IJE TRANSACTIONS C: Aspects Vol. 28, No. 9, (September 2015) 1251-1256

International Journal of Engineering

Journal Homepage: www.ije.ir

Catalytic Effect of Metal Species on Enhancement of CO_2 Gasification Reactivity of Biomass Char

P. Lahijani[a], M. Mohammadi[b*]

[a] *Biomass and Bioenergy Laboratory, School of Mechanical Engineering, Universiti Sains Malaysia, Nibong Tebal, Pulau Pinang, Malaysia*
[b] *Faculty of Chemical Engineering, Babol Noushirvani University of Technology, Babol, Iran*

PAPER INFO

Paper history:
Received 15 June 2015
Received in revised form 30July 2015
Accepted 03 September 2015

Keywords:
CO_2 gasification
Biomass Char
Catalyst
Boudouard Reaction

ABSTRACT

In the Boudouard reaction, where CO_2 is reacted with carbon (char) to produce CO, very high temperatures are required to shift the equilibrium towards CO production. This endothermic reaction is inherently slow and catalytic species are effective to speed up the reaction rate at temperatures below 900 °C. In this study, the catalytic effect of some alkali (K, Na), alkaline earth (Ca) and transition (Fe) metals on enhancing the CO_2 gasification reactivity of pistachio shell (PS) char was investigated. The CO_2 gasification studies were performed in a Thermogravimetric analyzer (TGA). Among the examined potassium species, K_2CO_3 showed the highest catalytic effect; wherein, complete carbon conversion was achieved 48.1% faster as compared to un-catalyzed PS char. The highest catalytic effect among the sodium salts was devoted to $NaNO_3$ which showed 57.7% enhancement in the reactivity of char. $CaCl_2$ and $Fe(NO_3)_2$ also showed the best catalytic performance among the examined calcium and iron species and improved the reaction rate by 64.6 and 46.1%, respectively.

doi: *10.5829/idosi.ije.2015.28.09c.01*

1. INTRODUCTION

Carbon dioxide (CO_2), as one of the major greenhouse gases, has significant contribution to the global warming. The global emission of CO_2, mostly emanating from consumption of fossil fuels, reached a total of 34.5 billion tones in 2012 [1]. This most serious human provoked environmental issue might have a chilling perspective, if it could not be alleviated through rational strategies.

Among the schemes proposed for reduction of CO_2 emission, CO_2 gasification which is the thermochemical conversion of this greenhouse gas in the presence of char to other useful products looks to be a promising solution. A straightforward reaction to activate CO_2 and split its constituent atoms is the "Boudouard reaction" also known as "char gasification" in which CO_2 is reacted with carbon to produce carbon monoxide [2]:

$$CO_2 + C \leftrightarrow 2CO \quad \Delta H = 172 \text{ kJ/mol} \qquad (1)$$

The CO produced via the Boudouard reaction can be utilized for production of methanol and Fischer-Tropsch hydrocarbons in combination with H_2 as well as synthesis of a number of chemicals [3, 4]. However, since the Boudouard reaction is highly endothermic and the natural surface chemistry of char is not potent enough to promote the heterogeneous gas-solid reaction, high temperatures, typically >700 °C, are required to shift the equilibrium of the reaction towards CO production.

Addition of catalyst to the carbon material is considered as an attractive option to accelerate the reaction rate at a lower reaction temperature [5-9]. Use of catalyst contributes to the higher gasification reactivity, reduce the capital costs and severity of the reactor condition [10-12].

A number of researches have been performed utilizing alkali, alkaline earth and transition metal salts and oxides as catalyst to enhance the gasification reaction of a range of carbonaceous materials. Huang et al. [13] investigated the influence of some alkali (K and Na), alkaline earth (Ca and Mg) and transition (Fe)

*Corresponding Author's Email: m.mohammadi@nit.ac.ir (Maedeh Mohammadi)

