# Fabrication of Activated Carbon Using Two-Step Co-Pyrolysis of Used Rubber and Larch Sawdust

Jing Geng,<sup>a</sup> Lu-Fei Li,<sup>a</sup> Wen-Liang Wang,<sup>c</sup> Jian-Min Chang,<sup>a,\*</sup> Chang-Lei Xia,<sup>b</sup> Li-Ping Cai,<sup>b</sup> and Sheldon Q. Shi<sup>b</sup>

Characteristics of the char produced in the co-pyrolysis of used rubber and larch sawdust were studied in the conversion of low-valued pyrolysis char into value-added activated carbon using two-step co-pyrolysis, namely pyrolysis and activation processes. The physicochemical characteristics of the chars were examined by X-ray diffraction (XRD), Brunauer–Emmett–Teller (BET), and scanning electron microscopy (SEM). The results revealed that after the two-step co-pyrolysis, the upgraded carbon had BET surface areas ranging from 600 m<sup>2</sup> g<sup>-1</sup> to 900 m<sup>2</sup> g<sup>-1</sup>, which were higher than the requirements for activated carbon (American Water Works Association B600 standard). Additionally, as the sawdust/rubber ratio increased, the BET value increased accordingly. A possible reaction mechanism is proposed based on the experimental results during the activation process.

Keywords: Used rubber; Larch sawdust; Co-pyrolysis; Activated carbon; BET surface value

Contact information: a: College of Material Science and Technology, Beijing Forestry University, Beijing 100083, China; b: Mechanical and Energy Engineering Department, University of North Texas, TX 72076, USA; c: College of Bioresources Chemical and Materials Engineering, Shaanxi University of Science & Technology, Xi'an 710021, China; \*Corresponding author: cjianmin168@126.com

#### INTRODUCTION

Used rubber/tires and waste biomass are important fossil-based wastes and biobased wastes, respectively (Ucar and Karagoz 2014). Approximately 1.5 billion tires are produced and over 800 million tires are scrapped worldwide annually (van Beukering and Janssen 2001; Williams 2013). The total amount of above-ground biomass in forests is estimated to be about 420 billion tons, and yearly global forest residues are increasing gradually (Parikka 2004). Hence, the effective disposal of these wastes has recently attracted a great deal of interest. The utilization of waste rubber and biomass through copyrolysis is a cost-effective technology for addressing this issue.

Co-pyrolysis is a promising solution for converting used rubber into value-added products by adding waste sawdust (Laird *et al.* 2009). There are numerous studies on the improvement of pyrolysis oil obtained from co-pyrolysis of wastes such as biomass, used tires, waste plastics, coal, and sewage sludge (Dorado *et al.* 2014; Martinez *et al.* 2014; Zhang *et al.* 2015). Low-valued char, the main pyrolysis product, has the potential to be converted to value-added activated carbon (Shen 2015). The American Water Works Association (AWWA) B600 standard requires that activated carbon has a specific surface area (SSA) in excess of 500 m<sup>2</sup> g<sup>-1</sup> as determined by N<sub>2</sub> adsorption at 77 K (Xia *et al.* 2016a,b,c,d).

Activation processes are mainly categorized as physical activation processes, chemical activation process, and physical-chemical activation process. Carrott *et al.* (2008) obtained activated carbon from kraft and hydrolytic lignin during physical activation process with CO<sub>2</sub>, obtaining the product with properties as good as those of materials obtained from other bio-resources or synthetic polymers. The biomass flax fiber was processed *via* chemical activation process using zinc chloride to produce mainly microporous activated carbons (up to 2400 m<sup>2</sup> g<sup>-1</sup>) (Williams and Reed 2006). Shim *et al.* (2016) prepared the porous activated carbons (ACs) from corn grains through physical (steam) and chemical–physical (H<sub>3</sub>PO<sub>4</sub>-steam) activations successfully.

Pyrolysis char has been used to prepare activated carbon. The BET surface areas of poplar wood char from gasification in a fluidized bed were examined by Klinghoffer *et al.* (2012). When the treatments were in gasification atmospheres of 90% H<sub>2</sub>O/10% N<sub>2</sub> at 750 °C for 60 min and at 920 °C for 30 min, 435 m<sup>2</sup> g<sup>-1</sup> and 687 m<sup>2</sup> g<sup>-1</sup> BET surface areas were achieved, respectively (Klinghoffer *et al.* 2012). Carriera *et al.* (2012) used vacuum pyrolysis to convert sugar cane bagasse into char materials for wastewater treatment by preparing an adsorbent for methylene blue adsorption with a BET surface area of 418 m<sup>2</sup> g<sup>-1</sup>. Bernardo *et al.* (2012b) also investigated the co-pyrolysis of plastic wastes, pine biomass, and used tires, producing activated carbon with an excellent surface area that could be used as a precursor for adsorbents.

