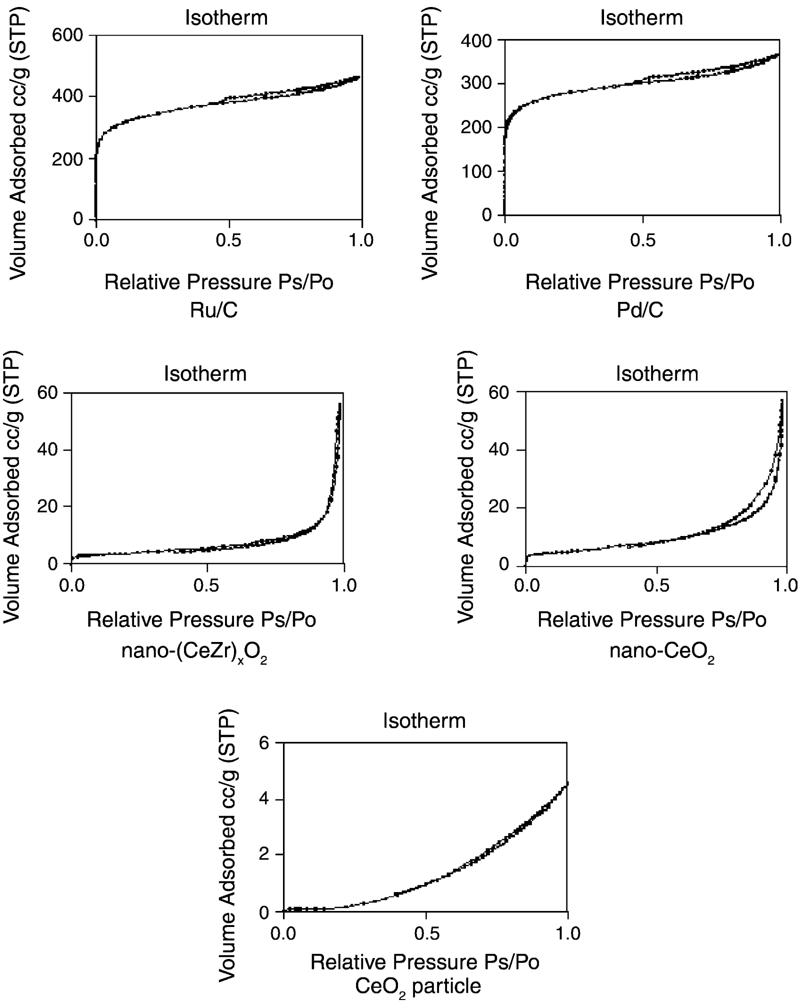

Fig. 4. The isothermal absorption and desorption characteristics of catalysts.

when cellulose is gasified completely to hydrogen and carbon dioxide. The mass fraction of hydrogen in sawdust is 5.27%, and hydrogen theoretical yield of sawdust is 16.08%.

4.1. *The effect of CMC addition on cellulose gasification*

In this experiment, 10 g water, 1.0 g cellulose and/or 0.4 g Ru/C catalyst and 0.2 g CMC powder were added and mixed in the autoclave. The autoclave with feedstock was installed and sealed. N_2 was used as purge gas. The initial pressure was regulated to 4.0 MPa by adding N_2 continuously and adjusting the values at lines. The temperature of feedstock was heated to 500 °C, and cooled by water when the temperature remains 20 min at 500 °C. The product gas was sampled and analyzed when the reactor autoclave was cooled to air temperature. At last, the reactor autoclave was opened, and the liquid and solid product were measured and analyzed.

Table 1
The surface area of catalysts

Catalyst	Surface area (m^2/g)
Ru/C	1196.86
Pb/C	962.42
$n(CeZr)_xO_2$	13.418
$nCeO_2$	19.079
CeO_2 power	0.888

