

Parametric study of pyrolysis and steam gasification of rice straw in presence of K_2CO_3

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Abstract—A parametric study of pyrolysis and steam gasification of rice straw (RS) was performed to investigate the effect of the presence of K_2CO_3 on the behavior of gas evolution, gas component distribution, pyrolysis/gasification reactivity, the quality and volume of synthetic gas. During pyrolysis, with the increase in K_2CO_3 content in RS (i) the instantaneous CO_2 concentration was increased while CO concentration was relatively stable; (ii) the yield of CO_2 and H_2 increased on the cost of CH_4 . During steam gasification of RS, with the increase in K_2CO_3 content in RS (i) the instantaneous concentration of CO_2 and H_2 increased while instantaneous concentration of CO and CH_4 decreased; (ii) the yield of CO_2 and H_2 production and total yield increased; and (iii) yield of CO and CH_4 production followed the order: 9% K_2CO_3 RS < 6% K_2CO_3 RS < raw RS < 3% K_2CO_3 RS < water-leached RS. Water-leached RS showed the highest pyrolysis reactivity, while steam gasification reactivity was proportional to K_2CO_3 content in RS. The results of this study reveal that the presence of K_2CO_3 during pyrolysis and steam gasification of RS effectively improves production of H_2 rich gas.

Keywords: Pyrolysis, Steam Gasification, Biomass, Rice Straw, K_2CO_3

INTRODUCTION

Rice is grown in over a hundred countries. Approximately 480 million metric tons (Mt) of milled rice was produced in 2014 [1]. Asian countries—China, India Indonesia, Bangladesh, Vietnam, Myanmar, Thailand, the Philippines, Japan, Pakistan, Cambodia, the Republic of Korea, Nepal, and Sri Lanka—account for 90% of the world's total rice production [1]. For every ton of grain harvested, about 1.35 tons of rice straw remain in the field [2]. Research is going on to explore efficient processes for energy recovery from agricultural waste [3,4]. According to Chakma et al. [5] the production of different biofuels from rice crop residues is economically viable. Bioenergy from rice crop residues provides simultaneous solution to issues of energy security and climate change risk in developing nations [6]. Gasification is considered as a key technology for energy recovery from biomass. It offers high flexibility in processing different kinds of feedstock materials as well as in the generation of different products [7]. Catalytic gasification is an efficient way to convert carbonaceous materials to optional gaseous products [8].

The initial step of gasification is pyrolysis, in which volatiles are released, leaving behind the solid char. Pyrolysis is followed by char gasification in which volatiles and char are further reacted and converted into the final products in the presence of reactive agents [9]. There is not a clear boundary line about these two stages

and the two processes interact with each other. The presence of inherent alkali and alkaline earth metallic species enhances the heterogeneous char gasification, as well as the homogeneous water-gas shift, steam-tars reforming, and hydrocarbon reforming reactions on biomass gasification [10]. The purpose of our study was to evaluate the effect of presence of K_2CO_3 during the pyrolysis and steam gasification of RS on gas evolution behavior, gas component distribution and quality and volume of gas in a laboratory fixed-bed reactor under atmospheric pressure. We also analyzed the effect of the presence of K_2CO_3 on reactivity during both pyrolysis and gasification. A fixed-bed gasifier was selected for this study due to its simplicity and production of relatively high H_2 content in the gaseous product [11].

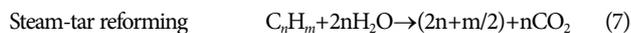
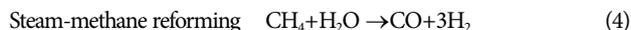
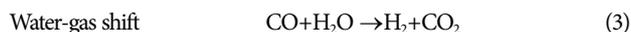
PYROLYSIS AND GASIFICATION REACTIONS

To clarify the reaction mechanisms of biomass gasification, researchers usually study gasification by two stages: pyrolysis and char gasification. During pyrolysis, volatiles (CO , CO_2 , CH_4 and H_2), tar and char are produced by thermal-chemical decomposition of biomass (cellulose, hemicellulose and lignin) [12]. During steam gasification, homogeneous and heterogeneous reactions between pyrolysis gas, tar, char and steam occur simultaneously. Steam as a gasifying agent is considered as one of the most effective techniques to produce synthetic gas with higher H_2 content [13]. The main reactions during pyrolysis and steam gasification are as under [14-16].

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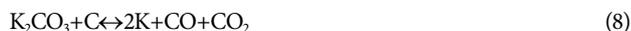
Potassium catalyst undergoes similar redox cycles under steam gasification conditions [23]. McKee [24] tried to explain the mechanism of redox cycle in localized sites where the catalyst and carbon interact as the following.