This work explored the characteristics of the char produced in the one-step copyrolysis of rubber/sawdust and the conversion of low valued rubber/sawdust into valueadded activated carbon using two-step co-pyrolysis, namely pyrolysis and activation processes. This study helps to elucidate the composition of the co-pyrolysis char and the conversion mechanism of the co-pyrolysis char to activated carbon.

#### EXPERIMENTAL

#### Materials

The rubber particles with an average size of 80 to 90 screen mesh were purchased from Ketai Rubber Scrap Mill, Tianjin, China. The larch wood was sourced from Small Xing-An Mountain, China, and it was dried and smashed into particles. Using a screen, particles with an average size of 80 to 100 screen mesh were collected for the experiment. The compositions of the rubber and sawdust were determined (Wang *et al.* 2014) and are presented in Table 1. The lignocellulosic compositions of larch sawdust were analyzed following the previously reported standard procedures (Ren *et al.* 2013), and results are summarized in Table 2.

Matorial	Ultimate Analysis (wt.%)			Proximate Analysis (wt.%) <sup>b</sup>					
Material	С	Н	O a	Ν	S	М	VM	FC	А
Rubber	84.10	6.69	7.21	0.39	1.61	4.70	54.53	26.28	14.49
Sawdust	53.40	4.64	41.85	0.11	0.00	10.94	71.59	16.68	0.79
<sup>a</sup> by difference; <sup>b</sup> air-dried basis.									

Motorial	Lignocellulosic Analysis (wt.%)			
Material	Cellulose	Hemicellulose	Lignin	Extractives
Sawdust	42.26	24.40	26.63	6.71

#### **Pyrolysis Process**

A laboratory-made 1 kg h<sup>-1</sup> stainless pyrolysis reactor (Fig. 1) with adjustable reaction temperature, heating rate, retaining time, and catalyst type was used for pyrolysis. Under a nitrogen atmosphere, the rubber particles mixed with sawdust were placed in a reactor that was pre-heated to 450 °C for 1.2 s. The initiation of primary pyrolysis reactions at this temperature released volatiles and formed char. The char was collected by a tourbillion separator, and the hot volatiles were condensed into pyrolysis oil and non-condensable gas.

The rubber and the sawdust were mixed with different blending ratios (w/w) of 1:2, 1:1, and 2:1 and then pyrolyzed in the reactor (signed as 1:2, 1:1, and 2:1). To explore the mechanism, the rubber and the sawdust were pyrolyzed separately to obtain the rubber char and the sawdust char as control groups.



Fig. 1. The 1 kg h<sup>-1</sup> stainless pyrolysis reactor

The chars obtained in the pyrolysis experiments were carbonized particulate residues impregnated with the pyrolysis oil. Using the Soxhlet method, a solvent extraction (CH<sub>2</sub>Cl<sub>2</sub>) was performed for 6 h. In order to decrease the ash content of the chars, a demineralization procedure was conducted with 1 M HCl for 1 h. A KOH activation procedure (the second co-pyrolysis process) was carried out to increase the surface area of the chars. The chars were impregnated with the activator of KOH with a ratio of 1:1 and then activated with N<sub>2</sub> at 100 mL min<sup>-1</sup>. The activation temperature was set as follows: the temperature was increased from room temperature to 500 °C at 10 °C min<sup>-1</sup>, maintained for 20 min, increased to 800 °C with the same heating rate, maintained for 20 min, and then

reduced to room temperature. The activated chars were washed with 0.1 M HCl and deionized water and oven-dried to a constant weight. The ash content of chars was detected in accordance with ASTM D1762-84 (2013).

#### Characterization

The X-ray diffraction (XRD) patterns of the original samples and chars were examined by the Shimadzu XRD–6000 (Shimadzu Co., Ltd., Kyoto, Japan) and collected from 5° to 45° using Cu K $\alpha$  radiation (60 kV, 80 mA) at a scanning rate of 2 ° min<sup>-1</sup>. The specific surface areas of the chars were measured in a Micrometrics TriStar II 3020 (Micrometrics Co., Ltd., Norcross, GA, USA) *via* nitrogen adsorption. Each sample was outgassed for 3 h at 77 K, and the surface area was calculated by the Brunauer–Emmett–Teller (BET) method. The mesoporous size distributions were calculated using the BJH (Barrett-Joyner-Halenda) method (Jacques *et al.* 2007) and the microporous size distributions were calculated using a Hitachi S-3400N scanning electron microscope (SEM; Hitachi Co., Ltd., Tokyo, Japan).