Fig. 6 shows that the effect of CMC addition on cellulose gasification with and without Ru/C catalyst. The CMC addition enhanced the gasification efficiency (GE (%) = the mass of gas product/the mass of biomass), carbon gasification efficiency (CGE (%) = the carbon fraction in gas product/the carbon fraction in biomass), hydrogen yield (YH_2 (%) = the mass of hydrogen yield/the mass of biomass) and potential hydrogen yield (PYH_2 (%) = YH_2 + CO yield + 4CH_4 yield, defined as the sum of measured hydrogen and the hydrogen which could theoretically be formed by completely shifting carbon monoxide and completely reforming hydrocarbon species). The mixture of cellulose and CMC could be near completely gasified with Ru/C catalyst. The influence of CMC on gasification was more serious with Ru/C catalyst. The yields of hydrogen, methane and carbon dioxide were improved obviously and the less fraction of carbon oxide was

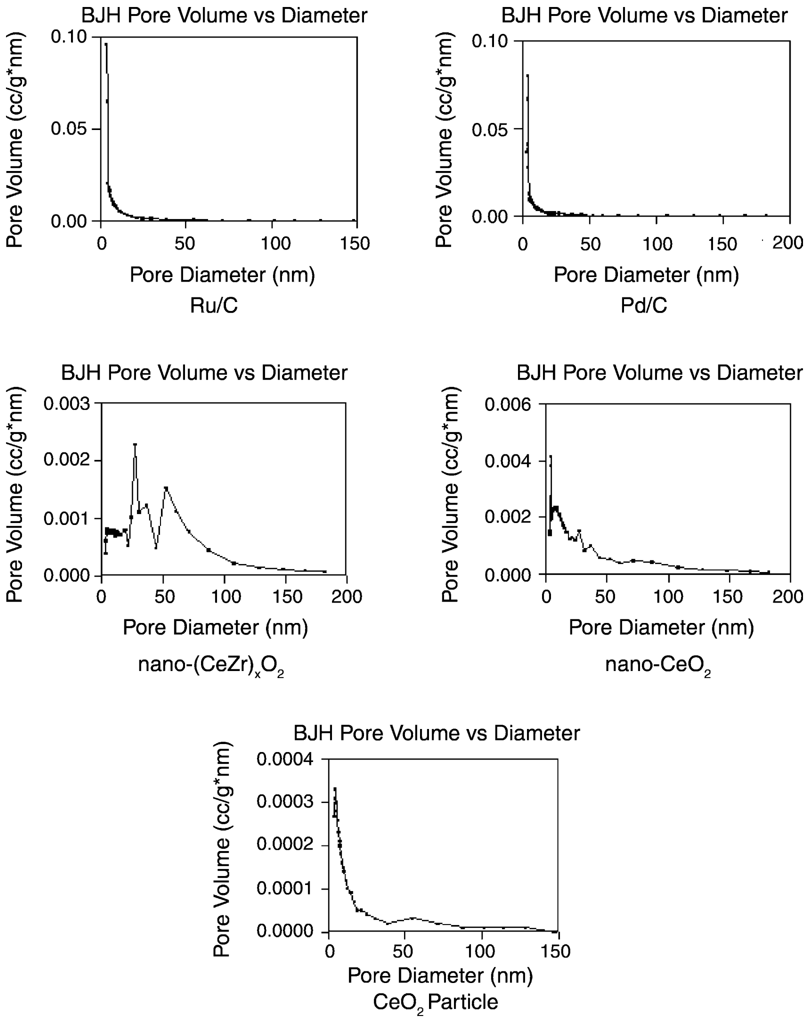

Fig. 5. The curves of catalysts pore volume vs. diameter.

produced with the addition of CMC. CMC addition would strengthen the mixing of cellulose and water to improve the reaction rate, and Na^+ from CMC would catalyze the water–gas shift reaction to improve the hydrogen fraction in product gas.

4.2. Catalyst performance with cellulose and CMC

Fig. 7 shows the effect of different catalyst on cellulose gasification. The gasification efficiency, carbon gasification efficiency, hydrogen yield and methane yield with Ru/C

