Co-production of Biochar, Bio-oil, and Syngas from *Tamarix chinensis* Biomass under Three Different Pyrolysis Temperatures

Muhammad Irfan,[a] Qimei Lin,[a,]* Yan Yue,[a] Xuan Ruan,[b] Qun Chen,[b,]*Xiaorong Zhao,[a] and Xinliang Dong [a]

Pyrolysis of *Tamarix chinensis* feedstock was performed at 300, 500, and 700 °C to investigate the characteristics of biochar, bio-oil, and syngas. Biochar yield decreased and syngas yield increased as the pyrolysis temperature increased. The biochar was characterized for elemental composition, surface, and adsorption properties. Values of pH, electrical conductivity (EC), ash, C, K, Na, and basic functional group contents all increased as the pyrolysis temperature increased, whereas P, Ca, Mg, and acidic functional groups decreased. The methylene blue adsorption capacity values were 1.78, 2.08, and 1.96 (mg g^{-1}) and iodine 256.48, 255.51 and 76.42 (mg g^{-1}) for the biochars produced at 300, 500, and 700 °C, respectively. The C and H contents in bio-oil ranged from 66 to 62% and 8 to 7%, while O changed from 25 to 29% when temperature was increased from 300 to 700 °C. The concentration of hydrocarbon gases, such as ethane, ethylene, propane, and acetylene, increased as the pyrolysis temperature increased. The sum of CO and CO_2 occupied great percentage of the total gas, while the H_2 concentration increased markedly to a maximum of 16% at 500 °C. Thus, *T. chinensis* is a potential feedstock for biochar and bioenergy production.

Key words: Tamarix chinensis biomass; Pyrolysis temperature; Biochar; Bioenergy

*Contact information: a: Department of Soil and Water Sciences, College of Resources and Environmental Sciences, China Agricultural University, Beijing 100193, China; b: Key Laboratory for Thermal Science and Power Engineering of Ministry of Education, Department of Thermal Engineering, Tsinghua University, Beijing 100084, China; *Corresponding author: linqm@cau.edu.cn, qunchen@tsinghua.edu.cn*

INTRODUCTION

The current high energy demand and mitigation of greenhouse gas emissions are two large problems, particularly in China. The Chinese government plans to reduce carbon emissions by 40% to 45% between 2005 and 2020 (Wang *et al*. 2014). Biomass resources play an important role in reducing the dependency on fossil fuel and mitigating global warming by reducing greenhouse gas emissions. The Inner Mongolia Autonomous Region, which is the third largest province in China, is rich in biomass resources. People in rural areas of Inner Mongolia usually produce energy by directly burning coal or biomass in traditional stoves, with very low energy efficiencies, which leads to serious environmental problems because of the release of carbon dioxide and sulfur dioxide. Very little attention has been given to replacing these traditional energy methods in this region with sustainable and environmentally-friendly technologies. Among others, pyrolysis technology appears to be a sustainable and environmentally-friendly technology that converts biomass into biochar and bioenergy (Laird *et al*. 2009). All of the co-products of pyrolysis are potentially valuable; thus, pyrolysis technology is considered to be a very efficient way to convert biomass into biochar and bioenergy (Song and Guo 2012).

Biochar, a product of biomass pyrolysis, is rich in carbonaceous material with the potential to sequester carbon (C) because of its highly recalcitrant nature (Laird *et al*. 2009; Lehmann *et al*. 2011). Biochar has been used as a soil amendment in agriculture and to sequester carbon. Amending soil with biochar improves the soil's physical properties (Mukherjee and Lal 2013), enhances fertility (Chan *et al*. 2008; Lehmann *et al*. 2011), and serves as a habitat for microorganisms (Jaafar *et al*. 2014). Because of its recalcitrant nature, biochar carbon can be stored for long periods, thus reducing CO_2, N_2O, and CH_4 emissions (Van Zwieten *et al*. 2010). Several studies have reported that biochar has a high adsorption capacity for contaminants and heavy metals (Xu *et al*. 2011; Abdel-Fattah *et al*. 2015).