PYROLYSIS AND GASIFICATION REACTIONS CATALYZED BY K_2CO_3

A catalyst has the main influence on the yield distribution percentage because it has the ability to decompose feed material significantly [17]. Mudge et al. [18] found K_2CO_3 was an effective catalyst for conversion of wood to synthesis gases and methane-rich gas. Lang [19] testified that alkali and alkaline earth metals, especially in the form of carbonate, are the superior catalysts for the gasification reactions. Li et al. [20] reported improvements in gas yield during the atmospheric steam gasification of peat catalyzed by K_2CO_3 when studied at the temperature range from 973 °K to 1,123 °K. High temperature and long reaction time favor the increase in gas yield, but they reduce caloric value of the gas product.

Karimi and Gray found K_2CO_3 as better catalyst than Na_2CO_3 for the steam gasification of bitumen coke [21]. According to McKee [22] potassium undergoes redox cycle for CO_2 gasification, in which the potassium catalyst is reduced and oxidized as illustrated in reactions (8), (9) and (10).



The sum of reactions (8), (9) and (10) gives the reverse Boudouard reaction:

Wang et al. [25] proposed oxygen transfer and intermediate hybrid mechanism as shown in Fig. 4(a) to explain the reasons for the increase in instantaneous release concentration of CO_2 and H_2 increase with the increase in potassium content. However, according to Wu et al. [26] the above mechanism failed to explain the selectivity of catalytic gasification. Wu et al. [26] also suggested a modified mechanism of K_2CO_3 -catalyzed steam gasification of ash-free char which is shown in Fig. 4(b).

EXPERIMENTAL SECTION

1. Materials Pretreatment

RS sample was obtained from Shenyang city, Liaoning province, China. Air-dried biomass was ground and sieved to a size of 0.15-0.25 mm. The elemental and proximate analyses of RS selected as raw material are given in Table 1. Inherent potassium in RS sample was removed by leaching the sample with deionized water. During leaching the sample at a ratio of 80 ml/g (deionized water to biomass) was placed into a constant temperature water bath oscillator maintained at 30 °C temperature for 3 h; after that the solution was filtered and residue was dried at 105 °C in the oven for 12 h. The potassium content of raw RS and water leached RS was determined by ICP-E8300. The removal efficiency of potassium content during leaching was up to 90.5%. In biomass gasification the catalysts can be added directly to the biomass by dry

Table 1. The elemental and proximate analyses of RS

Elemental analysis (wt%)														Proximate analysis (wt%)			
H	C	N	S	O	Cl	K	Na	Ca	Mg	Al	Si	Fe	Ti	Ash	VO	FC	M
5.53	39.77	0.82	0.24	53.6	0.44	0.81	0.32	0.45	0.018	0.35	4.99	0.349	0.005	15.84	71.55	6.62	5.99

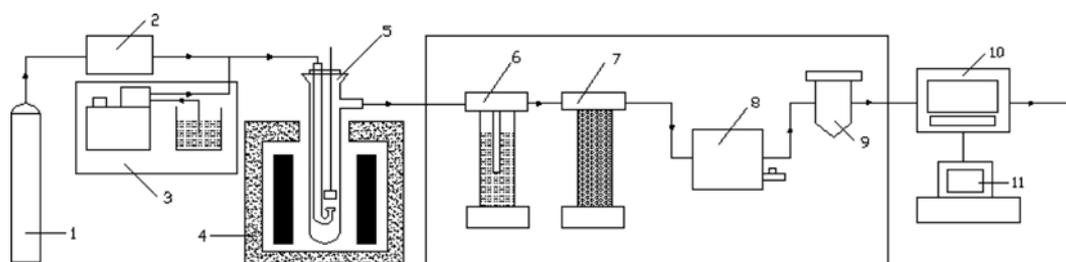


Fig. 1. The experimental setup for pyrolysis and steam gasification of RS.

(1) Nitrogen cylinder

(2) Mass flow meter

(3) Steam generating device

(4) Gasifier

(5) Quartz tube reactor

(6) Dust removing device

(7) Decoking unit

(8) Gas pump

(9) Condensing unit

(10) On-line infrared gas analyzer

(11) Computer

mixing or wet impregnation [27]. We used K_2CO_3 as potassium additive and adopted impregnation method to add K_2CO_3 to water leached RS to prepare the samples containing mass concentration of 3%, 6%, 9% K_2CO_3 .

2. Experimental Setup and Procedure

2-1. Pyrolysis Experiment

The experimental apparatus is shown in Fig. 1. One-gram sample in quartz crucible with micropores was placed in the most upper part of the quartz tube reactor. N_2 was purged (in a 200 ml/min flow rate) into the quartz tube reactor and reactor temperature was raised to 750 °C. After 15 min of achieving 750 °C the

sample was moved to reaction zone to start pyrolysis experiment. On-line infrared gas analyzer (GASBOARD-3100 type produced by Wuhan Sifang) was used to record instantaneous gas volume concentration data. The pyrolysis reaction lasted for 20 min.