Please cite this article as:P. Lahijaniand M. Mohammadi, Catalytic effect of metal species on enhancement of CO_2 gasification reactivity of biomass char,International Journal of Engineering (IJE), TRANSACTIONS C: Aspects Vol. 28, No. 9, (September2015) 1251-1256

metals on the CO_2 gasification reactivity of fir char and reported the catalytic effect of the metals in the order K> Na> Ca> Fe> Mg. In another study, Lahijani et al. [14] investigated the CO_2 gasification reactivity of pistachio shell char in the presence of nitrate catalysts. The catalytic effect of the catalysts were found to be in the order of $NaNO_3$> $Ca(NO_3)_2$> $Fe(NO_3)_3$> KNO_3> $Mg(NO_3)_2$. Similarly, Sun et al. [15] studied the effect of nitrate salts on CO_2 gasification reactivity of marcel chars and found the catalytic effect in the sequence of $NaNO_3$> $Ca(NO_3)_2$> $Fe(NO_3)_3$> KNO_3. Karimi and Gray [11] studied the catalytic effect of some alkali and alkaline earth compounds including Na_2CO_3, KCl, K_2CO_3, $CaCO_3$, CaO and MgO on steam gasification of bitumen coke. The results showed that the highest catalytic effect was devoted to Na_2CO_3 and K_2CO_3 and to a lower extent to KCl, while Mg and Ca did not promote the catalytic activity.

Although in many catalyzed CO_2 gasification studies, alkali metals presented higher reactivity than alkaline earth and transition metals [13, 16, 17], however, this might not be considered as a general pattern and the type of carbon char and its physicochemical properties also play an important role in the rate of the reaction. Based on this background, catalytic CO_2 gasification of pistachio shell (PS) char as a locally available lignocellulosic waste was studied. Several species of alkali, alkaline earth and transition metals were utilized and their catalytic influence on the CO_2 gasification reactivity of PS char was investigated.

2. MATERIALS AND METHODS

2. 1. Raw Material Pistachio shell (PS) was obtained from local market. The shells were thoroughly washed to remove the adhered salt and dried at 105 ^oC in an oven. The fixed carbon, volatile mater, moisture and ash content of the PS was determined using thermogravimetric analyzer (TGA, SDTQ-600), based on standard method [18]. The biomass sample was heated in TGA at 105 °C for 180 min to remove the moisture. The sample was then heated to 900 °C under N_2 to prevent oxidation. The weight loss calculated at this stage was referred to as volatile matter. The remaining sample (char) was then heated at 750 °C under air, so that the sample was completely combusted. The weight loss subsequent to combustion was considered as the fixed carbon, while the residual was ash.

The elemental composition of the biomass residue was determined using an elemental analyzer (Perkin-Elmer 2400 Series II CHNS/O). The analytical range for the elements was as follows: C (0.001-3.6 mg), H (0.001-1.0 mg), N (0.001-6.0 mg), S (0.001-2.0 mg) and O (0.001-2.0 mg). The results of ultimate and proximate analyses are presented in Table 1.

TABLE 1. Ultimate and proximate analyses of pistachio shell

Ultimate analysis (wt %)				Proximate analysis (wt %)			
C	H	N	O	Moisture	Fixed carbon	Volatile	Ash
58.2	4.1	1.0	36.7	2.8	16.3	79.8	1.1

2. 2. Pistachio Shell Char Preparation A lab-scale carbonization system was used to prepare PS char. The carbonization unit consisted of a stainless steel tube reactor with 4 cm diameter and 80 cm length, surrounded by a vertical tubular furnace. In each pyrolysis experiment, 100 g PS was loaded in the reactor and heated under a nitrogen flow of 400 ml/min at a heating rate of 15 ^oC/min to 900 ^oC. This temperature was maintained constant for 90 min. Selection of this temperature for carbonization was based on the degradation profile obtained for PS in Thermogravimetric analyzer (TGA) under N_2 as presented in Figure 1. As could be inferred from the weight loss profile, the carbonization temperature of 900 ^oC was high enough to ensure that complete decomposition of cellulose, hemicelluloses and lignin took place and carbonaceous char remained after devolatilization [19]. The derivative thermogravimetric analysis (DTG) profile of PS, which corresponds to the region where the slope of TG curve is almost constant, showed three peaks. The first small peak around 100 ^oC corresponds to the removal of moisture. The two sharp devolatilization peaks correspond to the decomposition of lignocellulosic compounds. After carbonization, the char sample was cooled down to room temperature under nitrogen. The carbonized sample was then crushed and ground to a particle size of <75 μm. The prepared char was stored in desiccator for further experiments.