The properties of biochar are not only a function of the raw feedstock, but are also dependent on the pyrolysis conditions, such as temperature, heating rate, and residence time. Characterizing the properties of biochar from different biomass materials under different production conditions will enable a mechanistic understanding of the effects of different biochars on soil properties and crop nutrients. Therefore, characterizing the properties of biochar is important before it can be used in agriculture and for environmental management.

The co-production of bio-oil and syngas with biochar makes pyrolysis technology more sustainable and environmentally-friendly than producing biochar alone. The bio-oil obtained from pyrolysis of a biomass consists of oxygenated organic molecules, such as esters, ethers, aldehydes, ketones, phenols, carboxylic acids, and alcohols that can be used to produce energy.

The bio-oil liquid produced from pyrolysis, and its upgrade to high-value chemicals used in industry, would significantly improve the economic feasibility of pyrolysis (Czernik and Bridgwater 2004; Bridgwater 2012). Syngas produced during pyrolysis is a mixture of H_2, CO, CO_2, CH_4, and volatile organic compounds (Crombie and Mašek 2015). The gases produced during pyrolysis can be used as a heat source for further pyrolysis or as dry biomass feedstock (Becidan *et al*. 2007).

Tamarix chinensis biomass has not been used to produce biochar or bioenergy using pyrolysis technology. *T. chinensis* is planted to control water and wind erosion in Inner Mongolia. *Tamarix chinensis* can tolerate drought and saline conditions, and it is widely distributed from northwest arid and semi-arid areas to sub-humid regions in northern China. Furthermore, it is used as a raw material in the paper industry. The large-scale development of forest-based bioenergy could result in social, economic, and ecological benefits.

The objectives of this study were to evaluate the physicochemical properties of *T. chinensis* biochar for use as a soil amendment and to perform a compositional analysis of the bio-oil and syngas from *T. chinensis* biomass for bioenergy purposes under different pyrolysis temperatures. *T. chinensis* biomass was collected from the Hetao region, Inner Mongolia, China. The feedstock biomass was subjected to 300, 500, and 700 °C temperatures, at a heating rate of 10 °C min^{-1} and a 2 h residence time using a fixed-bed biomass pyrolysis system. In this manner, the basic, surface, and adsorption properties of the biochars produced were characterized. The bio-oil and syngas byproducts were also analyzed for use as energy sources.

EXPERIMENTAL

Materials and Methods

Tamarix chinensis biomass and pyrolysis

The biomass, *T. chinensis*, was collected from the Hetao region, Inner Mongolia, China. Pyrolysis was conducted at 300, 500, and 700 °C for 2 h residence time in a fixed-bed pyrolysis system. As shown in Fig. 1, the system was composed of a fixed-bed pyrolyser, a condenser, a bio-oil trap, a coolant circulating pump, a heater controller, a data logger, and a carrier gas supply. The pyrolyser was composed of an electric heater and a stainless steel reactor, with an inner diameter of 80 mm and height of 1.3 m. The chopped biomass material was loaded into the pyrolyser and heated to the desired temperature, at a rate of 10 °C min^{-1}. At the end of pyrolysis, the biochar and bio-oil were weighed to calculate their yields. The weights of the biochar and bio-oil products were obtained, added together, and then subtracted from the original biomass weight to obtain the syngas product amount by difference. Syngas was collected in foil gas sampling bags. The composition of the collected gas samples was analyzed using an AutoSystem XL gas chromatograph (PerkinElmer, Waltham, MA, USA).

