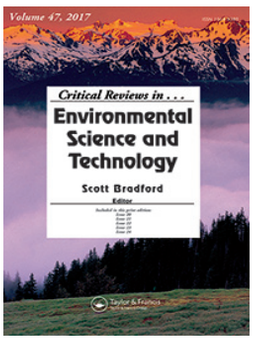

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Advances and future directions of biochar characterization methods and applications

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ABSTRACT

Biochar is a carbon-rich by-product of the thermal conversion of organic feedstocks and is primarily used as a soil amendment. Identification and quantification of biochar properties are important to ensure optimal outcomes for agricultural or environmental applications. Advanced spectroscopic techniques have recently been adopted in biochar characterization. However, biochar characterization approaches rely entirely on the user's choice and accessibility to the new technology. The selection of proper methods is vital to accurately and consistently assess biochar properties. This review critically evaluates current biochar characterization methods of proximate, ultimate, physicochemical, surface and structural analyses, and important biochar properties for various applications.

KEYWORDS

Advanced spectroscopic analysis; black carbon; characterization; pyrolysis; surface functional group

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Introduction

The International Biochar Initiative (IBI) defines biochar as a solid material produced from feedstock carbonization (IBI, 2015). Biochar is a by-product of the thermal decomposition of organic feedstocks, such as wood, plant leaves, and crop residue, produced under an oxygen (O_2) limited environment at <700 °C (Hans-Peter, 2013; Lehmann and Joseph, 2009). Fast pyrolysis, slow pyrolysis, and gasification are common methods used for biochar production (Ahmad et al., 2014b; Mohan et al., 2014). However, slow pyrolysis is the most extensively adopted method for biochar production because it offers the highest yield of biochar (Manyà, 2012). By contrast, fast pyrolysis and gasification lead to the highest yields of liquid (bio-oil) and gas (syngas), respectively (Mohan et al., 2014) (Figure 1). Typically, biochar contains more than 60% carbon (C) and residence time can be extended to >1,000 yr depending on the production and feedstock conditions (Kuzyakov et al., 2014; Zimmerman, 2010). Biochar properties depend on the feedstock (i.e., elemental composition, moisture, lignin, cellulose, hemicelluloses, and inorganic compounds) and thermal conversion conditions (Alexis et al., 2007; Böehm, 1994; Yip et al., 2007). Additionally, biochar-like materials (e.g., pyrogenic carbon) are found in soils worldwide where natural fires have occurred and/or historical management methods have been practiced (IBI, 2015). In most fires, because part of the vegetation can be partially burned in the areas with limited O_2 supply, some portion remains as char (Kuhlbusch and Crutzen, 1995).

Biochar has been used as a soil amendment material for more than 2,000 yr in the Amazon basin (Gul et al., 2015; Kuhlbusch and Crutzen, 1995). Amazonian Dark Earths or Terra Preta de Indio are soils within ancient human-built landscapes that are purported to have formed due to repeated application of char by residents over

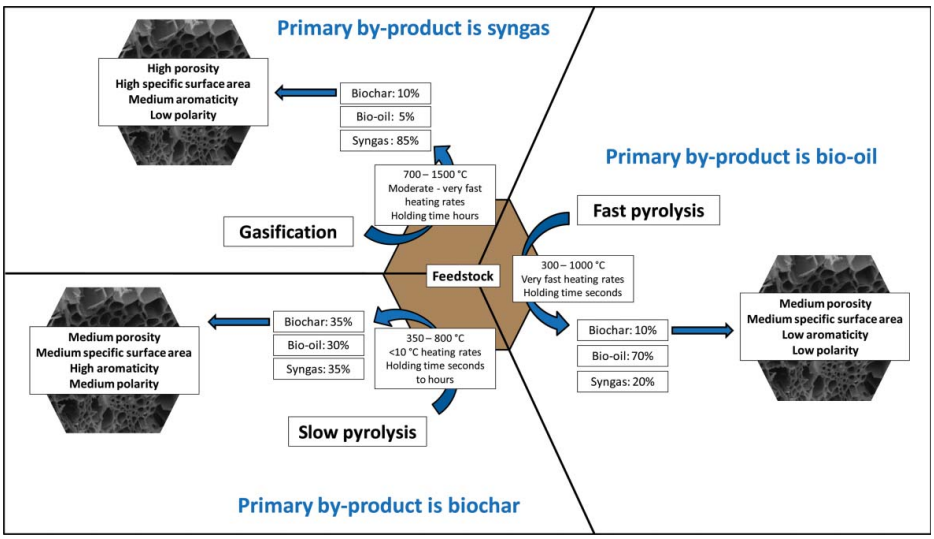

Figure 1. Biochar production methods and the estimated differences in biochar yields obtained from different methods (modified from Brewer et al., 2009; Mohan et al., 2014).

time; these soils have been identified as highly fertile agricultural soils (Graham, 2006; Neves and Petersen, 2006). A significant amount of stored C in the form of char led to the enhancement of soil fertility, playing an important role as a sink for atmospheric carbon dioxide (CO_2) (Ogawa et al., 2006). This process can promote the development of a fine and highly porous char that has similar properties to biochar.

Biochar has been shown to improve the biological, chemical, and physical properties of some soils (Jeffery et al., 2011; Lehmann et al., 2011; Mulcahy et al., 2013). Biochar has been used as an amendment to increase food security and cropland diversity in soils that are severely depleted and have limited supplies of organic matter, water, and chemical fertilizer inputs (nitrogen (N), phosphorus (P), potassium (K)) (Atkinson et al., 2010; Igalavithana et al., 2016). The application of biochar has led to water quality improvements with increased retention of nutrients for plant utilization in those soils (Steiner et al., 2010). Prevention of nutrient leaching is considered another crucial role of biochar in soils (Hussain et al., 2016). In addition to soil quality enhancement, C in biochar is highly resistant to degradation, which is an important characteristic for C sequestration in soil. Carbon can also increase soil aggregation and arable soil productivity (Hansen et al., 2015; Mao et al., 2012). Moreover, it is useful for soil and water remediation (Abid et al., 2016; Ahmad et al., 2014a; Mohan et al., 2014; Rizwan et al., 2016). In general, both inorganic (e.g., heavy metals) and organic (e.g., pharmaceuticals and polyaromatic hydrocarbons (PAHs)) contaminants show positive affinities for retention in the biochar matrix. Thus, biochar can effectively be used to remove pollutants from soil and water (Bair et al., 2016; Hale et al., 2012; Mohan et al., 2014, 2012; Park et al., 2015). However, biochar has also shown negative and neutral effects on biological, chemical, and physical properties of soils (Mukherjee and Lal, 2014). Hence, characterization of biochar and soil is essential prior to application.