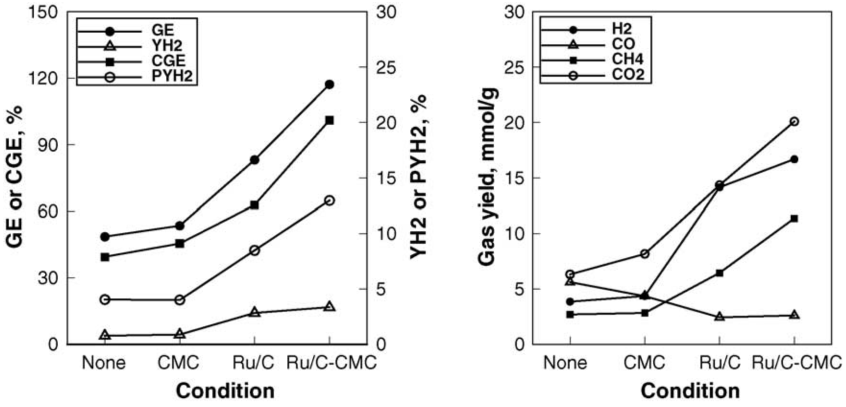

Fig. 6. The effect of CMC addition on cellulose gasification (water, 10 g; cellulose, 1.0 g; Ru/C, 0.4 g; CMC, 0.2 g; 500 °C; 20 min; Ini.p, 4.0 MPa).

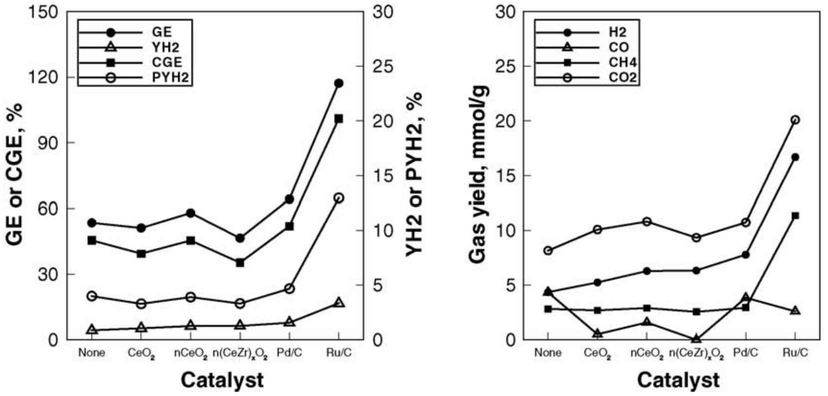

Fig. 7. The effect of different catalyst on cellulose gasification (water, 10 g; cellulose, 1.0 g; catalyst, 0.4 g; CMC, 0.2 g; 500 °C; 20 min; Ini.p, 4.0 MPa).

catalyst had the most value on these conditions, and Ru/C has the largest surface area among all the catalysts used in this experiment. Then, the surface area of catalyst is important for supercritical water catalyst gasification. Pd/C has the same amount of metal, the similar isothermal absorption and desorption curve, the similar profile of pore diameter and the similar large surface area, but the product gas with Pd/C catalyst was produced less than that with Ru/C catalyst at the same conditions. It can be concluded that Ru metal have more catalytic activity than Pd.

The methane yield was similar with CeO_2 particle, nano-CeO_2, nano-$(CeZr)_xO_2$, Pd/C catalyst and without catalyst. The more hydrogen, more carbon dioxide and less carbon oxide with catalyst were produced than that without any catalyst. Then, the reaction of water–gas shift was enhanced with these catalysts.

The catalysts of nano-CeO_2 and nano-$(CeZr)_xO_2$ have wider profile of pore diameter than CeO_2 particle as Fig. 4, and more hydrogen with nano-CeO_2 and nano-$(CeZr)_xO_2$ is produced than that with CeO_2 particle. It seems that the profile of pole diameter has identical trend with hydrogen production for this metal oxide type catalyst.

4.3. The gasification results with Ru/C catalyst

4.3.1. The effect of different initial pressure condition with different temperatures

In this experiment, 15 g water, 1.5 g cellulose, 0.3 g Ru/C catalysts and 0.3 g CMC powder were added and mixed in the autoclave. The initial pressure is regulated to 0.1, 0.5, 1.0, 1.5 or 2.0 MPa. The pressure of reaction was increased to 27 MPa, the temperature of reactions were 532, 509, 483, 482 or 457 °C, respectively. Fig. 8 shows that the gasification results on different initial pressure condition with different temperatures. The initial pressure had the more effect on gasification results because the gasification efficiency was still low even the reaction temperature was high as 532 °C when the initial pressure was 0.1 MPa. The temperatures were very similar when the initial pressures were 1.0 and 1.5 MPa.