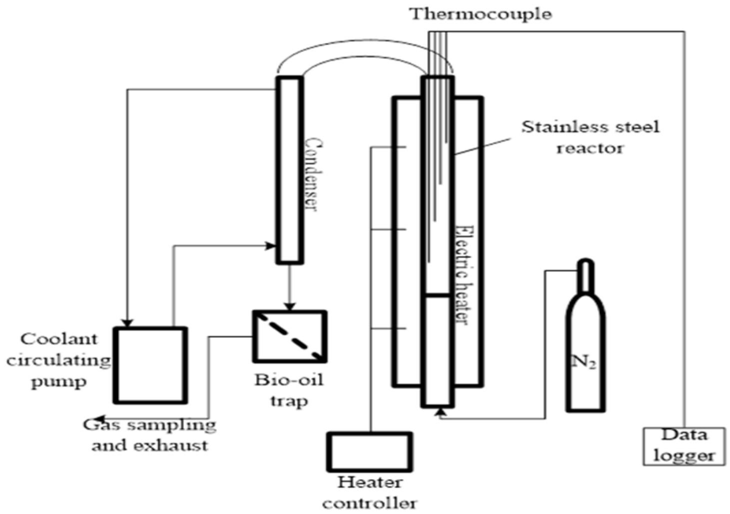

Fig. 1. Schematic diagram of a lab-scale pyrolysis reactor

Assessing biochar characteristics

Small quantities of raw material and biochar samples were oven-dried at 105 °C for 24 h and then ground to pass through a 0.25-mm mesh screen. The ash content was measured by heating at 500 °C for 4 h in a muffle furnace. The heating values were determined by bomb calorimetry. The carbon (C), hydrogen (H), and nitrogen (N) contents were determined with an elemental analyzer (EA3000, EuroVector, Via Tortona, Milan). Oxygen was calculated by the difference between the total and the sum of C, H, N and ash. The pH, electrical conductivity (EC), and the contents of water soluble elements, such as phosphorus (P), potassium (K), sodium (Na), magnesium (Mg), and calcium (Ca) were measured in a 1:10 (w/v) water suspension by mixing 10 g of sample with 100 mL of deionized water. The samples were shaken for 1 h on a reciprocal shaker and filtered through a 0.45-µm filter. The Na and K contents were measured by flame photometry

(FP640, Shanghai Electronic Analysis Instrument Limited Company, China), Ca and Mg were determined by inductively coupled plasma mass spectrometry (Model: 6300, Thermo Electronic Corporation, USA), and P was determined by spectrophotometry (UV2300, TECHCOMP, China).

Approximately 200 g of raw material and biochar was shaken for 30 min in 100 mL of 1 M HCl and allowed to stand overnight. After separation from acid, the samples were washed with distilled water until the EC value of the water suspension was < 20 μs/cm. Then the samples were oven-dried at 105 °C for 24 h. The total concentrations of surface acidic and basic functional groups in the samples were measured using the Boehm titration method (Boehm 2002). Surface area was measured by the mercury intrusion method (Pore Master GT-60, Quantachrome Instrument, USA). The method for cation exchange capacity (CEC) was adopted from Gaskin *et al.* (2008). Scanning electron microscopy (SEM; S-3400N, S-570 HITACHI, Japan) and Fourier-transform infrared (FT-IR) spectrophotometry analyses were carried out to study changes in the structure and the surface functional groups. Iodine adsorption was determined using a sodium thiosulfate volumetric procedure, according to the Chinese standard method GB/T 12496.8 (1999). Approximately 0.2 to 0.5 g of acid-washed biochar was mixed with 10 mL of HCl (1+9) and slowly heated for 30 s. Then, the samples were cooled to room temperature and shaken with 50 mL of a 0.1 M stock iodine solution for 15 min. The dilute filtrate was titrated with 0.1 M sodium thiosulfate until the solution was pale yellow. Two milliliters of 1.0% starch indicator solution was added and titrated until the solution was colorless. A blank titration was also performed without a biochar sample. The iodine value was calculated as follows:

$$A = 5(10C1 - 1.2C2V2) \times 127/M \tag{1}$$

where A is the iodine number (mg/g), $C1$ is the concentration of the iodine stock solution (M), $C2$ is the concentration of sodium thiosulfate (M), $V2$ is the titration reading (mL), and M is mass of the adsorbent (g).