2-2. Gasification Experiment

Compared with the pyrolysis experiment, the steam gasification experiment has an extra device (steam generating). Experimental procedure is basically the same as for the pyrolysis experiment. At 750 °C, steam was introduced into the reactor (at flow rate of 2 ml/min) under 200 ml/min N_2 entrainment. In addition, the quartz crucible was replaced by 200 mesh copper network during gasifi-

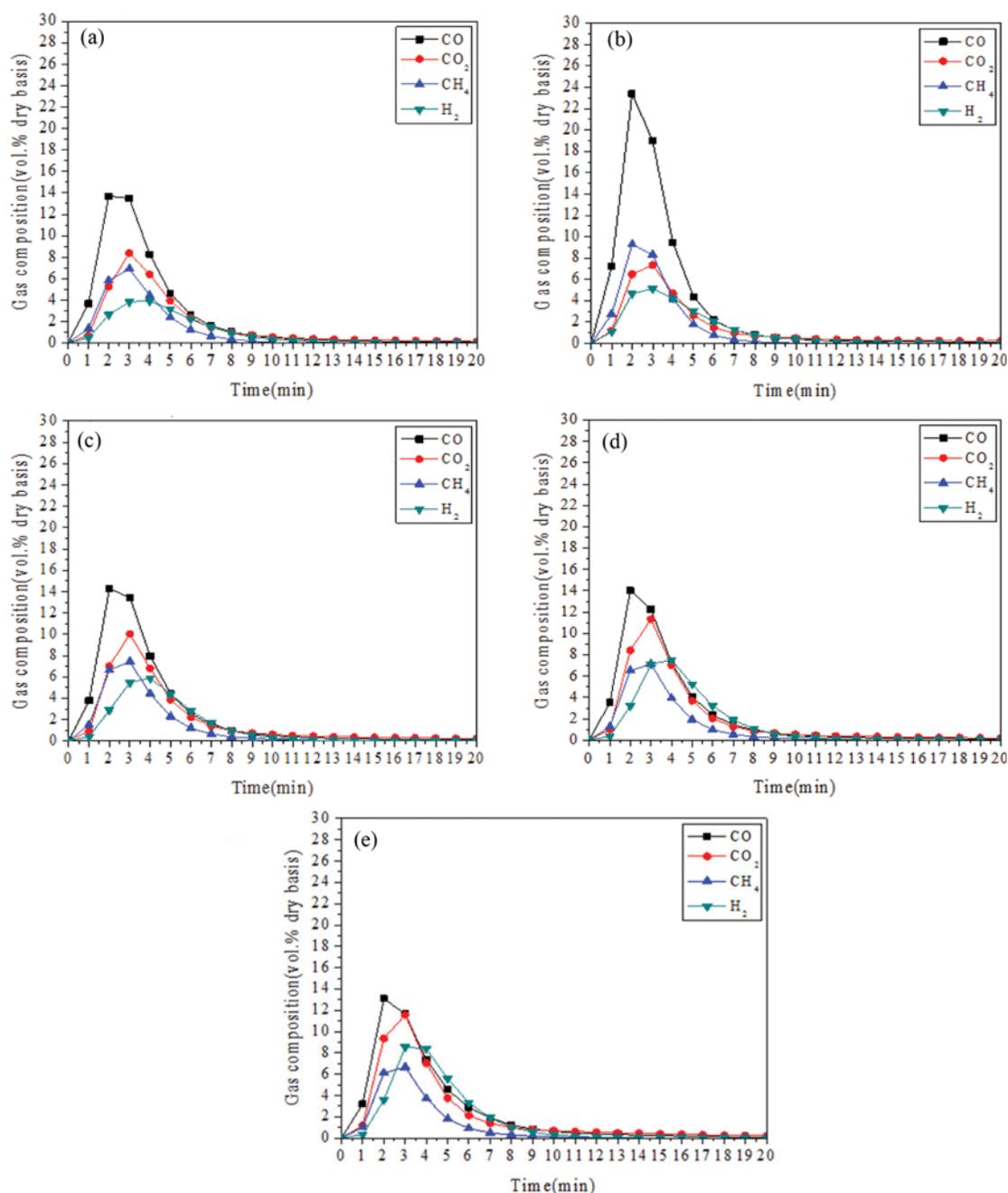


Fig. 2. The effect of presence of K_2CO_3 on gas transient release behavior during pyrolysis of (a) Raw RS, (b) water leached RS, (c) 3% K_2CO_3 -RS, (d) 6% K_2CO_3 -RS (e) 9% K_2CO_3 -RS.

cation in order to make RS contact with steam more intimate.