The primary understanding regarding the use of biochar focuses on its potential for soil fertility improvements, C sequestration in soil, and water and soil remediation. However, biochar use is currently being expanded to media other than soil to span a variety of disciplines (Ok et al., 2015). Many attractive features of biochar (e.g., its sustainability, easy production process, and low cost), as well as its unique primary and secondary properties (e.g., surface area, pore volume, pore size, pH, cation exchange capacity (CEC), electrical conductivity (EC) and surface functional groups) have helped expand its use in many different disciplines (Brown, 2009) (Figure 2). Next generation biochar applications (e.g., catalysis, medicinal uses, supercapacitors, gas adsorbent, fuel cell systems, and energy/gas storage) are still in their infancy. Although biochar applications are advancing, lack of knowledge about its mode of action causes uncertainties in different disciplines.

Although modes of action of biochar in many disciplines have not been revealed, in-depth studies of biochar properties and current methods used to identify and quantify biochar properties will facilitate our understanding of the potential roles of biochar in different applications. Moreover, effective and consistent characterization of biochar may extend its current uses and reveal new areas of

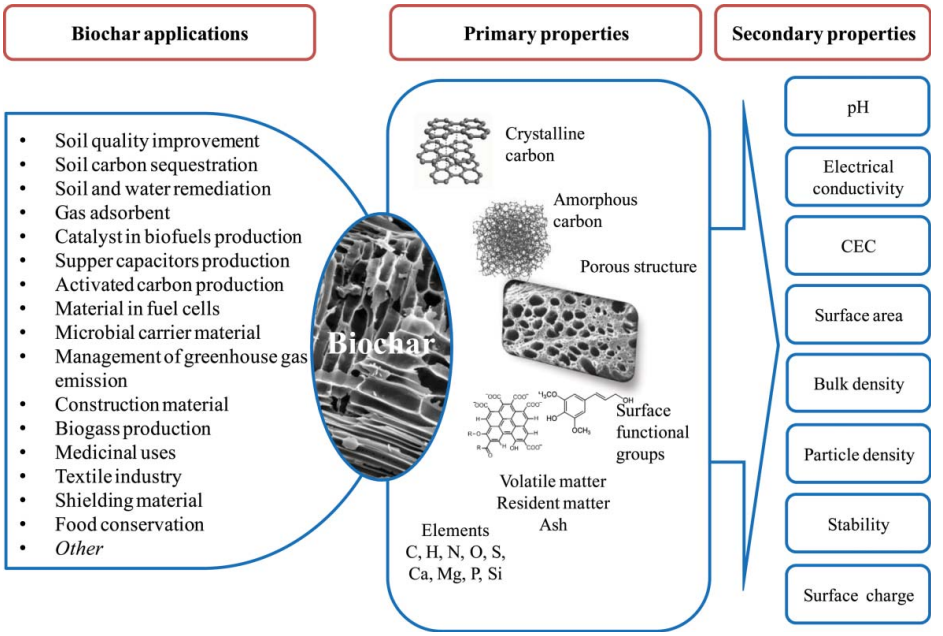

Figure 2. Biochar properties and applications. The primary properties of biochar are directly influenced by the feedstock and the production conditions. The secondary properties of biochar are factors of its primary properties. Both the primary and secondary properties of biochar facilitate different applications.

application. Therefore, in this review, we succinctly and critically evaluated biochar characterization methods and their advances, future trends in biochar applications, and biochar properties considered in different applications.

Significance of biochar characterization

Prior to using biochar in a particular application, it is essential to characterize biochar to optimize its use. Various methods are used worldwide to identify and quantify the biochar properties. The IBI documented the "standard product definition and product testing guidelines for biochar that is used in soil" (IBI, 2015). The latest version of these IBI guidelines was published in November 2015 and clearly states that biochar characterization is not fixed, and not implying that characterizing biochar with other suitable methods may produce unreliable data. However, the IBI strictly advises that biochar is characterized before utilizing it as a soil amendment. The mission of the IBI is to test and use biochar as a soil fertility enhancer and climate change mitigation tool (IBI, 2015). Hence, biochar characterization in other pertinent fields is not considered in the IBI guidelines. The European Biochar Foundation published the European Biochar Certificate (EBC), which also provides guidelines for biochar production as an agricultural amendment (Hans-Peter, 2013). Primary concerns of the EBC in biochar characterization are comparable with those of the IBI; it is not considered to be a solid statement of

guidelines that must be followed. Moreover, no documents have been published by international organizations on biochar characterization for use in other disciplines. Therefore, any available technology can be used for biochar characterization, although this could compromise the validity and reliability of the data. Poor accessibility to technologies and insufficient knowledge or perspective often leads to the selection of improper characterization methods.

According to the Scopus database, 4,365 biochar-based studies have been published since 2004 (queried on November 6, 2016). Most of these studies focused on the characterization methods, applications, and properties of biochar. We sorted these studies by searching for "biochar" and "characterization" or "property" or "properties" or "characteristics" or "parameter" or "parameters" in the article title, abstract, and keywords to arrange them systematically. We identified 3,934 articles that had been published since 2004 (queried on November 6, 2016) as articles, conference papers, articles in press, reviews and book chapters, conference reviews, and books. These studies were grouped into 26 subject categories: environmental science; agriculture and biological science; chemical engineering; chemistry; medicine; energy; engineering; earth and planetary sciences; immunology and microbiology; biochemistry, genetics, and molecular science; materials science; physics and astronomy; pharmacology, toxicology, and pharmaceuticals; social sciences, multidisciplinary, business management and accounting; economics, econometrics and finance; computer science; veterinary science; mathematics; nursing; arts and humanities; decision sciences; health professions; and neuroscience. Most articles were published in the subject category of environmental science (51.0%), followed by agriculture and biological science (34%), chemical engineering (21.0%), and chemistry (16.8%).

In the published literature, biochar characterization has been carried out with three main objectives: (1) to understand the physical and chemical properties of biochar, including variations of the biochar properties as a function of production conditions and feedstocks (Brewer et al., 2009; Keiluweit et al., 2010; Mukome et al., 2013; Novak et al., 2009) (Figure 3); (2) to evaluate the applicability of biochar in desired fields (Enders et al., 2012; Melligan et al., 2012; Yao et al., 2011; Zhou et al., 2014); and (3) to examine the biochar contaminants and ecotoxicological parameters (Oleszczuk et al., 2013; Zielińska and Oleszczuk, 2015).