Approximately 0.2 g of acid-washed biochar sample was used for the methylene blue adsorption analysis. The sample was shaken vigorously for 5 min with 25 mL of 0.1 mM methylene blue and allowed to stand for 24 h at room temperature. The absorption of the dilute filtrate was determined by a spectrophotometer (UV2300, TECHCOMP, China) at a wavelength of 660 nm. The adsorption capacity was calculated by the following equation,

$$qe = (Co - Cf) \times V/M \tag{2}$$

where qe is the adsorption by the material (mg/g), Co is the initial methylene blue concentration (mg/L), Cf is the final methylene blue concentration (mg/L), V is the volume taken (mL), and M is mass of the adsorbent (g).

Assessing the bio-oil characteristics

The bio-oil collected was maintained under a light-proof condition for several hours. The aqueous and non-aqueous fractions of the viscous bio-oil were separated using a separatory funnel. The carbon (C), hydrogen (H), nitrogen (N), and oxygen (O) contents in the non-aqueous fraction were determined with the EA3000 elemental analyzer, and the heating value was determined by bomb calorimetry. The water content of the bio-oil was determined using the MA-LA Automatic Fast Karl Fischer moisture meter (MA-1A, Shanghai BenAng Scientific Instrument)).

RESULTS AND DISCUSSION

Product Yields

The yields of biochar, bio-oil, and syngas, at the three pyrolysis temperatures, are presented in Fig. 2. Increasing the pyrolysis temperature resulted in a decrease in the biochar yield and increase in the syngas yield. The biochar yield decreased from 41% at 300 °C to 24% at 700 °C. The decline in biochar yield above 500 °C was only 3%, indicating the complete decomposition of hemicellulose and cellulose at 500 °C. A slight change in the bio-oil yield was observed, whereas increasing the pyrolysis temperature from 300 to 700 °C increased the syngas yield from 20% to 35%. The highest bio-oil yield was 41% at both 500 and 700 °C.

Hemicellulose and cellulose decompose easily during pyrolysis, whereas lignin is the most resistant part of the biomass to decomposition. Yang *et al*. (2004) reported that biochar originated from the lignin component of the biomass, whereas bio-oil came from cellulose component, at a pyrolysis temperature of 500 °C. The increase in syngas yield at 700 °C might be due to secondary decomposition of biochar and pyrolysis vapors at the high pyrolysis temperature (Ertaş and Alma 2010; Imam and Capareda 2012).

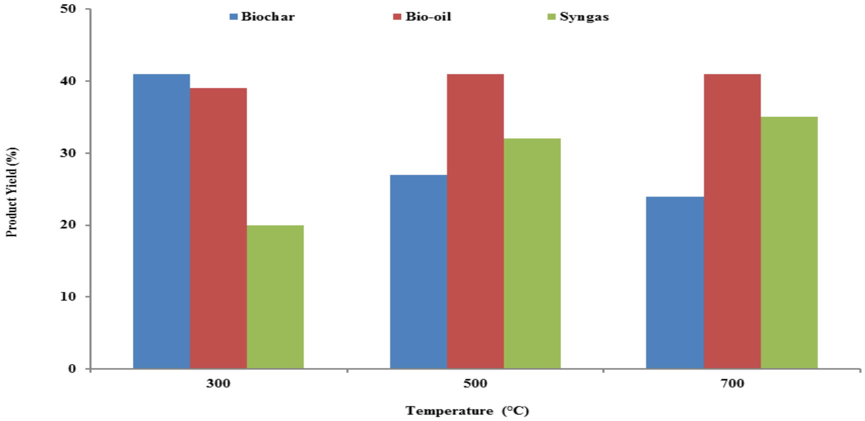

Fig. 2. The yields of biochar, bio-oil, and syngas produced from *Tamarix chinensis* biomass by a slow pyrolysis process in a solid-bed system at 300, 500, and 700 °C for 2 h