3. Analysis Methods

During RS pyrolysis and steam gasification process the instantaneous release concentration (volume percent of each component in gas) was recorded by GASBOARD-3100 on-line infrared gas analyzer. The carbon conversion rate (X) of RS during pyrolysis and steam gasification was calculated by the following equation [28].

$$X = \frac{\int_{t_0=0}^{t=t} \left(\frac{200 \times 10^{-3} \times (\varphi_{CO} + \varphi_{CO_2} + \varphi_{CH_4})}{22.4 \varphi_{N_2}} \times 12 \right) dt}{m_o} \times 100 \quad (15)$$

where m_o is the carbon mass of RS, t_0 is the initial time of RS pyrolysis/steam gasification, and φ_{CO} , φ_{CO_2} , φ_{CH_4} and φ_{H_2} are CO, CO₂, CH₄, H₂ instantaneous volume fraction at time t.

RESULTS AND DISCUSSION

1. The Effect of Potassium on Transient Gas Components During Pyrolysis and Steam Gasification of RS

Since pyrolysis and steam gasification are not completely independent and interact with each other, it is hard to divide the two stages clearly. However, they have different reaction characteris-

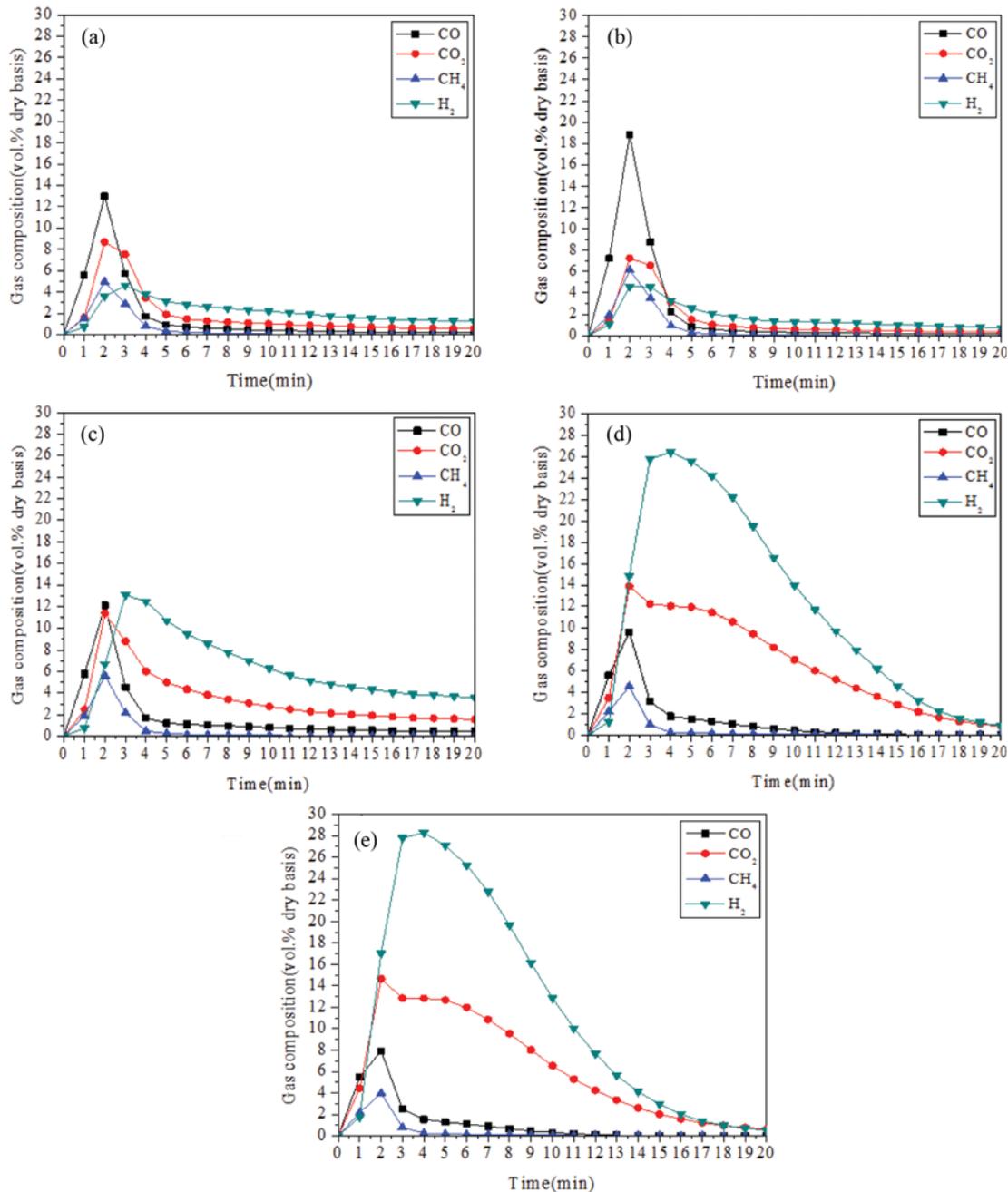


Fig. 3. The effect of presence of K₂CO₃ on gas transient release behavior during steam gasification of (a) Raw RS, (b) water leached RS, (c) 3% K₂CO₃-RS, (d) 6% K₂CO₃-RS, (e) 9% K₂CO₃-RS.

tics, and a qualitative manner was used to analyze them.