### **RESULTS AND DISCUSSION**

#### Yield of Pyrolysis Char

As shown in Fig. 2, the char yield of solo rubber pyrolysis was more than twice the amount of the solo sawdust because of the higher content of non-volatile materials such as carbon black and ash in rubber, as shown in Table 1. As the proportion of sawdust rose, the yield of co-pyrolysis char dropped. In a previous work (Wang *et al.* 2014), it was also observed that the proportion of rubber was negatively associated with the yield of pyrolysis oil from co-pyrolysis of rubber and sawdust. Adding more rubber inhibited the formation of condensable volatiles and promoted the production of solid product during the co-pyrolysis process.



**Fig. 2.** Yield of char from the co-pyrolysis of rubber and sawdust. 1:2, 1:1, and 2:1 represent the char obtained from the co-pyrolysis of rubber and sawdust with different blending ratios (w/w)

#### Yield of Activated Char after Upgrading Treatments

The preparation of activated carbon with pyrolysis char could increase the added value of pyrolysis char. The cooling process of pyrolysis char in the presence of pyrolysis volatiles would adsorb PAHs and other aromatic molecules (Bernardo *et al.* 2010; Bernardo *et al.* 2012a). Consequently, the decrease of PAHs in the co-pyrolysis oil of rubber and sawdust observed in our previous work was not only related to the oxygen-free radical reaction during the co-pyrolysis process (Wang *et al.* 2014), but also affected by the adsorption of co-pyrolysis char.

Prior to the KOH activation process, the pyrolysis chars were subjected to extraction and demineralization. Approximately 10% residual oil, which was absorbed by the solid char during the pyrolysis process, was extracted by CH<sub>2</sub>Cl<sub>2</sub> (Fig. 3). The extraction yield and the demineralization yield of the solo pyrolysis rubber char were all higher than those of the solo pyrolysis sawdust char. As more sawdust was added into the rubber, the char yield after procedures of extraction and demineralization notably decreased and then rose. The lowest values were 89.1% (1:1) and 50.9% (2:1). It could be inferred that the co-pyrolysis char could adsorb more ash and residue oil, which had very stable aromatic rings in sawdust and a long-chain of C–C bonds in the copolymer of rubber and recombination of thermal cracking products during the co-pyrolysis process (Laird *et al.* 2009).



Fig. 3. Yield of activated char after procedures of extraction, demineralization, and activation

After the activation process (the second co-pyrolysis process), the activated char prepared from solo pyrolysis sawdust char presented a high yield after a series of treatments. The co-pyrolysis char obtained from adding rubber into sawdust was unfavorable for obtaining a high activated char yield and the activated char yield of rubber char was as low as 19.5%.

Char	Ash (wt.%)				
Sample	Before Demineralization	After Demineralization	After Activation		
Rubber	28.70	13.19	16.18		
2:1	22.68	7.72	10.36		
1:1	23.68	8.03	10.02		
1:2	18.16	5.30	7.38		
Sawdust	10.02	4.14	5.96		

Table 3. Ash Content of Char before and	after Demineralization and Activation
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Figure 3 shows the reduction in char yield after the demineralization, indicating that much more ash was extracted from carbon due to the demineralization treatment. To understand the variation of ash in char, the ash content of pyrolysis char is presented in Table 3. The ash content of the char samples depended substantially on the type of feedstock. The rubber char had high ash content, but the char derived from sawdust showed lower ash content. Through different treatments, the ash content of co-pyrolysis char was between rubber char and sawdust char. After HCl demineralization and KOH activation, the ash content in the chars decreased remarkably compared with that of the chars before demineralization. The ash contents of the activated chars were slightly higher than those of the demineralized char, which may have been related to the activation method and the low activated char yield.

Sample	Total pore volume (m³/g)	Mesopore volume (m³/g)	Micropore volume (m <sup>3</sup> /g)
Rubber	0.2032	0.1960	0.0069
2:1	0.2170	0.2113	0.0039
1:1	0.1762	0.1684	0.0132
1:2	0.3481	0.3439	0.0033
Sawdust	0.0873	0.0807	0.0066

**Table 4.** Pore Structural Characteristics of Untreated Pyrolysis Char

Table 5. Pore Structura	I Characteristics	of Activated Char
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Sample	Total pore volume (m <sup>3</sup> /g)	Mesopore volume (m <sup>3</sup> /g)	Micropore volume (m <sup>3</sup> /g)
Rubber	0.5051	0.4943	0.0113
2:1	0.6387	0.6285	0.0089
1:1	0.4122	0.4092	0.0021
1:2	0.6729	0.6654	0.0087
Sawdust	0.5250	0.4098	0.1128

#### **Textural Characteristic Analysis**

There were mainly mesopores in untreated pyrolysis char and activated char, as presented in Tables 4 and 5. After the activation process (the second co-pyrolysis process),

the total pore volume and the mesopore volume increased noticeably. When the blending ratio of rubber/sawdust was 1:2, the total pore volume and the mesopore volume of the untreated char and the activated char were the highest, as shown in Table 5.