Basic Biochar Characteristics

Table 1 showed that the pH value of the raw feedstock was acidic (pH 5.12). However, the pH values of the biochar samples produced at 300 and 500 °C were 6.92 and 8.53, respectively. The pH increased abruptly to 11.98 in the biochar obtained at a pyrolysis temperature of 700 °C, which was associated with the enrichment of K and Na in the biochar. Such inorganic alkalis influence the alkalinity of biochar, and the effect becomes more important above 500 °C (Yuan *et al*. 2011; Fidel 2012). Some studies reported an alkaline pH of biochar at high pyrolysis temperature (Cantrell *et al*. 2012; Al-Wabel *et al*. 2013). This highly pyrolysed biochar can neutralize acidic soil and improve soil health. The EC values of the biochars produced at 300, 500, and 700 °C were 0.90, 1.10, and 1.27 (dS/m), respectively. Slight changes occurred in the EC values of biochar as the pyrolysis temperature increased. These changes were within the acceptable range, as applying biochar to soil may not contribute significantly to soil salinity.

The feedstock had the lowest ash content (3.45%); however, this value increased slightly as the pyrolysis temperature increased. The highest ash content (8.71%) was obtained in biochar pyrolysed at 700 °C. The ash content of feedstocks varies; woody feedstock has a lower ash content compared to straw, grass, or grain husks (Raveendran *et al*. 1995). In general, lower ash content leads to higher carbon content in the biochar.

The heating values of the biochar samples, obtained at the pyrolysis temperatures of 300, 500, and 700 °C, were 26.70, 28.07, and 27.90 (MJ/kg), respectively (Table 1). The heating value is comparable to the heating value of switchgrass derived biochar reported by (Sadaka *et al*. 2014).

The elemental contents in the feedstock and biochar samples are presented in Table 2. The total carbon content of the feedstock was 47.92%, whereas the total carbon content of the biochars increased from 68.92% to 78.50% and 82.79% as the pyrolysis temperature was increased from 300 °C to 500 and 700 °C, respectively. Increasing the pyrolysis temperature noticeably enhanced the carbon content, and the biochar became highly carbonaceous at 700 °C. This increasing carbon trend agrees with results reported by Chen *et al*. (2015), and is consistent with biochar ash content because a low ash content can lead to the formation of highly aromatic carbon during pyrolysis. Biochar usually contains a high carbon content when produced from a woody biomass, as it is rich in carbon compounds that are not easily decomposed, even at high pyrolysis temperatures. The nitrogen content of the biochar decreased at the highest pyrolysis temperature of 700 °C because of volatilization of nitrogen when the temperature increased from 300 °C to 700 °C. The atomic ratios of O/C, H/C, and (O+N)/C decreased in the biochars obtained at increasing pyrolysis temperatures relative to those in the biomass feedstock, indicating increasing aromaticity with temperature. Furthermore, water soluble K and Na contents also increased, whereas P, Ca, and Mg contents decreased as the pyrolysis temperature was increased (Table 2). The K and Na contents increased more than two-fold in the biochar pyrolysed at 700 °C, compared to that in the feedstock.

Table 1. Properties of the Feedstock and the Biochars Produced from *Tamarix chinensis* by Pyrolysis at 300, 500, and 700 °C for 2 h

Biochars	pH	EC (dS/m)	Heating value (MJ/kg)	Ash (%)	Surface area (m^2/g)	Pore volume (cm^3/g)	CEC (cmol/kg)
Feedstock	5.12	0.99	*	3.45	1.50	1.18	6.70
B300	6.92	0.90	26.70	5.36	1.89	0.93	5.38
B500	8.53	1.10	28.07	7.46	5.25	0.94	2.94
B700	11.98	1.27	27.90	8.71	4.92	1.06	3.53

*Not determined

Table 2. Elemental Concentrations of the Feedstock and the Biochars Produced from *Tamarix Chinensis* by Pyrolysis at 300, 500, and 700 °C for 2 h