2.3. Loading of Catalyst on PS Char In order to prepare metal catalyzed PS char, several metal compounds were introduced into the char framework by wet impregnation method.

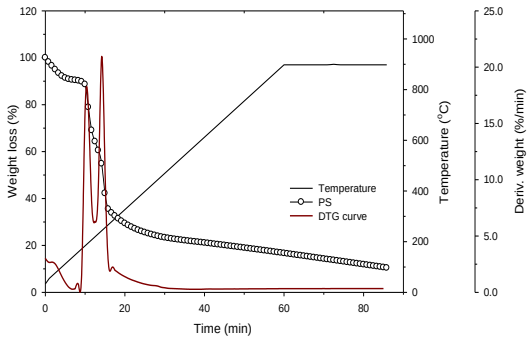

Figure 1. Weight loss, DTG and temperature profile for pyrolysis of PS in TGA under the atmosphere of N_2

For this purpose, several aqueous solutions of alkali (K and Na), alkaline earth (Ca) and transition (Fe) metals were prepared by dissolving or mixing quantitative amounts of various metal species in deionized water. Then, 1g of PS char powder was impregnated in 80 ml of the metal contained solution and stirred for 12 h. The solution concentration was adjusted to attain 3 wt% metal (Na, K, Ca and Fe) loading in the char. The mixtures were then oven dried at 105 ^{o}C for 48 h. The metal loaded PS chars were stored for CO_2 gasification experiments. Table 2 summarizes the chemicals used in this study to prepare metal catalyzed char.

2. 3. CO_2 Gasification Experiments CO_2 gasification reactivity of PS char, either pure or catalyzed, was studied under iso-thermal temperature condition in a Thermogravimetric analyzer (TGA, SDTQ-600). In each experiment, 7-8 mg of PS char was loaded in a ceramic pan and heated at a rate of 40 ^{o}C/min under N_2 atmosphere to the pre-set gasification temperature (875 ^{o}C). Selection of this temperature for gasification was based on our previous investigations [14]. At the onset of gasification, N_2 was switched to CO_2 (100 ml/min) for isothermal gasification. The weight loss of the char sample as a function of gasification time was recorded continuously. The gasification conversion (X) was calculated using the following Equation:

TABLE 2. Metal catalyzed PS chars used in CO_2 gasification experiments

Chemicals used	Sample name
-	PS char (pristine)
Potassium species	
$K_2S_2O_8$	PS char + Potassium persulfate
KNO_3	PS char + Potassium nitrate
K_2CO_3	PS char + Potassium carbonate
KCl	PS char + Potassium chloride
KOH	PS char + Potassium hydroxide
Calcium species	
$CaCl_2$	PS char + Calcium chloride
$Ca(NO_3)_2$	PS char + Calcium nitrate
CaO	PS char + Calcium oxide
Sodium species	
NaCl	PS char + Sodium chloride
Na_2CO_3	PS char + Sodium carbonate
$NaNO_3$	PS char + Sodium nitrate
$NaHCO_3$	PS char + Sodium bicarbonate
Iron species	
$FeCl_3.6H_2O$	PS char + Iron(III) chloride
$Fe(NO_3)_3.9H_2O$	PS char + Iron(III) nitrate
$Fe_2(SO_4)_3.H_2O$	PS char + Iron(III) sulfate

$$X = \frac{m_0 - m_t}{(m_0 - m_c - m_{Ash})} \times 100\% \quad (2)$$

where, m_0 represents the weight of the char at the gasification onset , m_t is the real time mass of the sample at time t, m_c is the mass of catalyst and m_{Ash} is the remaining mass of ash after completion of gasification. The weight loss and temperature profile in a typical gasification test in TGA are shown in Figure 2.

3. RESULTS AND DISCUSSION

The influence of metal catalysts on the gasification reactivity of PS char was studied by loading 3 wt% of various salts of alkali, alkaline and transition metal species, as listed in Table 2, into the char skeleton; the achieved results are presented and discussed as follows.