Figs. 2 and 3 show the effect of K_2CO_3 on gas transient release behavior of RS pyrolysis and steam gasification, respectively. As shown in Figs. 2 and 3 during RS pyrolysis process, compared to raw RS in water leached RS the instantaneous release concentration of CO , CH_4 , H_2 increased and instantaneous release concentration of CO_2 decreased. This was mainly because potassium was removed from RS sample. Compared with raw RS pyrolysis, impregnated 3%, 6%, 9% potassium RS released progressively high concentration of instantaneous H_2 , CO_2 . These results are in agreement with the earlier studies by Tada and Yasunishi [29]. The presence of potassium promotes formation of C=O double bond compound and generates some small molecules (such as hydroxyacetaldehyde, acids, CO_2 , etc.) [30,31]. According to Nishimura et al. [32] K_2CO_3 promotes straw cellulose structure decomposition by the following two ways: (1) promotes cleavage of hydrogen bond, C-C and C-H bond; and (2) promotes complete decomposition of cellulose glucosidic by heterolytic mechanism and generates some small molecules, such as acetic acid, formic acid, glyoxal, hydroxyacetaldehyde and acetol [32]. Hence RS impregnated with K_2CO_3 during pyrolysis releases high instantaneous concentration of H_2 , CO_2 .

When gas transient release behavior of five cases was compared (Fig. 3), the maximum appearance time of CO from gasification

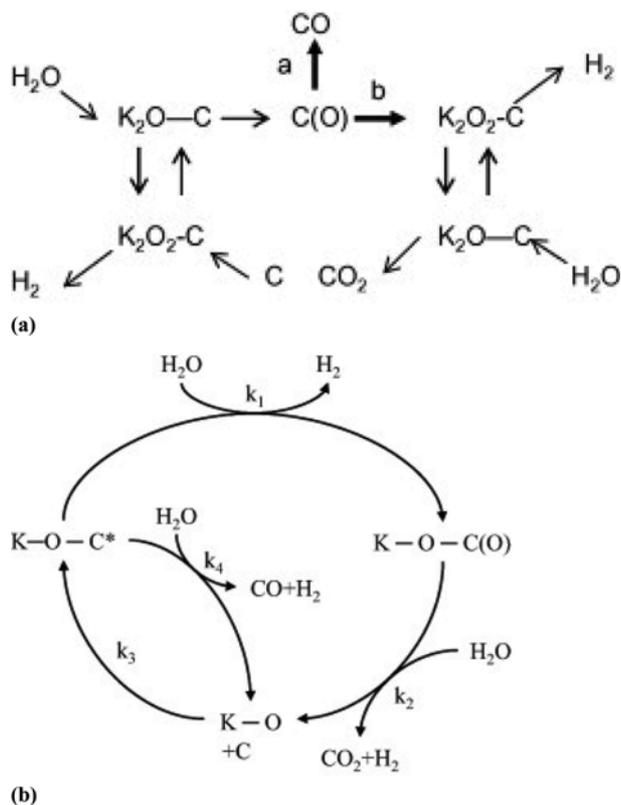


Fig. 4. (a) An oxygen transfer and C(O) intermediate hybrid reaction scheme during steam gasification of RS catalyzed by potassium proposed by Wang et al. [25], and (b) the mechanism of K_2CO_3 -catalyzed steam gasification of char for kinetic modeling suggested Wu et al. [26].

had no obvious change. For water leached RS, H_2 appeared 1 min early. For 3% K_2CO_3 RS and 6% K_2CO_3 RS no change in H_2 appearance time was noticeable, and for 9% K_2CO_3 RS, H_2 appearance was delayed by 1 min. This was because the volatilization of pyrolysis and char gasification interacts with each other during steam gasification. Pyrolysis gases CO , CH_4 , tar and other hydrocarbons react with steam by water-gas shift reaction (3), steam-methane reforming reaction (4), and steam-tar reforming reaction (7) to affect both gas composition as well as distribution. The presence of K_2CO_3 promotes all above reactions besides secondary cracking reaction of tar and other macromolecular hydrocarbons. As shown in Fig. 3, compared with gas transient release behavior of water leached RS gasification, it was found that CO , CH_4 instantaneous release concentration of raw RS, impregnated 3% K_2CO_3 , 6% K_2CO_3 , 9% K_2CO_3 RS increased significantly, while the CO_2 , H_2 instantaneous release concentration decreased (except for 3% K_2CO_3 which increased). Summarizing the results, with increase of impregnated K_2CO_3 content in RS, CO , CH_4 instantaneous release concentration decreased gradually and CO_2 , H_2 instantaneous release concentration increased progressively. In addition, H_2 instantaneous release concentration presented a sharp trend in the case of 6% K_2CO_3 RS gasification and this trend decreased for 9%. K_2CO_3 . The mechanism proposed by Wigmans et al. [25] in Fig. 4 well explains the reason for increase in release instantaneous