Biochars	C	H	N	O	O/C	H/C	(O+N)/H	P	K	Ca	Mg	Na
	%									g/kg		
Feedstock	47.92	6.28	0.95	44.85	0.936	0.131	0.956	0.39	3.15	1.12	5.78	0.64
B300	68.92	4.72	1.33	25.02	0.363	0.069	0.382	0.19	2.95	1.14	4.19	0.83
B500	78.50	2.85	1.38	17.26	0.220	0.036	0.238	0.01	5.42	0.81	2.67	1.03
B700	82.79	1.19	1.20	14.83	0.179	0.014	0.194	0.01	6.76	0.05	0.07	1.44

Biochar Surface Characteristics

The surface area and pore volume data are presented in Table 1. Surface area increased from 1.89 (m^2/g) in biochar pyrolysed at 300 °C to 5.25 (m^2/g) at 500 °C. Increasing the pyrolysis temperature to 700 °C noticeably decreased the surface area of the biochar. The pore volume increased slightly as the pyrolysis temperature increased. The pore volume values of the biochar samples obtained at 300, 500, and 700 °C were 0.93, 0.94, and 1.06 (cm^3/g), respectively. It has been found that the pyrolysis temperature strongly affects pore development (Fu *et al*. 2012). Although the surface area of the biochar was generally low, it can enhance nutrients and water retention in the soil-biochar environment.

Generally, the biochars produced had low CEC values and showed no consistent increase with increasing pyrolysis temperatures. The CEC (6.70 cmol/kg) was highest in the raw feedstock. Among the biochar samples, the lowest (2.94 cmol/kg) CEC value was recorded at 500 °C and the highest (5.38 cmol/kg) was obtained for the biochar produced at 300 °C. A lower CEC value in biochar pyrolysed at 500 °C compared with that produced at 400 °C has been previously reported in the literature (Gaskin *et al*. 2008).

The surface acidic and basic functional groups in the raw feedstock and biochar samples are shown in Fig. 3. The concentration of surface acidic functional groups on the raw feedstock was 0.89 (mmol/g). This concentration decreased sharply in the biochar samples from 1.18 to 0.46 (mmol/g) as the pyrolysis temperature increased from 300 to 700 °C.

The carboxylic acidity of the raw feedstock was 0.64 (mmol/g). This concentration decreased in the biochar samples from 0.68 to 0.27 (mmol/g) as pyrolysis temperature was increased from 300 to 700 °C. In contrast, the concentration of surface basic functional groups in the raw feedstock was 0.61 (mmol/g) and increased steadily in the biochar samples as pyrolysis temperature increased. The concentrations of surface basic functional groups in the biochar samples obtained at 300, 500, and 700 °C were 0.55, 0.63, and 0.84 (mmol/g), respectively. These results are in line with the increase in pH as the pyrolysis temperature was increased and coincides with data reported previously (Al-Wabel *et al*. 2013).

