



Tuning the physicochemical properties of biochar derived from Ashe juniper by vacuum pressure and temperature



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ARTICLE INFO

Keywords:

Vacuum pyrolysis
Biochar
Aromaticity
Elemental analysis
Surface area

ABSTRACT

Ashe juniper is a problematic plant in central Texas due to rapid expansion causing the alteration of rangeland plant communities and hydrological landscapes. The thermochemical conversion of Ashe juniper into biochar with specific properties is a promising option for valorization of Ashe juniper. This study presents the effects of temperature (350, 450 and 520 °C) and vacuum pressure (0.09, 0.7 and 3 kPa) on physical and chemical properties of biochar produced by vacuum pyrolysis from Ashe juniper. We found that temperature had a major effect on the degree of carbonization and surface functionality, as well as the O/C and H/C ratios regardless of vacuum pressure. It was determined that the effect of vacuum pressure severity on product properties depends on pyrolysis temperature. In addition, the vacuum pressure was a crucial factor for controlling the surface area and porosity at each constant pyrolysis temperature. This work also demonstrated that the biochar from Ashe juniper under vacuum pyrolysis showed improved aromaticity and surface area compared to the biochar derived from other biomass. Biochar produced at 450 and 520 °C showed potential application as an adsorbent, a catalyst support and a carbon sequestration agent. Methylene blue (MB) adsorption test confirmed that biochar produced at 450 °C and 0.7 kPa showed potential as an adsorbent.

1. Introduction

Biochar, a carbon-rich and porous solid material, has recently attracted increasing attention as a carbon sequestration agent with the ability to mitigate greenhouse gas emission [1]. It can also be used as a soil amendment capable of improving soil health and demonstrated potential as a contaminant adsorbent, carbon catalyst and catalyst support [1,2]. The biochar's diverse potential is attributed to its physicochemical properties-high surface area, porosity, surface functionality and the degree of carbonization [2]. Therefore, the development of the method to tune the desired properties of biochar would be helpful for use of biochar in various applications.

Biochar is normally produced through the slow pyrolysis of biomass. Peak temperature and pressure have critical effects on the yields, physical and chemical properties of biochar [2,3]. Increasing peak temperature tends to lower biochar yields [2–4] while increasing the fixed-carbon content [5–7]. The peak temperature also affects two important textural properties (surface area and porosity) with surface area increasing as peak temperature increases [2,3,5]. The elemental composition also depends on the peak temperature [2,8]. Further investigation of peak temperature revealed that increasing peak

temperature lowered the H/C and O/C ratios, which are related to the aromaticity of biochar [7,9,10]. The degree of aromaticity may be indirectly proportional to the degree of chemical recalcitrance in soils [6,11]. In addition, O/C and (O + N)/C are parameters related to polarity and surface functional groups on biochar [12]. Higher O/C and (O + N)/C indicates more oxygen-contained functional groups in biochar. The surface functionality acts as an active site for nutrient retention of biochar in soil and functionalization of biochar [2].

As pressure increases, biochar yield increases most likely due to an increased vapor residence time in the biomass particle which leads to a secondary charring reaction [6]. The contents of fixed carbon in biochar also increase with higher pyrolysis pressure [6,13]. Pressure conditions also influence the textural properties of the biochar produced. Increasing pressure led to a decrease of BET surface area of biochar due to a clogging of the pores by tar deposits [3]. High pressure thus inhibits the production of biochar with high surface area and good porosity. Well-developed biochar characteristics are required for the application of biochar as soil amendments, adsorbents or catalyst support applications.

To improve textural properties of biochar, the secondary charring reactions, which are the condensation of volatiles and solid-vapor phase

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reactions, need to be minimized. The secondary reaction can be prevented by quickly removing volatiles from biomass. Vacuum pyrolysis can be one of the best options to minimize the secondary reaction due to its special advantage of minimum vapor residence time during pyrolysis [11]. Several studies have shown that activated carbon with a high surface area and porosity can be developed under vacuum pressure [14]. Not only the textural properties but also the aforementioned chemical properties should be controlled for the specific application of biochar. The chemical properties can be changed by varying the pyrolysis temperature. However, the effect of vacuum pressure on the aforesaid chemical properties of biochar is rarely found in the literature [15]. Furthermore, synergistic effects of vacuum pressure and temperature on the textural and chemical properties of biochar are rarely investigated [15].

Here, Ashe juniper was selected as a biomass waste model for the investigation. Ashe juniper, a native species covering over 8 million acres of Texas rangelands [16], has rapidly spread across Texas altering the production, composition, and structure of rangeland plant communities [17]. The intrusion of Ashe juniper on Texas rangeland may influence the hydrological landscape of government-protected lands [17]. For example, it has been reported that approximately 40 to 43% of rainfall is intercepted by Ashe juniper [16]. Current best management practices for removing Ashe juniper over widespread areas include the use of hydraulic shears or a bulldozer, followed by burning [18], none of which are not economically feasible. One method of improving the economic value of Ashe juniper management is to use Ashe juniper to extract essential oils, which are commercially valuable in the flavor and fragrance industries [19]. Another method could be producing fences from Ashe Juniper [19]. However, a large quantity of Ashe juniper waste might be left even after both processes. Also, Ashe juniper might be a promising resource to produce high-quality carbonaceous materials due to relatively high carbon contents determined in the current study. Therefore, the conversion of Ashe juniper waste into biochar through pyrolysis will be one valorization method.

