**Steam reforming of tar derived from *Fallopia Japonica* stem over its
own chars prepared at different conditions**

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Abstract

In order to understand the promoting effect of char in different states on the steam reforming of biomass-derived tar during the gasification process, *Fallopia Japonica* stem (FJS) chars were prepared at different pyrolysis temperatures with different pyrolysis time, and used as the catalyst for the reforming of tar derived from FJS itself in a fixed-bed reactor. As-prepared FJS chars were characterized using Scanning Electron Microscope (SEM), Fourier Transform Infra Red spectroscopy (FT-IR), and Brunauer-Emmet-Teller (BET) surface area measurement for clarifying the relationship between the promoting effect and the physical and chemical structures of the char. It was found that BET surface area, chemical structure, and the contents of alkaline and alkaline earth metals (AAEM) in the char, which were obviously affected by pyrolysis temperature and time, had significant effect on char reactivity as well as the catalytic activity on tar reforming. The char obtained at lower temperature (<700 °C) had lower BET surface area but higher active chemical components, thus showed higher gasification reactivity but lower promoting property on tar reforming. With the increase of BET surface area, the reactivity of char itself decreased at first due to the decrease of active chemical components, and then increased to some extent due to the increase in BET surface area. On the contrary, the promoting effect of char on the tar reforming

37 increased at first and then decreased due to the decrease of AAEM species in the char
38 and the shrinkage of old larger pores in char which could hinder the contact between
39 FJS-derived tar and char.

40 **Keywords:** *Fallopia Japonica*; biomass char; pyrolysis; steam reforming of tar; char
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56 **1. Introduction**

57   Nowadays, the utilization of biomass as an alternative energy resource has been
58 attracted great attention over the world. Many researchers try to fully utilize biomass
59 through thermo-chemical conversion ways, which can produce various clean
60 intermediates for chemicals and fuel synthesis or direct generation of power [1, 2].
61 Pyrolysis, i.e., decomposition of biomass at certain temperatures in the absence of
62 oxygen, is a general thermo-chemical conversion method for biomass utilization. In this
63 process, biomass can be decomposed into non-condensable light gases such as H₂, CO,
64 CH₄ and CO₂, condensable liquid (tar), and carbonaceous solid residue (char) with ash
65 [3]. It is a main way to prepare biomass-derived tar as well as char. Biomass-derived tar
66 is an important feedstock for production of aromatic chemicals [4]. It can be also
67 generated during gasification process [3, 5]. However, in this case, the tar is usually
68 considered as the main threat for the operation since it could cause serious gas line
69 blocking and affect the normal operation of gasifier. Moreover, some light tar might also
70 enter the gas collector which can decrease the quality of the produced syngas [4]. Thus,
71 the removal of tar becomes necessity in the gasification process.

72   Hot cleaning gas technology, in which the tar is catalytically cracked and/or

reformed to syngas, is considered as the best way to treat the tar problem in the gasification process. Many researchers have tried to utilize natural minerals as catalysts for the tar reforming due to its abundance and low cost [6-9]. Some noble metals and Ni-based catalysts with good catalytic activity have been developed, but such catalysts are too expensive and yet have higher possibility of deactivation due to coke deposition [10]. As an alternative, coal and biomass derived char and/or char-supported catalysts have been applied as the tar reforming catalyst [10-14] due to its low cost but quite high catalytic activity. Wang et al. [10] studied the catalytic activities of wood char, coal char, Ni/wood char, and Ni/coal char for tar reforming. They found that these kinds of chars, especially coal char, were good supports for Ni catalyst. Zhang et al. [11] investigated the specific reactivity change of acid-washed brown coal char (H-form char) and brown coal char-supported iron catalyst before and after steam reforming reactions. They found that the specific reactivity of H-form char remained similar before and after reactions, but significant change on specific reactivity of brown coal char-supported iron catalyst occurred due to the change in char properties and iron-containing species in the catalyst. Min et al. [12] compared the catalytic activity of H-form char, char-supported iron catalyst, and char-supported Ni catalyst, and found that the difference of catalytic activity between char-supported iron catalyst and char-supported

Ni catalyst decreased with increasing reaction temperature. Min et al. [13] also compared the catalytic activities of biomass char, brown coal char, and biomass/brown coal char-supported iron catalyst for steam reforming reactions, and found that the iron-loaded biomass char showed higher activity for the tar reforming than iron-loaded brown coal char. Wang et al. [14] studied the catalytic steam reforming of cellulose-derived compounds, and found that the steam itself had minor effect on tar reforming without the presence of char-supported catalyst. Several studies concerned on the effects of the char preparation methods on the char properties [15-27]. It is found that the preparation parameters such as pyrolysis temperature and heating rate, and the initial characteristics of biomass such as water and ash content had great influences on the reactivity and structures of the prepared chars. Although these mentioned literatures described clearly the promoting effect of various char and various metal loadings on the tar reforming, neither of these literatures focused on understanding the promoting effect of char in different states on the tar reforming due to their different char preparation conditions. Moreover, it should be noted that the char itself will be reformed simultaneously during the reforming of tar, and how to recover the spent char and reuse it should be also important.