The fundamental research approach of biochar characterization has facilitated the development of new methods and advancement of the existing methods (Brewer et al., 2014; Enders et al., 2012). Biochar characterization methods are always independent of production feedstocks, methods, conditions, and properties of the final product (IBI, 2015). Various chemical characterizations of biochar have been carried out, ranging from biochar surface analysis to elemental composition. Biochar physical properties are commonly analyzed based on the surface area, pore size, and pore volume (Brewer et al., 2014). The bulk density and particle size distribution of biochar have also been determined in some studies (Abdullah and Wu, 2009; Jaafar et al., 2015). Biochar physicochemical characterization methods and depth of analysis mainly depend on the objectives of the study and available

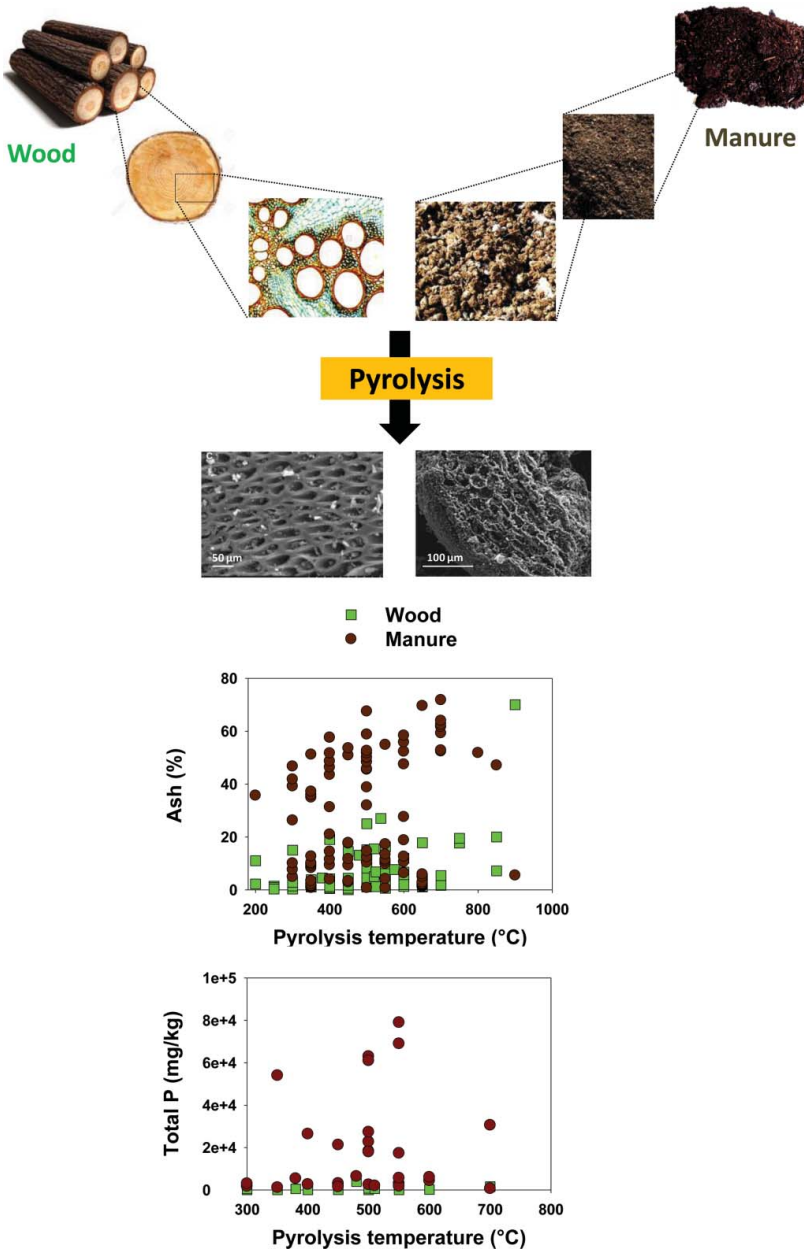

Figure 3. Changes in the biochar morphological characteristics, ash, and total P contents as a function of production feedstock. SEM images from Ahmad et al., 2013a; Lim et al., 2015. Data obtained from UC Davis Biochar Database, 2015.

facilities. Moreover, various methods, ranging from very basic to sophisticated, have been adopted to identify and quantify the biochar properties (Figure 4). The principles, similarities, and importance of systematically arranged biochar characterization methods used in environmental, agricultural, and biological sciences are discussed in the next section. The evaluation of the variation in biochar properties

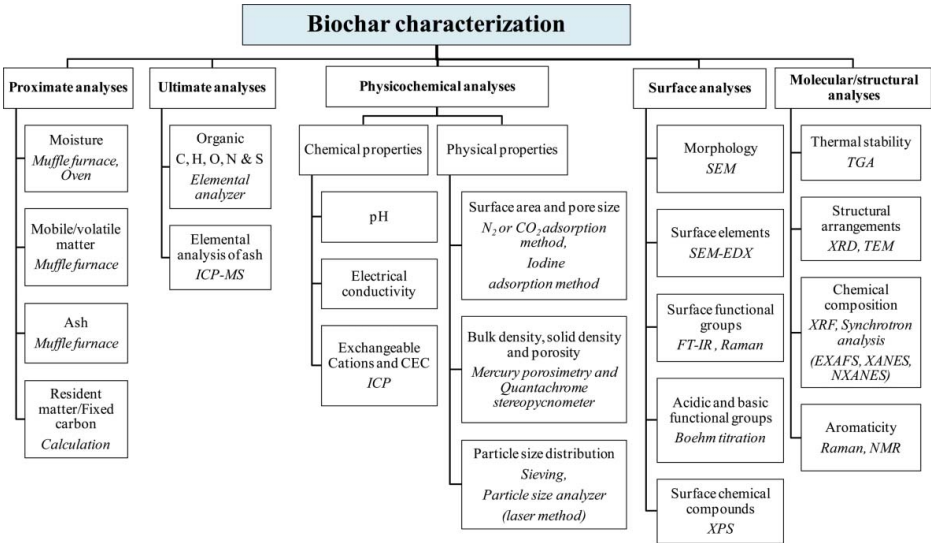

Figure 4. Graphical overview of biochar characterization indicating the analytical approaches for specific chemical and physical properties. ICP-MS: inductively-coupled plasma mass spectroscopy, SEM: scanning electron microscopy, SEM-EDX: SEM with energy-Dispersive X-ray spectroscopy (EDX), FT-IR: Fourier transform infrared spectroscopy, XPS: X-ray photoelectron spectroscopy, TGA: thermal gravimetric analysis, XRD: X-ray diffraction, TEM: transmission electron microscopy, XRF: X-ray fluorescence, NMR: nuclear magnetic resonance spectroscopy, EXAFS: extended X-ray absorption fine structure spectroscopy, XANES: X-ray absorption near-edge structure spectroscopy, NEXAFS: near-edge X-ray absorption fine structure spectroscopy.

as a function of production feedstocks, production methods, and conditions are not considered in this review; nevertheless, these areas are used as supporting materials to discuss biochar characterization methods and biochar applications.

Proximate analysis

Original methods of proximate analysis

Proximate analysis is a basic characterization procedure that can be used to determine the moisture, mobile matter/volatile matter (hereafter, referred to as volatile matter), fixed C/resident matter (hereafter, referred to as resident matter), and ash content in biochar (Joseph et al., 2009). The standard test method ASTM D1762-84, which was published by the American Society for Testing and Materials, is commonly used for this purpose (IBI, 2015). Briefly, air-dried ground samples are passed through a No. 20 (850 μm) sieve. Excessive grinding is not recommended because moisture and mobile matter can be lost due to the generation of heat. In addition, a large number of fine particles smaller than a No. 100 (150 μm) sieve are produced if the grinding time is too long. These very small particles are swept away during the evolution of gases when analyzing the mobile matter. It is recommended that particles retained in the 850-μm sieve not be used in the analysis; a precise explanation of this was not given in the original ASTM D1762-84 protocol.