Fig. 4. BET surface area of char from co-pyrolysis of rubber and sawdust

The BET surface area of a given char plays an important role in influencing its adsorption performance (Guerrero et al. 2008). As shown in Fig. 4, all untreated pyrolysis chars showed low BET surface area values except for the sawdust char. Even after KOH activation, the surface area value of rubber char was too low to be used as activated carbon because the high ash content and carbon black in rubber may have blocked the incipient porosity (Raveendran and Ganesh 1998; Helleur et al. 2001; Bernardo et al. 2012b). When the proportion of sawdust in the mixture was increased, the BET surface area of the copyrolysis char also increased. After the activation process (the second co-pyrolysis process), the surface area of the co-pyrolysis char reached 896.4  $m^2 g^{-1}$  when the blending ratio of rubber/sawdust was 1:2, which was much higher than the surface areas of the sawdust char (523.1 m<sup>2</sup> g<sup>-1</sup>) and the rubber char (92.8 m<sup>2</sup> g<sup>-1</sup>). It can be inferred that the copyrolysis char had more extraction yield and demineralization yield, resulting in it having more pores after extraction and demineralization, which promoted the activation reaction to obtain much cleaner activated char with higher BET values. The obtained BET surface areas were higher than the BET values (from  $429 \text{ m}^2 \text{ g}^{-1}$  to  $687 \text{ m}^2 \text{ g}^{-1}$ ) obtained from poplar wood by Klinghoffer et al. (2012).

#### **Mechanism of Activated Char Formation**

As shown in Fig. 5, some rubber char in the untreated co-pyrolysis char was attached to the surface of the sawdust char and entered into the pits of the sawdust char (Fig. 5, 2:1-A). After the activation process (the second co-pyrolysis process), the co-pyrolysis chars showed that the rubber char mixed well with the sawdust char and obtained higher specific surface area values by forming a more expanded porous structure.

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**Fig. 5.** SEM images of the char from co-pyrolysis of rubber and sawdust. A: untreated char; B: activated char

As shown in Fig. 6a, the untreated rubber char presented high crystallinity in the CaCO<sub>3</sub> phase. This likely occurred because the functional fillers improved the mechanical and biodegradable properties of rubber (Jin and Park 2008). The caking char was observed in Fig. 5 (Rubber-A). The diffuse X-ray peaks in the range of 10° to 30° demonstrated that the amorphous carbon structure was initially formed in the untreated char of sawdust during the graphite process of sawdust pyrolysis (Lin *et al.* 2011). After demineralization, most of the CaCO<sub>3</sub> in rubber was transformed into CaCl<sub>2</sub> as observed in Fig. 6b. This occurred corresponding to the decrease in ash content as demonstrated in Table 3. Fig. 6c shows that CaCO<sub>3</sub> was almost eliminated and a diffraction peak of K<sub>2</sub>O phase (at 32.8°) was observed

after activation. It was illustrated that a chemical reaction occurred at a high temperature between the KOH activator and the carbons in char, which generated a large quantity of small molecule gases such as CO and H<sub>2</sub>, resulting in the promotion of pore structure formation (Lillo-Rodenas *et al.* 2003). This was consistent with the many micro-pores in the co-pyrolysis chars observed in Fig. 5 (1:1-B).



**Fig. 6.** X-ray diffraction of the char from co-pyrolysis of rubber and sawdust. (a) Untreated char; (b) Demineralized char; (c) Activated char

## CONCLUSIONS

- 1. Low value rubber and sawdust were converted into value-added activated carbon through two-step co-pyrolysis (pyrolysis and activation).
- 2. The co-pyrolysis char produced by adding rubber into sawdust obtained low activated char yield but higher BET surface area values.
- 3. Upgrading procedures efficiently extracted the residue oil and reduced ash content to obtain much cleaner carbon.
- 4. During the activation process, a chemical reaction between the KOH activator and the carbons in char occurred at a high temperature, which generated small molecule gases such as CO and H<sub>2</sub>, resulting in the development of better porous structure and higher BET surface area values.

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