4.3.2. The comparison of cellulose gasification by SCW partial oxidation and gasification

In this experiment, the autoclave with feedstock was installed and sealed. When N_2 is used as purge gas, the

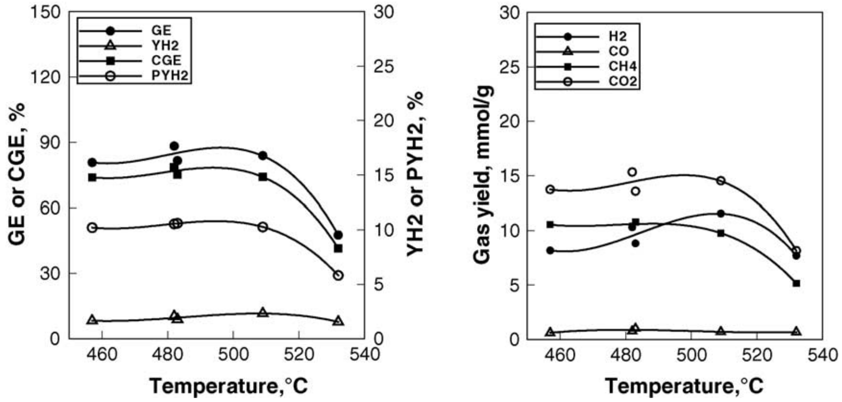

Fig. 8. The gasification results on different Ini.p conditions with different temperatures (27 MPa; residence time, 20 min; water, 15 g; cellulose, 1.5 g; Ru/C, 0.3 g; CMC, 0.3 g).

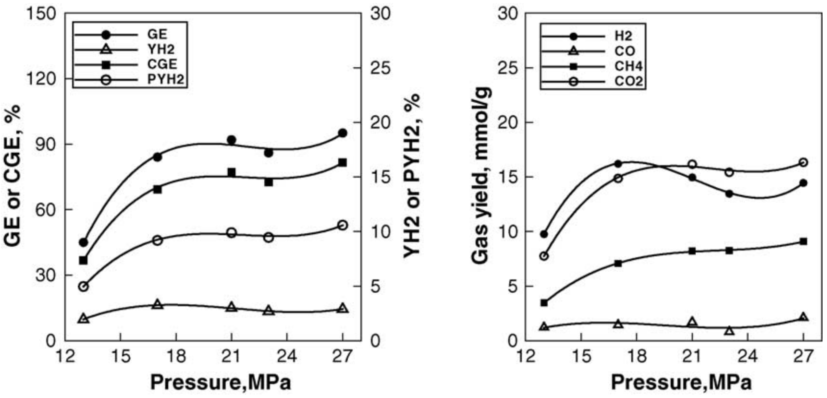

Fig. 9. The gasification results on different Ini.p condition with different pressures (water, 10 g; cellulose, 1.0 g; Ru/C, 0.2 g; CMC, 0.2 g; 500 °C; residence time, 20 min).

reaction condition is the completely gasification, and if N_2 is not used, the reaction condition is the supercritical water (SCW) partial oxidation because the oxide in the air will take part in the reaction. The initial pressure is 0.1 MPa. Table 2 shows the cellulose gasification result by SCW partial oxidation and gasification. The gasification has produced the more gas product with more hydrogen, carbon dioxide and methane and less carbon oxide.

4.3.3. *The gasification results on different initial pressure condition*

In this experiment, the initial pressure was regulated to 0.1, 1.0, 2.0, 3.0 or 4.0 MPa respectively, and the temperature of feedstock was heated to 500 °C, the pressured of reactions were 13, 17, 21, 23, 27 MPa, respectively.

The effects of initial pressure and the pressure of reaction on gasification results were shown the same trend. Fig. 9 shows that the hydrogen yield had the least value when the pressure of reaction was near the critical pressure. The yield of methane was increased and the yield of carbon oxide remained the same levels as the pressure of reaction (initial pressure) was increased.

Table 2
The comparison of cellulose SCWG with SCW partial oxidation

	Partial oxidation	Gasification
GE (%)	32.12	47.59
YH_2 (%)	0.89	1.54
CGE (%)	29.37	41.51
PYH_2 (%)	3.67	5.81
H_2 (mmol/g)	4.43	7.70
CO (mmol/g)	2.86	0.68
CH_4, mmol/g	2.77	5.16
CO_2 (mmol/g)	4.27	8.16

Operating conditions: water, 15 g; cellulose, 1.5 g; Ru/C, 0.3 g; CMC, 0.3 g; 500 °C; residence time, 20 min.