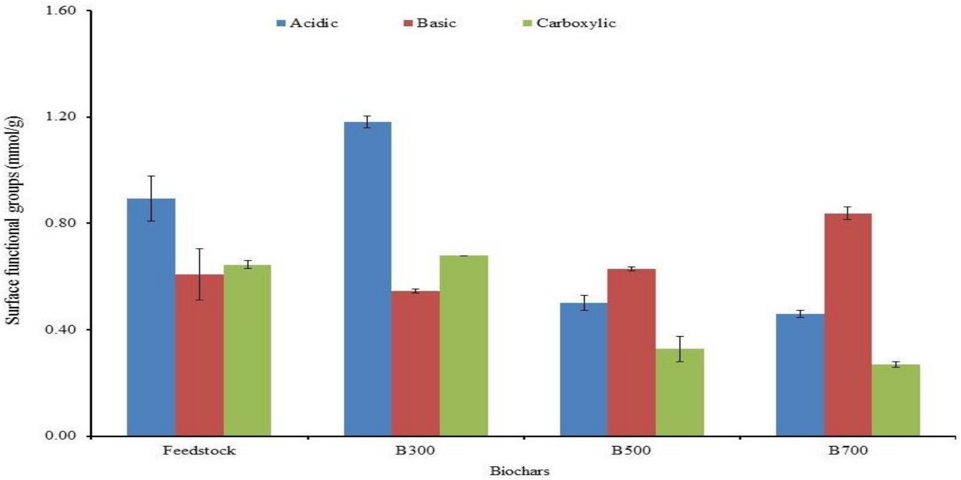

Fig. 3. Surface functional groups of the feedstock and the biochars produced from *T. chinensis* biomass by a slow pyrolysis process in a solid-bed system at 300, 500, and 700 °C for 2 h

Adsorption Capacity

The pyrolysis temperature did not affect either the iodine or the methylene blue adsorption capacities (Fig. 4). A similar iodine adsorption capacity (256 mg/g) was observed for the biochar samples produced at 300 °C and 500 °C, whereas the biochar obtained at 700 °C had a lower (76 mg/g) iodine adsorption capacity. The methylene blue adsorption capacity values were 1.78, 2.08, and 1.96 (mg/g) for the biochars produced at 300, 500, and 700 °C, respectively. No clear associations were observed between iodine or methylene blue adsorption capacities and the CEC and/or surface area. Although the biochar iodine and methylene blue adsorption capacities were poorly correlated with their surface properties, biochar could be effectively used to remove organic pollutants from aqueous solutions. Many studies have reported that adsorption is a complex process determined by surface area, as well as the surface chemistry of the material (Guo *et al*. 2003; Chen and Yuan 2011).

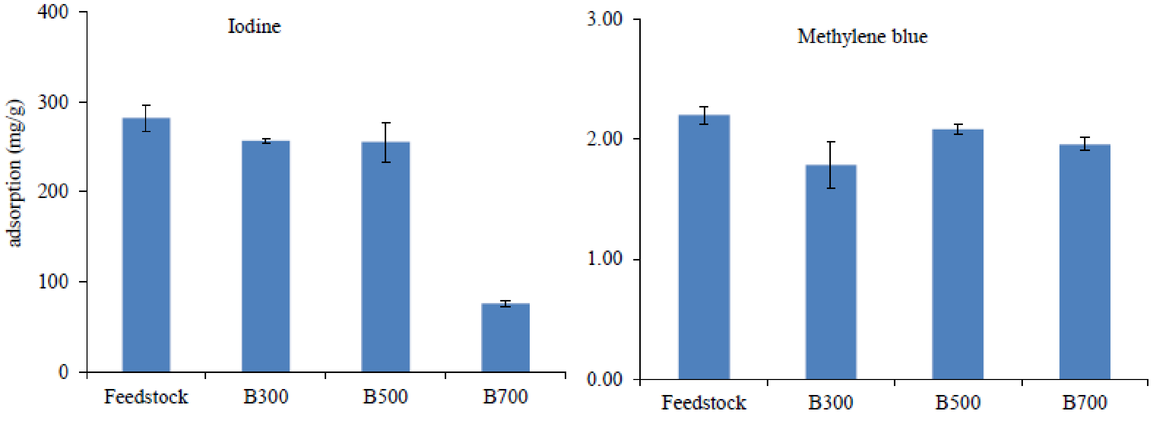

Fig. 4. Adsorption capacities of (a) iodine and (b) methylene blue of the biochar produced from *T. chinensis* by a slow pyrolysis process in a solid-bed system at 300, 500, and 700 °C for 2 h

Bio-Oil Characteristics

The carbon content was 65.87, 62.09, and 61.65% for bio-oil samples obtained at 300, 500, and 700 °C, respectively (Table 3). The oxygen content increased from 24.77% to 29.04% when the pyrolysis temperature increased from 300 to 500 °C and remained constant at 700 °C. Slight changes occurred in the N and H contents as the pyrolysis temperature increased. The heating value of the bio-oil samples obtained at 300, 500, and 700 °C were 30.43, 27.51, and 26.03 (MJ /kg), respectively, indicating its potential use as a biofuel.