However, the particle size distribution obtained from the 850-μm sieve can show a significant variation, as it is the first sieve through which the biochar passes after grinding. Therefore, to maintain a uniform particle size distribution in every sample, retaining the particles in the 850-μm sieve is not recommended. In addition, it is not possible to liberate absolute volatile matter from large biochar particles due to the significant amount of covered porous structures. Hence, the use of sieves between 850 and 150 μm is recommended. This also allows for the comparison of diverse biochars produced under similar pyrolysis programs.

Prior to determining the biochar moisture content, ASTM D1762-84 recommends that empty crucibles be heated at 750 °C for 10 min in a heated muffle furnace to ignite all of the residues remaining in the crucibles after cleaning. The crucibles should then be cooled in a desiccator for 1 hr. Finally, 1 g of biochar is placed in each crucible and heated at 105 °C for 2 hr to determine the moisture content gravimetrically (Ahmad et al., 2012a). The biochar can then be used to determine the amount of volatile matter following a stepwise procedure provided in ASTM D1762-84. Briefly, the covered crucibles are kept on the furnace outer ledge at 300 °C for 2 min, on the edge of the furnace at 500 °C for 3 min, and in the closed furnace at 950 °C for 6 min. In ASTM D1762-84, it is recommended that nichrome wire baskets be used to hold the biochar-containing crucibles in each step. After completing the heating steps, the weights of the crucibles with the remaining contents are determined after cooling for 1 hr in a desiccator.

The method used to determine the ash content in biochar is explained in ASTM D1762-84. The crucibles are uncovered and heated at 750 °C for 6 hr in a muffle furnace immediately after determining the amount of volatile matter. The weights are measured after cooling the crucibles in a desiccator for 1 hr and then, the sample heating step is repeated at the same temperature for 1 hr until the weight loss is less than 0.0005 g.

The moisture (Eq. 1), volatile matter (Eq. 2), and ash (Eq. 3) content calculations are as follows (ASTM D1762-84, 2013).

$$\text{Moisture}(\%) = [(w_1 - w_2) \,/\, w_1] \times 100 \tag{1}$$

$$\text{Volatile matter}(\%) = [(w_2 - w_3) \,/\, w_2] \times 100 \tag{2}$$

$$\text{Ash}(\%) = (w_4 \,/\, w_2) \times 100 \tag{3}$$

Here, w_1 is the weight of the air-dried sample, w_2 is the weight of the sample after being heated at 105 °C, w_3 is the weight of the sample after being heated at 950 °C, and w_4 is the weight of the residue after being heated at 750 °C.

To determine the moisture, volatile matter, and ash content in biochar, ASTM D5142-04 (ASTM D5142 – 04, 2004) ("standard test methods for proximate analysis of the sample of coal and coke by instrumental procedure") is also used (Ghani et al., 2013). The two methods specified in ASTM D1762-84 and ASTM D5142-04 are fairly similar. The method described by ASTM D5142-04 specifies a temperature range of 104–110 °C to determine the moisture content. The peak temperature for determining

the mobile matter content is 950 ± 20 °C and the holding time is 7 min. The peak temperature is achieved with a heating rate of 50 °C/min. The furnace should have an inert atmosphere; the use of N_2 gas at a rate of 2–4 vol/min is recommended. A stepwise procedure for determining the volatile matter content, similar to the one given in the ASTM D1762-84 method, is not provided in the ASTM D5142-04 method.

ASTM D5142-04 provides a stepwise heating procedure to determine the ash content. If the volatile matter is determined during the previous sample ashing, the temperature is increased to 700–750 °C and then heated to 900–950 °C until the weight is constant. A holding time at 700–750 °C was not recommended for this method. If the amount of volatile matter in the sample is not determined, the sample is heated at 450–500 °C for 1 hr, at 700– 750 °C for 2 hr, and then at 900–950 °C until the weight is constant.

Angin (2013) used two other methods, ASTM 3174-12 (ASTM D3174-12, n.d.) and ASTM 3175-11 (ASTM D3175-11, n.d.) (ASTM D3175-11), to determine the ash and volatile matter contents, respectively. Both methods were developed to determine the amounts of ash and volatile matter in coal and coke, which have very similar properties to biochar. However, the use of ASTM D3175-11 in proximate analysis of biochar is limited in normal laboratory conditions because it requires a special electric furnace and platinum crucibles.

Proximate analysis is also needed for the resident matter in biochar and the following equations can be used for this calculation (Enders et al., 2012).

$$\text{Resident matter \%} = \left(\left[w_2 - w_3 - w_4\right] / w_2\right) \times 100 \tag{4}$$

$$\text{Resident matter \%} = 100 - (\text{Moisture\%} + \text{Mobile matter\%} + \text{Ash\%}) \tag{5}$$

Here, w_2, w_3, and w_4 are the same as the variables mentioned in Eqs (1) to (3). The methods adopted by the authors for biochar proximate analysis are listed in Table 1. The ASTM methods, methods modified from the ASTM methods, and author's developed methods have been used to determine the proximate analysis in biochars.

Modified methods for biochar proximate analysis

The ASTM methods were not developed for the proximate analysis of biochar, but for other materials similar to biochar. Therefore, researchers have made various changes to these methods and/or have developed new methods. Rutherford et al. (2012) determined the moisture and ash contents in biochar using a method based on ASTM methods. Biochar was heated overnight at 105 °C to determine the moisture content and the amount of ash was determined at 750 °C. However, they did not specify the heating time used to determine the ash content.

Numerous efforts have been made to modify the ASTM D1762-84 method (Dean, 1999; Enders et al., 2012; McLaughlin et al., 2009; Ronsse et al., 2013). Enders et al. (2012) modified this method to determine the volatile matter content. Samples preheated in the outer ledge and at the edge of the furnace were omitted.

Table 1. Methods used for biochar proximate analyses in articles within the fields of environmental science, agriculture, and biological science.