3. 1. Effect of Potassium Species Figure 3 illustrates the catalytic effect of potassium species on the carbon conversion of the PS char. It can be observed that the rate of carbon conversion of catalyzed char was higher than that of non-catalyzed (pristine) char for all potassium species. The char reactivity in the presence of K_2CO_3 was considerably high and complete carbon conversion was achieved in 27.1 min, while this time was 51.7 min for pure PS char. The CO_2 gasification reactivity of char followed the sequence of K_2CO_3-char>K_2SO_4-char> KNO_3-char> KOH-char> KCl-char> raw-char. Such enhancement in the reactivity of catalyzed char was confidently attributed to the significant effect of potassium on increasing the reaction centers which play an important role in chemisorption of CO_2 on the char. It is also most probable that these potassium sites catalyze the breakage and transfer of oxygen from CO_2 to the char surface and subsequent release of CO, based on the Boudouard reaction [13].

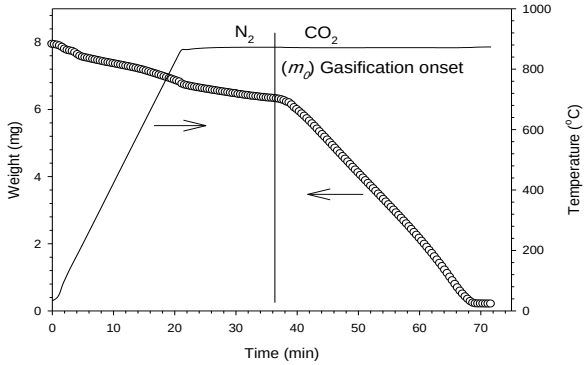

Figure 2. Temperature profile and weight loss in a typical CO_2 gasification experiment in TGA

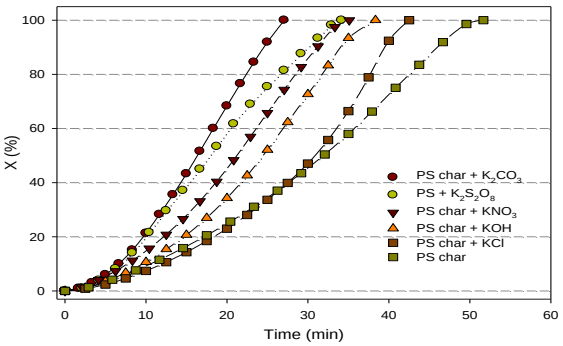

Figure 3. Effect of potassium species on carbon conversion of PS char in CO_2 gasification at 875 ^oC

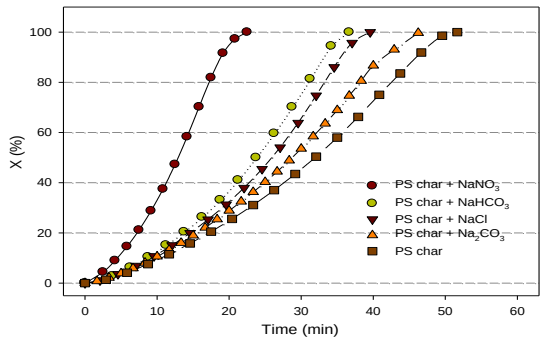

Figure 4. Effect of sodium species on carbon conversion of PS char in CO_2 gasification at 875 ^oC

3. 2. Effect of Sodium Species The carbon conversions of 3% sodium salt loaded char compared to the raw char are shown in Figure 4. The gasification reactivity of all sodium loaded PS chars improved in comparison to the raw PS char in the order of $NaNO_3$-char> $NaHCO_3$-char> NaCl-char> Na_2CO_3-char> raw-PS char which indicates the encouraging effect of this alkali metal on improving the reactivity of biomass char during the CO_2 gasification. The char conversion in the presence of $NaNO_3$ was considerably higher than that of the raw char. The time required for complete conversion of raw PS char was almost 2.3 as that of $NaNO_3$ loaded char. The complete carbon conversion for $NaNO_3$, $NaHCO_3$, NaCl and Na_2CO_3 catalyzed chars was achieved in 22.4, 36.7, 39.6 and 44.5 min, respectively, which was considerably shorter than the corresponding time required for pristine char (51.7 min). These results signify the catalytic effect offered by sodium as alkali metal and the contribution of Na species to increase the number of active sites for the Boudouard reaction on the char surface. It has been suggested that the mechanism through which alkali metals catalyze the Boudouard reaction involves gaseous intermediate compounds M (g), CO (g) and CO_2 (g), where M (g) represents K and Na [20]. The suggested mechanism involves the reduction, oxidation and carbonation reactions as described below:

Reduction: M_2CO_3 (s, l) + 2C (s) ↔ 2M (g) + 3CO (g) (3)

Oxidation: 2M (g) + CO_2 (g) ↔ M_2O (s, l) + CO (g) (4)

Carbonation: M_2O (s, l) + CO_2 (g) ↔ M_2CO_3 (s, l) (5)

Overall: 2C (s) + $2CO_2$ (g) ↔ 4CO (g) (6)

Reaction (3) was assumed to be the rate controlling step, whereas, reactions (4) and (5) were expected to proceed rapidly. Since Na (g) is highly active, it is unlikely that reaction (4) control the overall reaction rate [21].

3. 3. Effect of Calcium Species Calcium is known as a potential catalyst for enhancing the char reaction rate during gasification. Generally, the catalytic activity of calcium might be inferior to that of alkali metals. However, its agglomeration tendency and volatilization during gasification is low [22, 23]. These properties are considered as the superior advantages of calcium over potassium and sodium. The influence of some calcium compounds (3 wt%) on enhancing the gasification reactivity of PS char was investigated; the results are illustrated in Figure 5. As observed in the figure, not all of the calcium specious improved the conversion rate of char during gasification. The reactivity of PS char profoundly enhanced in the presence of $CaCl_2$ and $Ca(NO_3)_2$ by 64.6 and 48.7% as compared to the pristine PS char. However, CaO was not efficient in enhancing the char reactivity. The gasification reactivity of the CaO loaded char was even less than that of raw PS char. Such behavior might be attributed to the un-even or deficient dispersion of CaO on the char surface. It has been reported that the catalytic activity of calcium is significant only when it is well dispersed in the carbon matrix [24, 25]. Considering the very low solubility of CaO in water, it can be speculated that the wet impregnation method, which was used in the current work, was not efficient for homogeneous dispersion of CaO on the char surface.

3. 4. Effect of Iron Species Iron species which are unlimitedly available have been used as promising catalysts for improving the rate of the Boudouard reaction. Figure 6 depicts the catalytic effect of iron species (3 wt%) on the carbon conversion of the PS char. As expected, the carbon conversion rate of the catalytic gasification was higher than that of non-catalytic gasification and the catalytic activity of the iron species followed the sequence of $Fe(NO_3)_3$-char>$FeCl_3$-char> $Fe_2(SO_4)_3$-char> raw-char. The carbon conversion rate of $Fe(NO_3)_3$, $FeCl_3$ and $Fe_2(SO_4)_3$ catalyzed char was 46.2, 32 and 17.4% faster than that of pristine PS char, respectively.

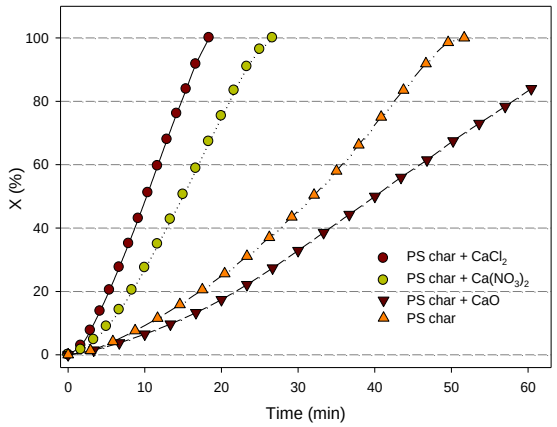

Figure 5. Effect of calcium species on carbon conversion of PS char in CO_2 gasification at 875 ^oC