Fallopia Japonica (FJ) is one type of fast-growing perennial herbaceous

biomass which is easily found on the road side, near the river, or in unused landscape [28-30]. This kind of biomass is classified as non-commelinid fibrous magnoliophyta biomass which has high content of lignin and intermediate content of cellulose, hemicellulose, total soluble sugars, protein, and mineral compounds [31]. However, it is seldom studied in details as a feedstock for energy source. Therefore, FJ, particularly its stem part, was selected as the biomass feedstock in this study and its gasification properties were investigated at first. Then, in order to understand the promoting effect of char in different states on the steam reforming of biomass-derived tar during the gasification process, *Fallopia Japonica* stem (FJS) chars were prepared at different pyrolysis temperatures and pyrolysis time, and used as the catalyst for the reforming of FJS-derived tar in fixed-bed reactor. The relationship between the promoting effect and the physical and chemical properties of char prepared at different conditions was discussed.

2. Experimental

2.1 Materials

Fallopia Japonica as a biomass feedstock was obtained from Aomori city, Japan, by harvesting the same stem parts from the living plant at autumn season. Prior

to the experiment, FJS were washed and then dried in air under sunlight for several days, and called as the as-received FJS. The as-received FJS were then cut and sieved to a size of $1.0 < d_p < 2.8$ mm and dried in oven at 105 °C for 24 hours before storage for further use. Table S-1 shows the intrinsic properties of the as-received FJS. Ultimate analyses were conducted using Vario EL cube elemental analyzer. Ash content of FJS was determined by averaging the measurement results of 3 remaining solid residues after calcination of FJS at 800 °C for 3 h. Table S-2 shows the compositions of biomass ashes which are obtained by averaging the results of 3 times measurement from XRF analysis (Energy Dispersive X-Ray Spectrometer, EDX-800HS, Shimadzu). For comparison purpose, cedar and rice straw were also analyzed using the same procedure.

2.2. FJS char preparation and characterization

In order to investigate the effect of pyrolysis temperature on the physical and chemical structure of char, FJS chars were prepared by pyrolyzing FJS at different final pyrolysis temperatures (500-800 °C) for 1 h with a slow heating rate of 15 °C /min. The obtained chars were called as SC500-1h, SC600-1h, SC700-1h, and SC800-1h, respectively. In order to investigate the effect of pyrolysis time on the physical and chemical structure of char, various FJS chars were prepared by pyrolyzing FJS at 700

°C and for 1, 2, and 4 hours, respectively. The obtained chars were called as SC700-1h, SC700-2h, and SC700-4h, respectively.

Morphologies of the obtained chars were observed using SEM (Hitachi SU6600, Japan). Functional groups on the surface of char were analyzed using FT-IR (JASCO 4200, USA). Surface area of char was examined using Brunauer-Emmet-Teller (BET) method (NOVA 4200e, Quantachrome Instruments, USA).

2.3. Steam reforming of tar over char

Steam reforming of the FJS derived tar over the FJS derived char was conducted in a fixed bed reactor [32], in which FJS particles were placed above the char layer and separated with quartz wool in a holder, and steam was flowed from the top of the reactor to the bottom with carrier gas. For each test, 0.4 g of FJS and 0.25 g of char were used. The height of char bed was about 2 cm. During the experiment, 0.1 g/min steam was introduced as a gasifying agent. Two reaction temperatures, i.e., 650 and 700 °C, were used in this study. The produced gases were flowed through an iced-cold trap and a gas dryer chamber, and then collected in a gas bag. The gases were analyzed by a gas chromatography (GC 7890A, Agilent Technologies USA) using two TCD units. To investigate the gas contribution of the steam reforming of the char, the same amount of

char was gasified alone under the same operation conditions and its gas yield was used as a subtrahend for the total gas yield. All of the gas yields were expressed in mmol per gram of dry ash free biomass/char (mmol/g.daf).