4.3.4. *The effect of amount of catalysts on cellulose gasification*

In this experiment, different amount of catalyst (0, 0.2, 0.4, 0.8 and 1.6 g) were added into the autoclave. Fig. 10 shows that the amount of catalyst has great influence on gasification. When the amount of catalyst was increased to 0.8 g, the gasification efficiency, the carbon gasification, potential hydrogen yield and the yield of methane and carbon dioxide were increased. And when the amount of catalyst was

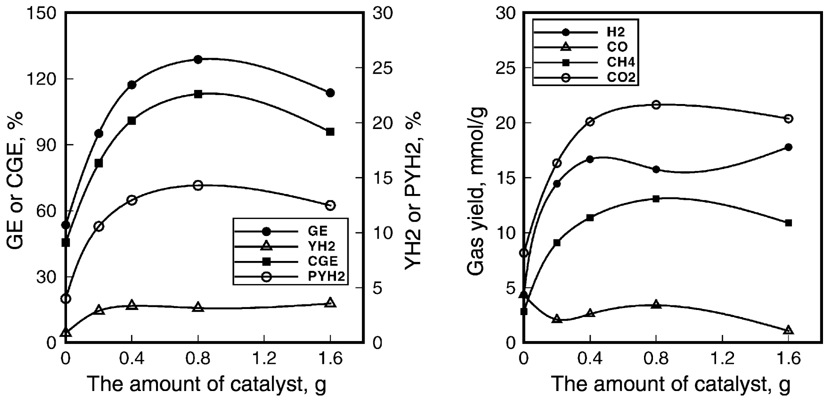

Fig. 10. The effect of amount of catalysts on cellulose gasification results (water, 10 g; cellulose, 1.0 g; CMC, 0.2 g; 500 °C; residence time, 20 min).

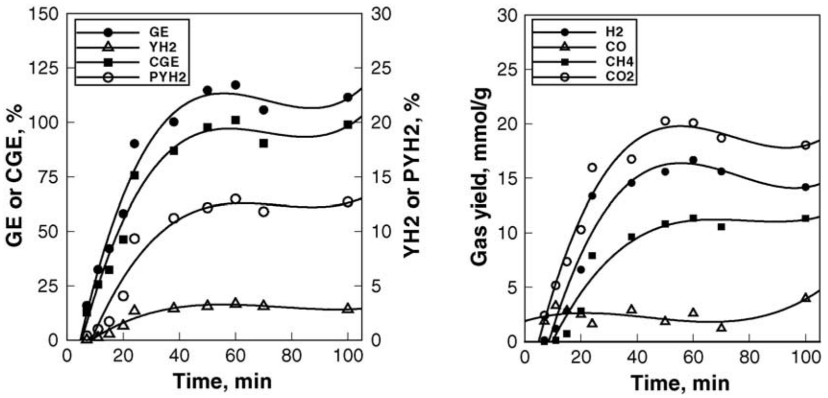

Fig. 11. The effect of reaction time on cellulose gasification results (water, 10 g; cellulose, 1.0 g; Ru/C, 0.4 g; CMC, 0.2 g; Ini.p, 4.0 MPa).

increased from 0.8 to 1.6 g, these values were decreased. And the hydrogen yield had the most value at the amount of catalyst is 0.4 g.

4.3.5. *The effect of reaction time on cellulose gasification*

In this experiment, the temperature of feedstock was heated to 250, 300, 350, 400, 450 or 500 °C, respectively, and cooled by water at once in some operating runs. At the others conditions, the temperature remains 10, 20, 30, or 60 min, respectively, when the feedstock temperature was heated to 500 °C. Fig. 11 shows the cellulose supercritical water gasification results at different reaction times. The gasification was reached the most gas product and hydrogen yields, when the temperature remains 20 min when the feedstock temperature was heated to 500 °C.