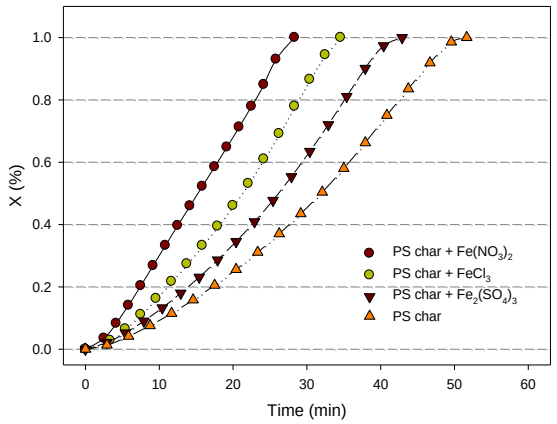

Figure 6. Effect of iron species on carbon conversion of PS char in CO_2 gasification at 875 ^oC

4. CONCLUSION

The influence of several metal species on the CO_2 gasification reactivity of PS char was investigated. The following results were concluded:

The investigated potassium salts enhanced the CO_2 gasification reactivity of char in the order of K_2CO_3-char >K_2SO_4-char > KNO_3-char > KOH-char > KCl-char > raw-char.

Sodium species were also very effective in promoting the char reaction rate in the sequence of $NaNO_3$-char > $NaHCO_3$-char > NaCl-char > Na_2CO_3-char > raw-PS char.

Among the investigated calcium species, $CaCl_2$ and $Ca(NO_3)_2$ profoundly enhanced the char reactivity. However, the reaction rate in the presence of CaO became worsen as compared to pristine char. This was hypothesized to be due to the inefficiency of impregnation method for homogeneous dispersion of catalyst on the char surface.

The catalytic effect of iron salts on promotion of the char reactivity was noticeable, following the order of $Fe(NO_3)_3$-char >$FeCl_3$-char > $Fe_2(SO_4)_3$-char > raw-char.

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P. Lahijani[a], M. Mohammadi[b]

[a] *Biomass and Bioenergy Laboratory, School of Mechanical Engineering, Universiti Sains Malaysia, Nibong Tebal, Pulau Pinang, Malaysia*
[b] *Faculty of Chemical Engineering, Babol Noushirvani University of Technology, Babol, Iran*

PAPER INFO

Paper history:
Received 15 June 2015
Received in revised form 30 July 2015
Accepted 03 September 2015

Keywords:
CO_2 gasification
Biomass Char
Catalyst
Boudouard Reaction

چکیده

در واکنش بودوارد که در آن CO_2 با کربن (ذغال) واکنش داده می‌شود تا CO تولید کند به دماهای بسیار بالا نیاز است تا تعادل واکنش را به سمت تولید CO سوق دهد. این واکنش گرماگیر ذاتا کند است و ترکیبات کاتالیستی می‌توانند سرعت واکنش را در دماهای کمتر از 900^oC افزایش دهند. در این تحقیق، اثر کاتالیستی برخی از فلزات قلیائی (K, Na)، قلیائی خاکی (Ca) و واسطه (Fe) روی افزایش سرعت واکنش گازی سازی ذغال پوست پسته (PS) با CO_2 مورد بررسی قرار گرفت. مطالعه مربوط به گازی سازی در دستگاه TGA انجام شد. از بین ترکیبات پتاسیمی مورد بررسی، K_2CO_3 بیشترین اثر کاتالیستی را داشت به طوری که در حضور آن زمان مورد نیاز برای رسیدن به میزان تبدیل کامل به اندازه 48.1% سریعتر از زمان مشابه برای ذغال بدون کاتالیست بود. بیشترین تاثیر کاتالیستی در بین نمکهای سدیم متعلق به Na_2NO_3 بود که موجب بهبودی به میزان 57.7% در سرعت واکنش شد. $CaCl_2$ و $Fe(NO_3)_2$ بهترین اثر کاتالیستی در میان ترکیبات کلسیم و آهن را داشتند که به ترتیب به میزان 64.6 و 46.1% موجب بهبود سرعت واکنش شدند.

***doi**: 10.5829/idosi.ije.2015.28.09c.01*