Table 3. Properties of Bio-Oil Produced from *Tamarix Chinensis* by a Slow Pyrolysis Process in a Solid-Bed System at 300, 500, and 700 °C for 2 h

Temperature (°C)	Heating value (MJ/kg)	Water content (%)	Carbon (%)	Oxygen (%)	Nitrogen (%)	Hydrogen (%)
300	30.43	22.65	65.87	24.77	1.23	8.13
500	27.51	18.76	62.09	29.04	1.48	7.40
700	26.03	13.22	61.65	29.87	1.90	6.58

The water content in the bio-oil decreased from 22.65% to 13.22% when the pyrolysis temperature increased from 300 °C to 700 °C. The elemental composition and higher heating value data of the bio-oil at the three different pyrolysis temperatures agreed with those reported by Kim *et al.* (2013). The higher heating value could be attributed to high C content, indicating potential utilization of *T. chinensis* biomass as feedstock to produce fuel energy.

Syngas Composition

The composition of the syngas produced under the three pyrolysis temperatures is presented in Table 4. The carbon monoxide (CO) concentration increased from 29.31% at 300 °C and to a maximum of 61.74% at 500 °C, whereas, the carbon dioxide (CO_2) concentration showed the reverse trend. The CO_2 concentration decreased from 56.60% at 300 °C and to a minimum of 19.51% at 500 °C. This opposite trend with temperature occurred because CO_2 is released by cracking and reforming of carboxyl groups at low temperatures, whereas, CO is released by a secondary reaction at high temperatures (Luo *et al*. 2004; Xu *et al*. 2009; Fu *et al*. 2011). The concentration of hydrocarbon gases, such as ethane, ethylene, propane, and acetylene, increased as the pyrolysis temperature increased, except in the case of methane, which decreased rapidly from 9.28% to 1.45% when temperature was increased from 300 to 700 °C. The H_2 concentration increased markedly to a maximum of 15.78% at 500 °C, indicating that increasing the pyrolysis temperature was favorable for H_2 production. Thermal cracking of heavy hydrocarbons and aromatic condensation could contribute to a release of H_2 at high pyrolysis temperatures. Thermal decomposition of lignin at high pyrolysis temperatures is known to cause the release of H_2, CH_4, and C_2 hydrocarbons (Yang *et al*. 2006; Becidan *et al*. 2007). The thermal breakdown of biomass constituents, at temperatures up to 500 °C, mainly affects gas composition; whereas, secondary cracking is the dominant factor responsible for variations in the gas composition above 500 °C (Yang *et al*. 2006; Becidan *et al*. 2007; Chen *et al*. 2012).

Table 4. Syngas Volume Fractions Produced from *Tamarix chinensis* by Pyrolysis at 300, 500, and 700 °C for 2 h

Temperature (°C)	CH_4 (%)	C_2H_6 (%)	C_2H_4 (%)	C_3H_8 (%)	C_2H_2 (%)	H_2 (%)	CO (%)	CO_2 (%)
300	9.28	0.94	0.70	0.42	0.16	2.60	29.31	56.60
500	1.92	0.48	0.32	0.17	0.07	15.78	61.74	19.51
700	1.45	6.57	8.95	4.45	1.57	9.90	38.18	28.93

CONCLUSIONS

1. To summarize, *Tamarix chinensis* biomass was pyrolyzed to co-produce biochar, bio-oil, and syngas at temperatures of 300, 500, and 700 °C. The product yields were measured, and the characteristics of the different biochars were analyzed.

2. The biochars produced at high pyrolysis temperatures had high pH values and ash and C contents, which is useful for ameliorating soil acidity, improving soil fertility, and sequestering C.
3. Overall, the biochar produced at 500 °C may have a greater potential to adsorb contaminants.
4. The energy content in the bio-oil and syngas produced was satisfactory for use as bioenergy sources.
5. These preliminary results show that *T. chinensis* biomass is useful for producing biochar and bioenergy.

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