3. Results and discussion

3.1. Gasification properties of FJS

In the present work, in order to compare the steam gasification behaviors of FJS with other kinds of biomass, i.e., cedar and rice straw, steam gasification of each biomass was carried out in the fixed bed reactor at reaction temperature of 650 and 700 °C with steam flow rate of 0.1 g/min. Fig. 1 shows the comparison of gasification properties of FJ stem (FJS) with other kinds of biomass. It can be seen that FJS showed higher gas yield when compared with other two kinds of land-based biomass, suggesting that FJ is suitable to be used as biomass feedstock for producing syngas. However, to date, no report on this kind of biomass for gasification is found. The higher yield of FJS samples compared with other biomass samples is likely due to the intrinsic property of FJS, which might be similar in the case of co-gasification of two different biomass. Kaewpanha et al. observed the synergy effect in co-gasification of Japanese cedar and brown seaweed and found that the gas yield of Japanese cedar increased significantly when co-gasified with brown seaweed due to high content of potassium in

brown seaweed [32]. As shown in Table S-2, more alkali and alkaline earth metals (AAEM) especially K_2O , which could promote tar decomposition as well as char reforming during pyrolysis and steam gasification [33-35], was found in FJS ash than other two kinds of biomass. This intrinsic property of FJS should have some effects on the surface area and the reactivity of residual char [36].

3.2. Gasification properties of FJS char

Fig. 2 compares the gasification properties of different FJS chars prepared by pyrolysis at different temperatures. As-prepared chars were gasified at 700 °C for 2 h with the same condition as for steam reforming of tar over char. In case of SC500-1h char, a high gas yield was obtained while very low char was remained after the gasification. By comparing with SC500-1h, the gas yields for SC600-1h and SC700-1h were decreased and the remained char increased after the gasification. However, for the char obtained at higher pyrolysis temperature, i.e., 800 °C, the gas yield increased again while the remained char decreased. It indicated that the FJS char obtained at lower pyrolysis temperature or higher pyrolysis temperature had higher gasification property. In fact, when FJS was pyrolyzed at lower temperature, more volatiles including tar and AAEM species might still be remained in the char so that its reactivity kept at a high

level. On the other hand, when FJS was pyrolyzed at high temperature, i.e., 800 °C, most of volatiles might be decomposed and result in more porosity in the char matrix [22] and such porous structure could be benefit for the gasification reactivity due to more gasification agent, i.e., H₂O contacting with the char. The increase of gasification reactivity makes char itself easier to be gasified, leading to the increasing of gas yield and the decreasing of char yield. However, if char is considered to be used as catalyst and/or catalyst support, the char itself should have high catalytic activity for tar reforming but low self-gasification reactivity. In other words, the char itself should be difficult to be gasified. On the other hand, SEM morphologies of cross sections of SC500-1h, SC700-1h, and SC800-1h chars, as shown in the Figs. S-1a, b, and c in the supplementary data, indicated that the pore wall became thinner and more porous structure and small cavities appeared with the increase in pyrolysis temperature for the char preparation. The similar results were also reported by others [23, 37-40]. Table 1 shows the BET surface areas of these chars. One can see that the BET surface area of SC800-1h char was much higher than that of SC700-1h char. It could be the main reason why the reactivity of SC800-1h char was higher than that of SC700-1h char.

FT-IR spectrum measurements (as shown in Fig. S-2 in the supplementary data) indicated that the original FJS contained many functional groups which are hydroxyl,

217 aliphatic, olefin, aromatic, carbonyl, and ether. After pyrolyzed at 500 °C, the peaks of
218 hydroxyl, aliphatic C-H structure, and C=C olefinic groups decreased significantly but
219 still remained while the C-O peak of ether structure disappeared and the pattern itself
220 was different with that of original FJS. This indicated that the volatile components were
221 still attached on the surface or within char matrix. This may be the reason why the
222 gasification reactivity of SC500-1h was high. The pattern of FT-IR spectrum of
223 SC600-1h char was quite similar to that of SC500-1h but the peak intensity decreased.
224 This reduced intensity means that more volatiles have been moved out so that the BET
225 surface area increased to some extent (Table 1). As more volatiles were released from
226 the char, the gas yield from the gasification of char should be decreased, implying the
227 decrease of gasification reactivity of char which is in agreement with Fig. 2. On the
228 contrary, the FT-IR spectra of SC700-1h and SC800-1h chars show different patterns
229 where the peak of C=C aromatic structure almost disappeared. The disappearance of
230 C=C aromatic structure peak indicated that the carbon structures of SC700-1h and
231 SC800-1h chars should be different with those of SC500-1h and SC600-1h chars. Since
232 the carbon structures of SC700-1h and SC800-1h was nearly similar, the gasification
233 reactivity difference of both chars should be attributed to the BET surface area
234 difference between them.