4.3.6. *Comparison of sawdust and cellulose gasification*

In this experiment, cellulose and sawdust were gasified in autoclave. Table 3 shows that sawdust can be gasification near completely, and the gasification results of cellulose and sawdust show the similar characteristics. The results of cellulose gasification were better than sawdust. Maybe other compound in sawdust is difficult to gasification than cellulose. The research of cellulose gasification had the important values on other raw biomass gasification.

Table 3
The gasification results of sawdust and cellulose

	Sawdust	Cellulose
GE (%)	98.08	117.23
YH_2 (%)	2.53	3.34
CGE (%)	77.2	93.7
PYH_2 (%)	11.73	12.92
H_2 (mmol/g)	12.65	16.68
CO (mmol/g)	1.29	2.60
CH_4 (mmol/g)	11.18	11.37
CO_2 (mmol/g)	16.87	20.09

Operating conditions: water, 10 g; sawdust, 1.0 g; Ru/C, 0.2 g; CMC, 0.4 g; 500 °C; residence time, 20 min.

4.4. *The comparison with previous gasification results*

Fig. 12 shows the comparison of this work with previous gasification results. Catalyst and temperature have great effect on the hydrogen yield and potential hydrogen yield. The gasification with Ni/Na_2CO_3 catalyst produced more hydrogen than that with Pt/C and Pd/Al_2O_3 at 350 °C. The gasification with Ni/Na_2CO_3 catalyst produced more hydrogen than that with only Ni or Na_2CO_3 addition, respectively.

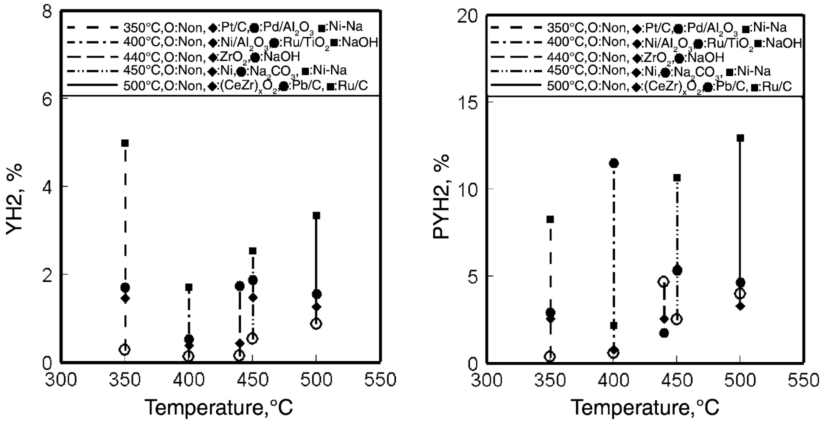

Fig. 12. The comparison of this work with previous gasification results. 350 °C [20]; 400 °C [19]; 440 °C [12]; 450 °C [3]; 500 °C, this work.

The NaOH catalyst had better hydrogen production catalytic activity than ZrO_2, Ni/Al_2CO_3 and Ru/TiO_2 at the same condition. The effect of catalyst on PYH_2 had the same trend as on YH_2 except that the PYH_2 value with Ru/TiO_2 catalyst is more than that with NaOH and the PYH_2 value without catalyst is more than that with catalyst. In this work, the hydrogen yield was the most except that with Ni/Na_2CO_3 at 350 °C. The potential hydrogen yield with Ru/C has the most value than that on other condition since in higher temperature. Maybe the temperature of this work is a little higher than previous gasification temperature.

5. Conclusion

In an autoclave, the mixture of cellulose and CMC was catalytically gasified in supercritical water with Ru/C, Pd/C, CeO_2 particle, nano-CeO_2 and nano-$(CeZr)_xO_2$ as catalyst. It can be concluded that cellulose with CMC addition can be gasified to produce more gas and hydrogen than that without CMC, and Ru/C has more catalytic activation than Pd/C, CeO_2 particle, $n$$CeO_2$ and $n(CeZr)_xO_2$ as catalyst at the same condition with CMC. The cellulose has the similar gasification results with raw biomass sawdust. The gasification operating conditions were optimized with Ru/C catalyst. The 10 wt.% cellulose or sawdust with CMC can be gasified near completely with Ru/C catalyst to produce 2–4 g hydrogen yield and 11–15 g potential hydrogen yield per 100 g feedstock at the condition of 500 °C, 27 MPa, 20 min residence time in supercritical water.

Acknowledgments

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