Feedstock	Pyrolysis temperature (°C)	Moisture	Mobile matter/volatile matter	Ash	Reference
■ Anaerobic digestion residue ■ Palm bark ■ Eucalyptus	400	Overnight heating at 105 °C	Not determined	Not determined	(Sun et al., 2013)
■ Rubber wood sawdust	450, 550, 650, 750, 850	Not determined	Not determined	Not given	(Ghani et al., 2013)
■ Miscanthus × giganteus	600	Not determined	Heating at 900 °C for 7 min (derived from standard procedure CEN/TS 15148:2005)	Not determined	(Melligan et al., 2012)
■ Solid hydrolysis residual materials 1 ■ Solid hydrolysis residual materials 2 ■ Solid hydrolysis residual materials 3 ■ Solid hydrolysis residual materials 4					
■ Giant reed (*Arundo donax* L.)	200, 300, 350, 400, 500, 600	Not determined	Not determined	Heating at 750 °C for 4 hr	(Wang et al., 2013)
■ Switchgrass ■ Corn stover	500 (slow pyrolysis), 500 (fast pyrolysis), 750 (gasification)	ASTM D1762-84	ASTM D1762-84	ASTM D1762-84	(Brewer et al., 2009)
■ Hardwood	500 (slow pyrolysis)	ASTM D1762-84	ASTM D1762-84	ASTM D1762-84	(Brewer et al., 2009)
■ Corn stover	700 (gasification), 450 (fast pyrolysis)		Heating at 950 °C	Not given	(Lee et al., 2010)
■ Miscanthus sacchariflorus	300, 400, 500, 600	Not given	Not given	Not given	(Kim et al., 2013)
■ *Pinus banksiana* (pine wood) ■ *Phleum pratense* (timothy grass) ■ *Triticum aestivum* (wheat straw)	450	ASTM D1762-84	ASTM D1762-84	ASTM D1762-84	(Nanda et al., 2013)
■ Rice straw (*Oryza sativa*)	300, 400, 500, 600, 700	Not determined	ASTM D1762-84	ASTM D1762-84	(Wu et al., 2012)
■ Corn stover ■ Switchgrass	500 (fluidized bed fast pyrolysis), 600 (freefall fast pyrolysis), 550 (freefall fast pyrolysis), 500 (freefall fast pyrolysis), 732 (air-blown gasification), 500 (slow pyrolysis)	ASTM D1762-84	ASTM D1762-84	ASTM D1762-84	(Brewer et al., 2011)
■ Red oak	500 (fluidized bed fast pyrolysis)	ASTM D1762-84	ASTM D1762-84	ASTM D1762-84	(Brewer et al., 2011)
■ Mixed hardwood	∼400 (kiln slow pyrolysis)	ASTM D1762-84	ASTM D1762-84	ASTM D1762-84	(Brewer et al., 2011)
■ Wood waste	∼800 (air-blown gasification)	ASTM D1762-84	ASTM D1762-84	ASTM D1762-84	(Brewer et al., 2011)
■ Eastern hemlock	550 (Auger fast pyrolysis)	ASTM D1762-84	ASTM D1762-84	ASTM D1762-84	(Brewer et al., 2011)
■ Ponderosa pine shavings ■ Tall fescue straw	100, 200, 300, 400, 500, 600, 700	ASTM D1762-84	ASTM D1762-84	ASTM D1762-84	(Keiluweit et al., 2010)
■ Safflower seeds (*Charthamus tinctorius* L.)	400, 450, 500, 550, 600	Not determined	ASTM 3174-12 and ASTM 3175-11	ASTM 3174-12 and ASTM 3175-11	(Angın, 2013)
■ Peanut shells ■ Chicken litter ■ Pine wood	450	Not determined	ASTM D3172	ASTM D3172	(Smith et al., 2013)

■ Miscanthus grass	350, 360, 370, 400, 425, and 450 (intermediate pyrolysis)	ASTM D1762-84	Not determined	ASTM D1762-84	(Brewer et al., 2014)
■ Mesquite wood	300, 350, 400, 450, 550, 600, 700 (slow pyrolysis)	ASTM D1762-84	Not determined	ASTM D1762-84	(Brewer et al., 2014)
■ Wood ■ Straw ■ Green waste ■ Algae	300, 450, 600, 750 (slow pyrolysis)	Not determined	ASTM D1762-84 (modified)	ASTM D1762-84 (modified)	(Ronsse et al., 2013)
■ Corn straw	450	Not determined	Not determined	ASTM D1762-84	(Zhao et al., 2013)
■ Macadamia shells	Not given	ASTM D121	ASTM D5142	ASTM D7582	(Spokas et al., 2011)
■ Oak hardwood sawdust	500 (fast pyrolysis)	ASTM D121	ASTM D5142	ASTM D7582	(Spokas et al., 2011)
■ Macadamia shells	650 (fast pyrolysis)	ASTM D121	ASTM D5142	ASTM D7582	(Spokas et al., 2011)
■ Corn stover	515 (fast pyrolysis)	ASTM D121	ASTM D5142	ASTM D7582	(Spokas et al., 2011)
■ Pine wood chips	465 (slow pyrolysis)	ASTM D121	ASTM D5142	ASTM D7582	(Spokas et al., 2011)
■ Peanut hulls	481 (slow pyrolysis)	ASTM D121	ASTM D5142	ASTM D7582	(Spokas et al., 2011)
■ Perennial grass (*Arundo donax* L.)	Not given	ASTM D3173	ASTM D3175	ASTM D3174	(Saikia et al., 2015)
■ Pine saw dust	680	Not determined	Not determined	ASTM D5630	(Srinivasan et al., 2015)
■ *Jatropha curcasa* ■ *Pongamia pinnata* ■ *Tung*	300, 400, 450	ASTM D3172-07	ASTM D 3173–87	ASTM 3174-04	(Kumar and Pant, 2015)
■ Pine wood	350 – 600	ASTM D2216	ASTM D1762-84	ASTM D1762-84	(Yargicoglu et al., 2015)
■ Leaves and bark	400, 580	CEN 14775	Not determined	CEN 14774	(Lievens et al., 2015)
■ White wood	500	Not determined	Not determined	ASTM D 2896-11	(Shahkarami et al., 2015)
■ Water hyacinth	350	Not determined	ASTM D1762-84	ASTM D1762-84	(Doumer et al., 2015)
■ Soybean stover ■ Peanut shells	300, 700	24 hr heating at 105 °C (modified method by McLaughlin et al. (2009))	Heating in a covered crucible at 450 °C for 30 min (modified method by McLaughlin et al. (2009))	Heating in an open crucible at 700 °C (modified method by McLaughlin et al. (2009))	(Ahmad et al., 2012a)
■ Pine needles	300, 500, 700	Overnight heating at 105 °C for a constant temperature	Heating in a covered crucible at 450 °C for 1 hr	Heating in an open crucible at 700 °C for 1 hr	(Ahmad et al., 2013b)
■ Tea waste	300, 700	As explained in Ahmad et al. (2013b)	As explained in Ahmad et al. (2013b)	As explained in Ahmad et al. (2013b)	(Rajapaksha et al., 2014)
■ Buffalo weed (*Ambrosia trifida* L.)	300, 700	24 hr heating at 105 °C	Heating in a covered crucible at 450 °C for 1 hr	Heating in an open crucible at 700 °C for 1 hr	(Ahmad et al., 2014a)
■ Bur cucumber	300, 700	24 hr heating at 105 °C	Heating in a covered crucible at 450 °C for 1 hr	Heating in an open crucible at 700 °C for 1 hr	(Vithanage et al., 2014)