To the best of our knowledge, there is no report focused on vacuum pyrolysis of Ashe juniper as a feedstock for biochar production. The main objective of this project is to evaluate the effects of temperature and vacuum pressure on the properties of biochar derived from Ashe juniper biomass. The specific objectives are as follows:

- a To characterize the combined effect of temperature and vacuum pressure on the biochar properties (elemental composition, degree of carbonization, proximate analysis, surface functionality, and textile structures),
- b To determine the potential application of tuned biochar based on the properties, and
- c To evaluate the removal capacity of biochar as an adsorbent on a target chemical.

2. Material and methods

2.1. Feed material

Ashe juniper was obtained by thinning from Hills Country (TX, USA). Ashe juniper was crushed and dried for 2 h before pyrolysis. The volatility, fixed carbon and ash contents were determined following ASTM E870-82 [20]. The elemental composition was determined with an ultimate analyzer (Vario MICRO elemental analyzer).

2.2. Vacuum pyrolysis for the production of oils and biochar

Ashe juniper was pyrolyzed in a vacuum pyrolysis machine as shown in Fig. 1. For each experiment, 10 g of the raw biomass was placed in a glass flask (Chemglass), before it was inserted into an electrical furnace. The main flask was connected to a condenser for collection of the heavy fraction (Fraction 1) located before a cold trap

(−196 °C) for the light fraction (Fraction 2). A mechanical vacuum pump placed after the cold trap provided the vacuum condition for vacuum pyrolysis of biomass. The reactor system was evacuated at 0.09, 0.7 and 3 kPa (denoted as 1, 2 and 3) under air atmosphere when temperature conditions were set to 350, 450 and 520 °C at a heating rate of 20 °C/min controlled by PID controllers under a vacuum condition. Biochar produced at certain temperature and pressure was named B-temperature-pressure. For example, B-350-1 refers to the biochar produced at 350 °C and 0.09 kPa. Pyrolysis condition is denoted as temperature-pressure. At the desired temperature, the system was maintained for 1 h. Each vacuum pyrolysis was conducted three times at an experimental condition. As a control experiment, biochar was produced at a temperature of 350, 450 and 520 °C under nitrogen atmospheric pressure condition. Biochar produced for the control experiment was named B-temperature-N.

2.3. Characterization of biochar

The elemental composition of biochar was analyzed with an ultimate analyzer (Vario MICRO elemental analyzer). The proximate analysis of biochar was conducted following ASTM D3172 [21]. The nitrogen adsorption isotherms of biochar were obtained by nitrogen (Airgas, 99.999%) adsorption at −196.15 °C (NOVA 4200e). Biochar samples were degassed for 3 h at 300 °C. The BET surface area was calculated from the Brunauer-Emmett-Teller (BET) equation [22]. The micropore volume was calculated by the Dubinin-Radushkevich (DR) equation [23]. The total volume was analyzed at a relative pressure of $P/P_0 = 0.99$. The mesopore volume was determined from the difference between total pore volume and micropore volume. The functional groups were investigated by Fourier transform infrared spectroscopy (Shimadzu IRAffinity-1 FTIR). The spectra were recorded between 400 and 4000 cm^{-1} with a resolution of 4 cm^{-1} .

2.4. Methylene blue (MB) adsorption test

A 30 ppm methylene blue solution was prepared in deionized water. Each biochar, derived from vacuum pyrolysis and conventional pyrolysis, was submerged in solution at a ratio of 50 mg of biochar to 20 ml of solution. Each batch was shaken at 100 rpm for 24 h. Then, the solution was filtered and the filtrate was collected. The absorbance of the filtrate was measured at 664 nm using UV-spectroscopy. The amount of MB adsorbed on the biochar at equilibrium q_e (mg/g) was calculated as:

$$q_e = V(C_0 - C_e)/w \quad (1)$$

where C_0 and C_e are the concentration of MB at initial and equilibrium. V is the volume of the MB solution, and w is the mass of dry biochar.

3. Results and discussion

3.1. Biomass characteristics

Table 1 shows the comparison of Ashe juniper characteristics to some typical feedstocks. The moisture content of Ashe juniper is higher than all the other raw materials listed: rice straw [24], corn stover [7], woodchip [25] and saw dust [26]. Similar to woodchip (0.3%) and sawdust (0.45%), Ashe juniper (0.6%) exhibited relatively lower ash content compared to other lignocellulosic biomass. Elemental analysis revealed that Ashe juniper comprised the highest carbon element content among the listed raw materials. It suggested that Ashe juniper might be a promising precursor to develop carbon-based functional materials.

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