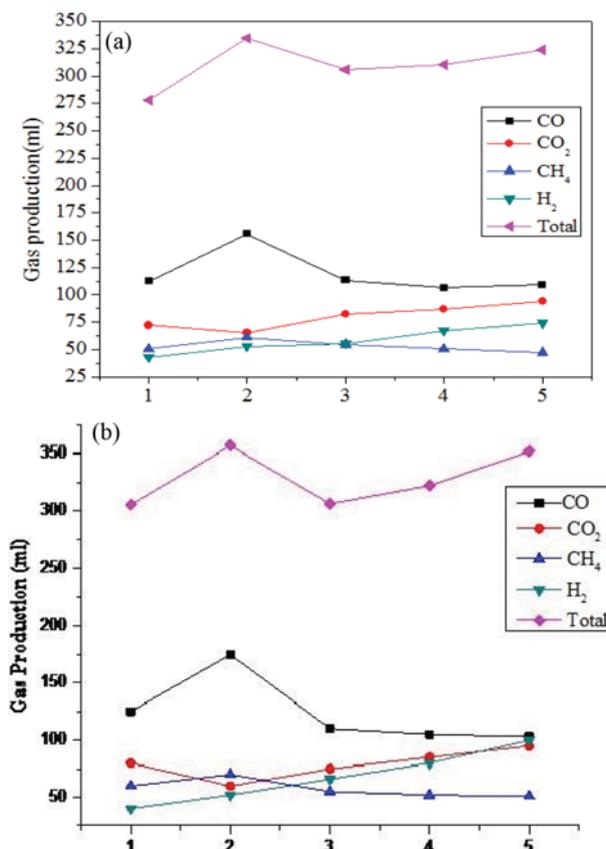


Fig. 5. The effect of K_2CO_3 on gas component distribution and total gas yield during (a) pyrolysis, and (b) steam gasification of (1) Raw RS, (2) water leached RS, (3) 3% K_2CO_3 RS, (4) 6% K_2CO_3 RS, and (5) 9% K_2CO_3 RS.

concentration of CO_2 , H_2 with the increase of K_2CO_3 content in RS. K_2CO_3 catalysis RS pyrolysis by promoting CO_2 , H_2 generation, and promoting CO , CH_4 , tar and hydrocarbons reacting with steam to contribute CO_2 and H_2 . When pyrolysis happens, char is produced instantaneously, so reaction (2) is accompanied with the entire pyrolysis process.

2. The Effect of Potassium on Gas Component Distribution and Total Yield of Gas Production During RS Pyrolysis and Steam Gasification

Fig. 5 shows the effect of K_2CO_3 on individual component as well as total gas production from pyrolysis and steam gasification of RS. During pyrolysis, CO production in water leached RS was largest, and after K_2CO_3 was impregnated, CO production decreased. CO_2 production increased with the increase in K_2CO_3 content, while the production of CH_4 decreased slightly. H_2 production followed the order: raw RS < water leached RS < 3% K_2CO_3 RS < 6% K_2CO_3 RS < 9% K_2CO_3 RS. Total gas production increased with the increase of potassium content in RS.

During RS steam gasification, CO production followed the order: 9% K_2CO_3 RS < 6% K_2CO_3 RS < raw RS < 3% K_2CO_3 RS < water leached RS. CO_2 , CH_4 production exhibited the same regularity as during pyrolysis. H_2 production and total production were in proportional to K_2CO_3 content in RS and followed the order: water leached RS < raw RS < 3% K_2CO_3 RS < 6% K_2CO_3 RS < 9% K_2CO_3 RS. This is