Fig. 3 compares the gasification properties of different FJS chars prepared by pyrolysis at 700 °C for different pyrolysis time. These chars were also gasified at 700 °C for 2h with the same condition as before. There is only slight increase of gas yield when prolonging the pyrolysis time from 1 to 2 h. When the pyrolysis time was prolonged to 4 h, the gas yield increased significantly. It should be noted that the char remained decreased with the increase in pyrolysis time. From the morphologies of cross-sections of SC700-1h, SC700-2h, and SC700-4h chars (as shown in Fig. S-3 in the supplementary data), it can be seen that the pore morphology of SC700-1h was relatively homogeneous. As the pyrolysis time prolonged to 2 h, many new smaller pores were developed while some of the old pores were shrunk. Furthermore, as the pyrolysis time was prolonged to 4 h, it is found that more new pores grew while fewer old pores were remained. Table 1 presents the BET surface areas of SC700-1h, SC700-2h, and SC700-4h chars. One can see that the BET surface area increased greatly when the pyrolysis time prolonged from 2 to 4 h. This may be the main reason why the reactivity of SC700-4h char increased greatly when compared with both of SC700-1h and SC700-2h chars. The FT-IR spectra of SC700-1h and SC700-2h (as shown in Fig. S-4 in the supplementary data) indicated that aromatic structures were still remained on the surface of char. However, For SC700-4h char, almost no peak

related to the aromatic functional groups existed on the char surface.

Considering the total gas yield from the gasification of char itself with respect to the BET surface area of FJS char, Fig. 4 can be obtained. One can see that gas yield decreased with the increase of BET surface area to approximately 26 m²/g, and then increased with the increase of BET surface area until approximately 100 m²/g and then the gas yield remained almost stable with the increase of the BET surface area. In this study, with the change of pyrolysis condition, the char with different BET surface area can be obtained. In general, high surface area is beneficial for its gasification. However, in order to obtain higher surface area, higher temperature and/or longer pyrolysis time are necessary, and as such, more AAEM species will also be evaporated from the char. In general, these AAEM species have greater promoting effect on the gasification of char itself. Thus, by increasing pyrolysis temperature and/or prolonging holding time at certain value, the increase of BET surface area in exchange with the decrease of AAEM species should give the results as shown in Fig. 4.

3.3. Steam reforming of tar over FJS char

Fig. 5 compares the gas yield from gasification of FJS without and with the FJS char prepared by pyrolysis at different temperatures. As stated above, in this experiment,

the FJS-derived tar was carried with the carrier gas as well as steam into the char layer below the biomass layer, and reformed on the surface of the char. Note that the gas yields produced from the steam reforming of char itself was subtracted from the total gas production yields to obtain the net gas yield from steam reforming of tar over char beds, assuming that the gas yields from the char itself are same in both cases with and without the tar. One can see that more gas was produced in the presence of char, suggesting that the FJS derived tar was steam reformed in the char bed. No significant difference was observed when using SC500-1h and SC600-1 h chars while much larger amount of gas was produced when SC800-1h chars were used. As shown in Fig. 2, SC500-1h and SC600-1h chars themselves were easily gasified since they contained more volatiles as well as more AAEM species. In this case, during the gasification process, some gases such as H₂, CO, CO₂ and CH₄ were produced from the char itself, and these produced gases surrounded the char particles and hindered the biomass-derived tar to contact with its surface. In addition, the existence of aromatic functional group in SC500-1h and SC600-1h chars, as shown in Fig. S-2, will in turn decrease the reactive sites on char and lowered its promoting effect [12]. On the other hand, SC700-1h char itself was not easy to be gasified due to its low BET surface area and low contents of volatiles as well as AAEM species. In this case, nascent tar derived

from the biomass should be difficult to penetrate deeper into the char matrix. As such, the promoting effect for the tar reforming is also not so obvious. However, for SC800-1h char, it is easier to be gasified due to its high BET surface area. Simultaneously, the promoting effect for the tar reforming is also obvious since the possibility of nascent tar penetrating more deeply into the char matrix is high due to its high BET surface area.