Sample-containing crucibles were directly placed inside the heated furnace and held at 950 °C for 8 min. The samples were then placed in a refractory brick oven until they cooled to around 200 °C and were transferred to a desiccator. Enders et al. (2012) analyzed the low-temperature volatile matter at 350 °C. Samples in covered crucibles were placed in a furnace preheated to 105 °C. The furnace was then heated to 350 °C by increasing the temperature at a rate of 5 °C/min and held at 350 °C for 2 hr. The crucibles were then quickly transferred to a desiccator and weights were measured in cooled crucibles. Ronsse et al. (2013) also made several modifications to the ASTM D1762-84 method. Biochar was heated at 950 °C for 11 min without any preheating processes to determine the amount of volatile matter; this may have been done to avoid the complexity of the original ASTM D1762-84 method. However, the authors did not compare the original method with the modified method. Their method to determine the ash content was also different from the original method in ASTM D1762-84. Ronsse et al. (2013) heated the biochar at 750 °C for 2 hr instead of 6 hr. The accuracy of ash determination when using a modified heating time depends on the biochar structural stability and the amount of heat-sensitive inorganic compounds (Dean, 1999; Ronsse et al., 2013).

High-temperature exposure for a long period of time (when determining the ash content) can result in the volatilization of carbonates and some elements (e.g., P, K, and sulfur (S)). Hence, the ash content could be underestimated in biochars that have high ash content, such as biochar from manure (Dean, 1999; Enders et al., 2012). Performing calculations based on the amount of mobile matter determined at 950 °C can lead to an overestimation because the ash in biochar is on top of the crucible and the crucible cover creates poor anoxic conditions inside, which often induces complete burning (Enders et al., 2012). Hence, researchers have made modifications to the ASTM D1762-84 procedure to overcome these limitations of the proximate analysis. In the modified proximate analysis, a temperature of 450 °C is used to determine the volatile matter content. The ashing temperature was also reduced to 500–550 °C to avoid the volatilization of elements and carbonates (McLaughlin et al., 2009). However, the authors are still used the unmodified ASTM D1762-84 procedure in the biochar proximate analysis (Güereña et al., 2013; Laird et al., 2010; Lehmann et al., 2011; Nelissen et al., 2014; Ronsse et al., 2013). The IBI (2015) also recommended using ASTM D1762-84 for determining the moisture, mobile matter, and ash content of biochar used for soil amendment.

Ultimate analysis

Biochar ultimate analysis mainly focuses on individual elements and chemical compounds. Among these, the amounts of C, hydrogen (H), N, O, and S in the organic fraction of biochar are the main concerns. In most cases, the amount of S in biochar is negligible (Cheah et al., 2014), and the amount of N is also often very low (except for feedstocks that have higher N content) (Spokas et al., 2012). In addition, high heat sensitivity reduces the N content in biochar (Yuan et al., 2010). Consequently,

the amounts of C, H, and O in the organic fraction are the main elements considered in the ultimate analysis in many studies. The ultimate analysis is often performed in parallel with proximate analysis and elemental analyzers are employed (Almaroai et al., 2014; Rajapaksha et al., 2014; Saikia et al., 2015). The combustion temperature (>900 °C) of elemental analyzers is sufficient to combust all of the constituents, including ash. Hence, subtracting the ash and moisture content obtained via proximate analysis for the initial biochar weight is important to determine the organic fractions of C, H, O, N, and S (Rajapaksha et al., 2014). Moreover, it is possible to calculate the volatile and resident percentages of C, H, O, N, and S depending on the study objectives. However, scientists have analyzed the total organic C, H, O, N, and S elemental composition without determining whether the elements are volatile or resident (Ahmad et al., 2012a; Brewer et al., 2009; Sun et al., 2013).

The ash in biochar is composed of minerals; therefore, the analysis of ash composition is also included in the ultimate analysis. Fundamentally, biochar ash can be divided into two groups: acid-soluble and acid-non-soluble. The proportion of these fractions is determined by the feedstock used to produce the biochar. Usually, plant material-derived biochar has a higher percentage of acid-soluble ash (0.5–25% of biochar composition) than acid-non-soluble ash (0–8% of biochar composition) (McLaughlin et al., 2009). Furthermore, the individual elements in ash are analyzed to obtain the detailed biochar composition. The ultimate analysis allows understanding of the elemental composition of biochar in detail. However, minor errors can occur during the calculation of organic C, H, O, N, and S, as well as during the ashing process, because some elements (e.g., P and K) can volatilize. Moreover, the determination of elements can be tricky in high carbon-containing biochar (McLaughlin et al., 2009).

The ultimate analysis is a good tool to predict the biochar performances in different fields. It also provides information about biochar carbonization and potential stability (Budai et al., 2013). The H and O contents of biochar decrease as the pyrolysis temperature increases (Figure 5). Consequently, the ratios of H/C and O/C decrease, demonstrating enhanced biochar aromaticity and carbonization, as well as reduced surface polarity (Uchimiya et al., 2010).

Relationship between proximate and ultimate analyses

Biochar proximate analysis requires a simple laboratory process; the cost of analysis is low compared to that of ultimate analysis. Proximate analysis separates biochar characterization into four categories: moisture, volatile matter, ash, and resident matter. Biochar volatile matter and resident matter contain mainly organic C, H, O, N, and S, while ash contains inorganic compounds. The moisture, volatile matter, and ash contents are determined by thermal gravimetric analysis and the resident matter is determined by calculating the difference between the initial weight and the moisture, volatile matter, and ash. Pearson's correlation coefficient was calculated to analyze the relationship between the proximate analysis used to determine the

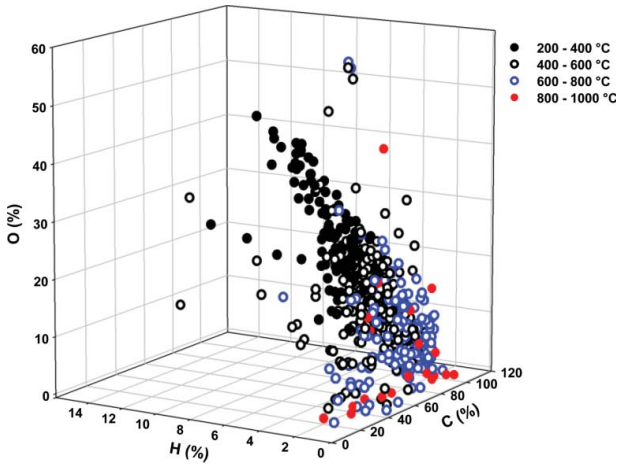

Figure 5. Behavior of biochar organic carbon (C), hydrogen (H), and oxygen (O) (percentages) as a function of pyrolysis temperature. Data obtained from reference (UC Davis Biochar Database, 2015). All biochars listed in the database, which were produced from different feedstocks (e.g., algae, hulls, manure, grass, corn stover, nutshells, pomace, sludge, hardwood, softwood, and others), were considered.

volatile matter and ash contents and the ultimate analysis used to determine the C%, H%, O%, N%, S%, H/C, and O/C molar ratios (Figure 6). The University of California Davis Biochar Database (UC Davis Biochar Database, 2015) (biochar.ucdavis.edu) was used to collect biochar data analyzed with proximate and ultimate analysis methods. Forty biochars produced from different feedstocks (i.e., manure, corn stover, nutshell, hardwood, softwood, and paper sludge) at different production temperatures (i.e., 300, 350, 400, 450, 500, 550, and 600 °C) were selected. The data from biochar proximate and ultimate analyses varied in the following ranges: ash = 0.30–38.60%; volatile matter = 23.50–61.10%; C = 59.90–91.10%; H = 1.80–4.90%; O = 8.50–31.50%; H/C = 0.28–0.97; O/C = 0.07–0.36%.