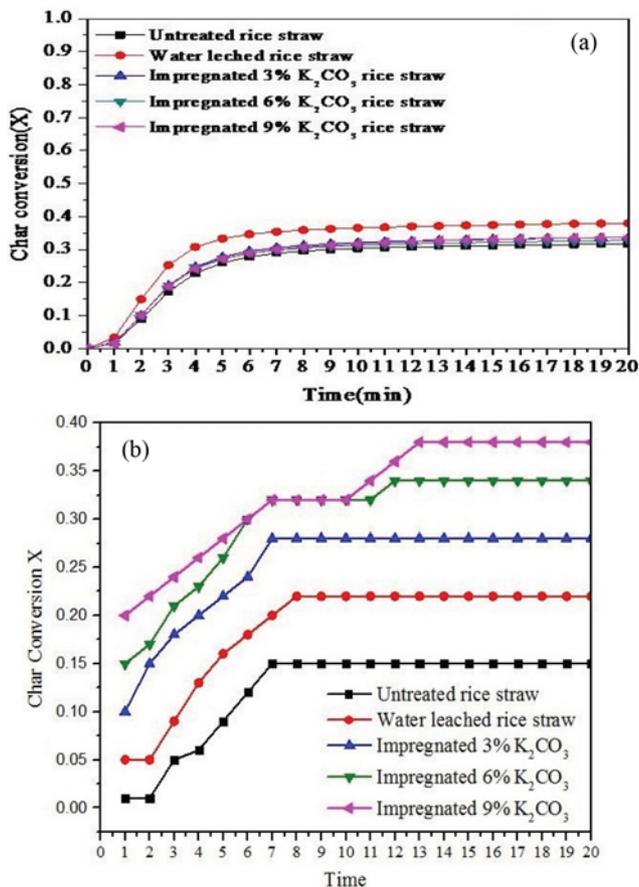


Fig. 6. The effect of K_2CO_3 on reactivity during (a) pyrolysis and (b) steam gasification of raw RS, water leached RS, 3% K_2CO_3 RS, 6% K_2CO_3 RS and 9% K_2CO_3 RS.

mainly due to the catalysis effects of K_2CO_3 on mainly reactions (2), (3). CO production decreased, while CO_2 , H_2 and total gas production increased. It is observed that the maximum H_2 yield percentage was produced at 9% of K_2CO_3 in the studied range (3%, 6%, and 9%) of K_2CO_3 catalyst in both pyrolysis and steam gasification.

3. The Effect of K_2CO_3 on Reactivity During RS Pyrolysis and Steam Gasification

Carbon conversion rate is usually used to evaluate the reactivity during pyrolysis and steam gasification. The reactivity of RS during pyrolysis and steam gasification was analyzed by carbon conversion rate, and results are shown in Fig. 6(a) and (b). During RS pyrolysis process, carbon conversion rate of all five samples of RS (raw RS, water leached RS, 3% K_2CO_3 RS, 6% K_2CO_3 RS and 9% K_2CO_3 RS) reached maximum at 7 min, and there were consistent gas release characteristics during pyrolysis that ended at 7 min (Fig. 6). For all five samples of RS, water leached RS had the highest carbon conversion rate and showed the highest reactivity. Impregnated K_2CO_3 RS had almost the same carbon conversion rate. The reasons are that during pyrolysis, K_2CO_3 promotes the release of CO_2 while at the same time inhibits the release of CO and CH_4 . Both promotion and inhibition effect are proportional to K_2CO_3 content.

During RS steam gasification process, with the increase of K_2CO_3 content in RS both carbon conversion rate as well as steam gasification reactivity increased. This trend increased significantly from 3% K_2CO_3 RS to 6% K_2CO_3 RS; however, from 6% K_2CO_3 RS to 9% K_2CO_3 RS this trend was non-significant.

4. The Effect of K_2CO_3 on Quality of Gas Evaluation During RS Pyrolysis and Steam Gasification

H_2 yield and H_2/CO ratio are regarded as a standard to evaluate gasification gas quality [33-35]. Fig. 7 shows the effect of K_2CO_3 on H_2 yield during RS pyrolysis and steam gasification. During pyrolysis as well as steam gasification H_2 yields followed the order: water leached RS < raw RS < 3% K_2CO_3 RS < 6% K_2CO_3 RS < 9% K_2CO_3 RS, whereas the numerical values were 15.5%, 15.8%, 18.2%,

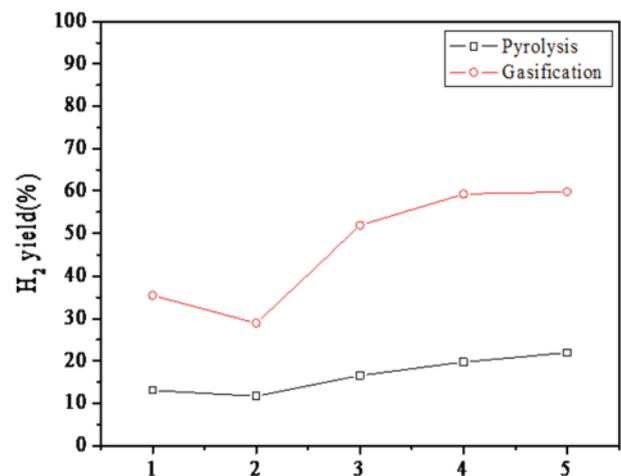


Fig. 7. The effect of K_2CO_3 on H_2 yield during pyrolysis and steam gasification of (1) Raw RS, (2) Water leached RS, (3) 3% K_2CO_3 RS, (4) 6% K_2CO_3 RS, and (5) 9% K_2CO_3 RS.