Fig. 6 compares the gas yield from gasification of FJS without and with the FJS char prepared by pyrolysis at 700 °C for different pyrolysis time. It can be seen that the yield of CO, CO₂, and particularly H₂ increased significantly when chars were utilized. However, the increase of CH₄ was not quite significant in the presence of char and the CH₄ yields at 650 and 700 °C were quite the same. Of all the gas yields, the H₂ yield increased significantly when SC700-2h char were used but decreased when SC700-4h char was used. Table 2 shows that the content of alkaline species, i.e., K, in the as-prepared char decreased with the increase in pyrolysis time for the char preparation. This might be the main reason why the gas yield decreased when SC700-4h char was used as catalyst, even though its BET surface area increased to a great extent since AAEM species in general have good catalytic activity for the tar reforming.

Considering the total gas yield from the gasification of FJS in the presence of

FJS char with the increase of the BET surface area, Fig. 7 can be obtained. One can see that the promoting effect for the tar reforming increased at first and then decreased with the increase of BET surface area. As stated above, with the increasing of BET surface area of char, the contents of AAEM species in the char could be decreased. However, these AAEM species generally have great promoting effect on the tar reforming. Therefore, in the present study, an optimum surface area, i.e., approximately 100 m²/g, is existed.

4. Conclusions

Gasification properties of *Fallopia Japonica* stem (FJS) and its char prepared at different conditions, and the steam reforming of biomass-derived tar over these prepared char were investigated in details, and the following results were obtained:

(1) Comparing with other kinds of land-based biomass, FJ is a kind of easily gasified biomass feedstock due to its high content of AAEM species.

(2) Pyrolysis temperature had significant effect on the promoting properties of char for the tar reforming due to its obvious influence on the evolution of pore and the change of carbon chemical structure. The char itself prepared at lower temperatures had higher self-gasification reactivity but showed lower promoting property for the tar

reforming. On the other hand, the promoting effect increased to some extent at first and then decreased when the char was prepared with the increase in the pyrolysis time, indicating that the optimum pyrolysis time existed for the preparation of char with the most catalytic activity for the tar reforming.

(3) The BET surface area of the prepared char had great effect on the gasification properties of char itself as well as on the steam reforming of tar over the char. The promoting effect for the tar reforming increased at first and then decreased with the increase of BET surface area due to the decrease of AAEM species content. An optimum surface area, i.e., approximately 100 m²/g, is existed in the present study.

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Table captions

Table 1. BET surface area of FJS char prepared by pyrolyzed at different conditions

Table 2. Ash compositions of FJS char prepared by pyrolyzed at 700 °C for different
pyrolysis time.

Figure captions

Fig. 1 Comparison of gasification properties of FJ stem (FJS) with other kinds of
biomass (Gasification temperatures: 650 and 700 °C).

Fig. 2 Gasification properties of different FJS chars prepared by pyrolysis at different
temperatures. (Gasification temperatures: 700 °C).

Fig. 3 Gasification properties of different FJS chars prepared by pyrolysis at 700 °C for
different pyrolysis time. (Gasification temperatures: 700 °C).

Fig. 4 The relationship between gas yield and char remained from gasification of char
itself and its BET surface area.

Fig. 5 Gas yield from gasification of FJS without and with the FJS char prepared by
pyrolysis at different temperatures. (Gasification temperatures: 700 °C).

Fig. 6 Gas yield from gasification of FJS without and with the FJS char prepared by
pyrolysis at 700 °C for different pyrolysis time. (Gasification temperatures: 700 °C).

Fig. 7 The relationship between gas yield from gasification of FJS with the FJS char

469 and BET surface area of the FJS char.

Table 1. BET surface area of FJS char prepared by pyrolyzed at different conditions

Char code	BET surface area (m ² /g)
S500-1h	8
S600-1h	19
S700-1h	26
SC700-2h	60
S800-1h	105
SC700-4h	189

Table 2. Ash compositions of FJS char prepared by pyrolysis at 700 °C for different pyrolysis time.

Element	Concentration (%)		
	SC700-1h	SC700-2h	SC700-4h
Ca	19.0	42.4	62.5
K	74.2	53.5	33.5
P	3.6	1.9	1.2
S	1.1	0.97	0.96
Si	0.6	0.62	0.32
Mn	0.09	0.16	0.18
Sr	0.04	0.09	0.12
Fe	0.06	0.06	0.08
Cu	0.05	0.05	0.01
Zn	0.06	0.03	0
Others	1.2	0.22	1.13

Figure-1

Figure-2

Figure-3

Figure-4

Figure-5

Figure-6

Figure-7