There was no correlation between the ash and ultimate analysis results (i.e., C%, H%, O%, N%, and their molar ratios). However, strong correlations were detected among volatile matter and C%, H%, O%, H/C, and O/C (Figure 6). Biochar N% has no correlation with biochar volatile matter and ash contents. Hence, the derived statistical equations can be used as a potential calculation method to determine biochar C%, H%, and O% without using ultimate analysis.

Physicochemical analysis

In addition to proximate and ultimate analyses of biochar, many other physicochemical parameters have been evaluated depending on the study objectives. Physicochemical properties of biochar are determined by the feedstock and production conditions (Ahmad et al., 2014b). Methods to determine the physicochemical

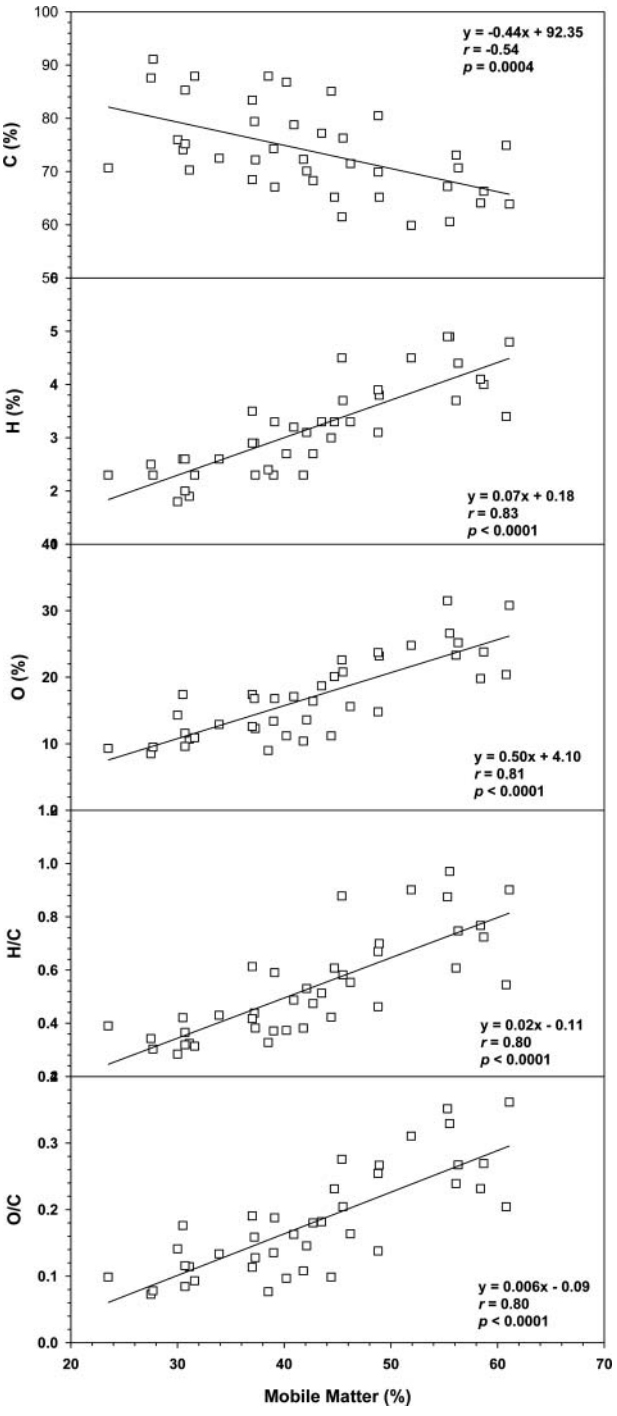

Figure 6. Pearson correlation analysis comparing mobile matter/volatile matter and ultimate analysis data. Data obtained from UC Davis Biochar Database, 2015.

properties of biochar have been derived from the analyses of soil, charcoal, coal, and compost (Mukome and Parikh, 2015). Biochar properties, such as pH, electrical conductivity (EC), and cation exchange capacity (CEC), are similar to those of soil. Hence, methods developed for soil analysis are compatible with biochar analysis. In this section, the commonly used methods for determining the physicochemical properties of biochar are discussed.

pH

The pH of biochar is an essential property used to study pH-dependent phenomena in environmental, agricultural, and biological sciences. Biochar pH ranges from acidic to alkaline but is commonly reported to be alkaline. Additionally, the pH of biochar tends to increase with increasing pyrolysis temperature (UC Davis Biochar Database, 2015) (Figure 7). Ahmad et al. (2014b) reported a range of pH from <5 to >12. Khan et al. (2014) studied highly acidic biochar (pH of 3.0) produced from hardwood, while other authors utilized biochars with a pH range of 4.0–5.8 produced from hardwood, softwood, algae, switchgrass (*Panicum virgatum*), and grasses (Enders et al., 2012; Ippolito et al., 2016; Ronsse et al., 2013; Wang et al., 2013). Different methods have been applied to determine the pH of biochar; the most frequent method, which involves mixing biochar and deionized water in a mass ratio of 1:20, was found in articles published in the fields of environmental, agricultural, and biological sciences (Table 2). Sun et al. (2013) used biochar at the same mass ratio as water (instead of using deionized water) to determine the pH of biochar. Distilled water was also used to determine the biochar pH (Spokas et al., 2011). In addition, biochar-to-deionized water mass ratios of 1:1, 1:5, and 1:10 have been reported (Al-Wabel et al., 2013; Liu et al., 2015; Mimmo et al., 2014; Sun et al., 2013). Mukome et al. (2013) used biochar-to-deionized

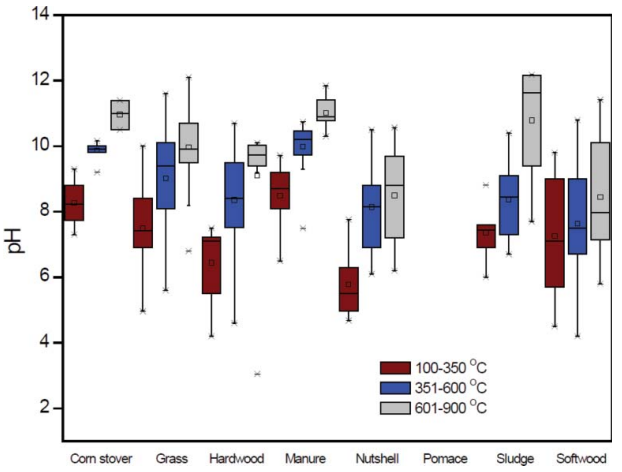

Figure 7. Relationships between biochar pH, pyrolysis temperature, and feedstock. Data obtained from UC Davis Biochar Database, 2015.