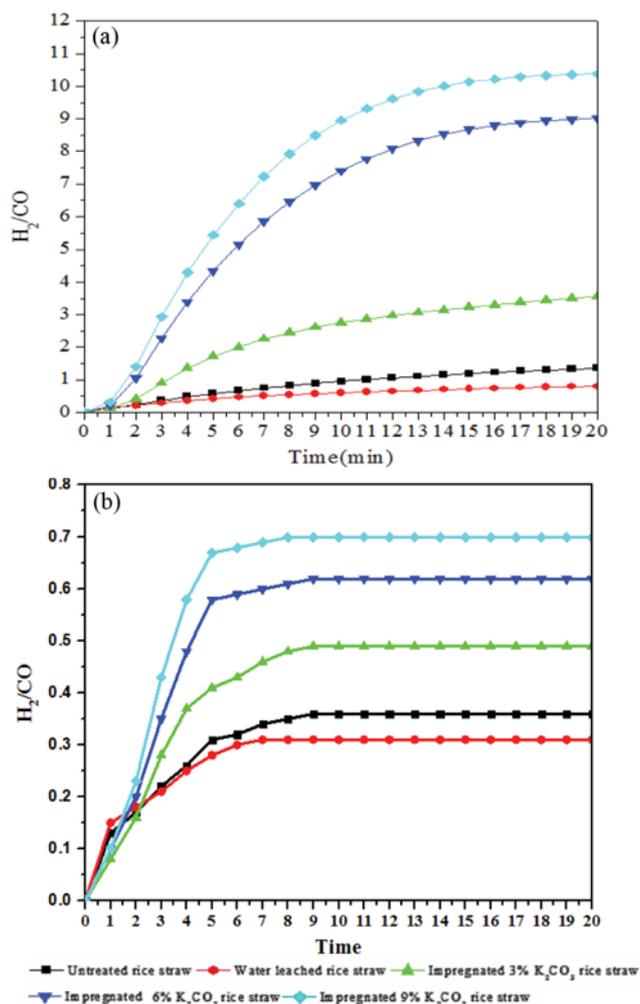


Fig. 8. The effect of K_2CO_3 on H_2/CO ratio during pyrolysis and steam gasification of raw RS, water leached RS, 3% K_2CO_3 RS, 6% K_2CO_3 RS and 9% K_2CO_3 RS.

21.6% and 22.9%, respectively. While during steam gasification, H_2 yield was 28.9%, 35.5%, 51.9%, 59.2% and 59.8% respectively.

Fig. 8 shows the effect of K_2CO_3 on H_2/CO ratio during RS pyrolysis and steam gasification. During both RS pyrolysis and steam gasification, H_2/CO ratio was proportional to K_2CO_3 content. Higher the K_2CO_3 content, the higher H_2/CO ratio. During RS pyrolysis, H_2/CO ratio was less than 1, which is unfavorable to gas end-use. However, during steam gasification of RS, H_2/CO ratio of raw RS gasification was 1; such gas can be applied to engine power. When K_2CO_3 content was higher than 3%, H_2/CO ratio was much greater than 3, and H_2 yield was more than 50%. A high H_2/CO ratio is required for producing liquid fuels through Fischer-Tropsch synthesis and also benefits the production of H_2 rich gas [36].

CHALLENGES AND FURTHERS STUDIES

Potassium is one of the major nutrients and the potassium content in the RS is comparatively high [37]. The inherent alkali metals account for the high reactivity of biomass char [38]. The impregnation of potassium in RS has advantages of lower operating

temperature, lower investment cost and selectivity to certain gas products (higher H_2 content). However, subsequent recovery of impregnated K_2CO_3 is difficult and not always cost effective. It also increases K_2CO_3 content in the residual ash after char gasification [27]. Besides K_2CO_3 content in RS, other process parameters like steam/biomass ratio, reactor temperature, mean residence time, biomass particle size distribution also play a major role in the H_2 yield during pyrolysis and steam gasification. Their synergistic interactive effect in presence of K_2CO_3 needs further study studies and analyses.

CONCLUSIONS

This study evaluated the effect of the presence of K_2CO_3 during the pyrolysis and steam gasification of RS. We found that the increase in K_2CO_3 content in RS during the pyrolysis of RS resulted in increase in instantaneous concentration of both H_2 and CO_2 , while during subsequent gasification the instantaneous concentration of CO and CH_4 decreased while CO_2 and H_2 increased when the K_2CO_3 content in RS was increased. During gasification, both the production and yield of CO_2 and H_2 increased with the increase in K_2CO_3 content in RS. Water-leached RS showed the highest carbon conversion rate and reactivity during pyrolysis, while K_2CO_3 impregnated RS and raw RS showed almost the same carbon conversion rate. However, the gasification reactivity of RS was proportional to its K_2CO_3 content. When K_2CO_3 content in RS was higher than 3%, H_2 concentration in gas produced was more than 50%. The yield of H_2 was highest at 59.8% when K_2CO_3 in RS was 9% (Fig. 7). These results indicate that the presence of K_2CO_3 during pyrolysis and steam gasification of RS effectively assists in production of H_2 -rich synthetic gas.

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