Table 2. Methods commonly used by researchers to determine the biochar pH; taken from articles within the subject categories of environmental science, agriculture, and biological science.

Solution	Biochar:solution		Reference
Deionized water	1:20	Mass ratio	(Angın, 2013; Inyang et al., 2012; Xue et al., 2012; Yao et al., 2013a, 2011; Zhou et al., 2013)
	1:10	Mass ratio	(Stefaniuk and Oleszczuk, 2015)
	1:5	Mass ratio	(Liu et al., 2015; Sun et al., 2013)
	1:2	Mass to volume	(Mukome et al., 2013)
	1:3	Mass to volume	(Mukome et al., 2013)
	1:1	Mass ratio	(Mimmo et al., 2014)
Ultra-pure water	3:50	Mass ratio	(Hmid et al., 2014)
Distilled water	1:5	Mass to volume	(Spokas et al., 2011)
Water	1:20	Mass to volume	(Yao et al., 2010)
	1:20	Mass to volume shaken at 90 °C for 2 hr	(Kizito et al., 2015)
	1:10	Mass to volume	(Al-Wabel et al., 2013; Jindo et al., 2012; Vithanage et al., 2016)
KCl	1:10	Mass ratio	(Al-Wabel et al., 2013)
1 M KCl	1:20	Mass to volume	(Wu et al., 2012)
0.1 N KCl	1:10	Mass ratio	(Ronsse et al., 2013)

water mass-to-volume ratios of 1:2 and 1:3 with an equilibration time of 1 hr to determine the pH of biochar. The IBI (2015) recommended a biochar-to-deionized water mass-to-volume ratio of 1:20 with a shaking time of 1.5 hr when determining the biochar pH. In another study, Rajkovich et al. (2012) used a comparatively high mass-to-volume ratio between biochar and deionized water (i.e., 1:20) with shaking to facilitate the equilibration between biochar and deionized water prior to determining the pH. Rajkovich et al. (2012) derived this method from a standard methodology used to determine the pH of compost (IBI, 2015).

Potassium chloride (KCl) has also been used with different biochar-to-solution ratios to determine biochar pH (Ronsse et al., 2013; Wu et al., 2012). This method was primarily developed to determine the soil pH in acidic soils (USDA, 2015). Cation-exchangeable sites in acidic soils can be highly occupied by exchangeable aluminum ions (Al^{3+}). However, determining the pH with deionized or distilled water is not possible when measuring the effect of Al^{3+} ions on soil pH because soil's ability to release Al^{3+} into the solution is negligible. Potassium ion (K^{+}) displaces Al^{3+} ions from the exchangeable sites and increases the H^{+} ion concentration in the solution. Hence, the acidic soil pH is always determined to be around one unit less when measured in KCl than when measured in water (USDA, 2015). Wu et al. (2012) compared the pH value analyzed with water to the pH analyzed with 1 M KCl at a biochar-to-solution ratio of 1:20 (mass to volume, with 1 hr of shaking) in 20 different biochars produced from rice straw. The pH of these biochars was alkaline and ranged from 9.19 to 10.87 and from 8.45 to 10.15 with water and KCl, respectively. Student's t-test was performed in the present study, which showed that these two methods did not significantly differ. Therefore, the use of KCl to determine the pH of alkaline biochar is not recommended. However, the question remains whether KCl can be used to determine the pH of acidic biochars. We could not find a comparison study in the literature for acidic biochars. Hence,

we performed a correlation analysis to compare the total Al content in the biochar and the pH (data obtained from UC Davis Biochar Database, 2015) and found no correlation between them. Deionized water or distilled water might be sufficient to determine the biochar pH. Al-Wabel et al. (2013) used both water (biochar-to-water ratio of 1:10) and KCl (biochar-to-KCl ratio of 1:10) to determine the net biochar surface charge (i.e., $\Delta pH = pH_{(KCl)} - pH_{(water)}$) because it also depends on variable charge components and the point of zero charge.

Electrical conductivity

Electrical conductivity (EC) is associated with water-soluble ions in biochar, similar to those in soil (Rajkovich et al., 2012) and biochar-deionized water suspensions are used to determine EC. Feedstock properties and production conditions are the main drivers of biochar EC (IBI, 2015). The ability to transfer electricity via biochar matrix in electrical cells has been considered and its constant-current charge-discharge abilities have been tested for supercapacitor development (Jiang et al., 2013). However, those measurements significantly differ from those used to determine the biochar EC. They depend on the biochar porous structure, surface area, and the number of crystalline C structures (Jiang et al., 2013). Knowledge of biochar EC is important for biochar applications in agriculture and soil/water remediation. The EC affects plant growth, soil microbial communities, and soil physical properties (e.g., the structure and hydraulic conductivity), thereby indirectly influencing soil nutrient cycling (Gul et al., 2015; Wang et al., 2015). Some authors have failed to provide the methods used to determine the biochar EC in their studies (Dong et al., 2015; Jarvis et al., 2014; Lu et al., 2014). Alternatively, some researchers have determined the EC using a biochar-to-water ratio of 1:10 (mass to volume) (Chintala et al., 2014; Hmid et al., 2014; Jindo et al., 2012; Vithanage et al., 2016), while Stefaniuk and Oleszczuk (2015) used the same ratio with deionized water. Moreover, Fang et al. (2014) determined the EC using a 1:5 ratio of biochar-to-deionized water. Kizito et al. (2015) used biochar boiled with deionized water prior to determining the EC. Briefly, 1 g biochar and 20 mL deionized water were shaken for 2 hr at 90 °C; no information was available about suspension cooling before the measurements. The authors mentioned that they used a heating method to enhance the dissolution of soluble biochar components. Biochar heating with water would be useful to determine the EC in applications affected by higher temperatures because an increase in temperature induces more ions to be released from the biochar matrix due to the dissolution of inorganic compounds. Pituello et al. (2015) also used a 1:20 biochar-to-deionized water ratio without a heating step. The IBI (2015) suggested using the same suspension for pH and EC analyses, as explained above under subtitle of pH.

Exchangeable cations and cation exchange capacity

Available nutrients (macro and micro) in biochar are important for its use in agricultural applications and the nutrient availability depends on biochar feedstock

and production conditions (Igalavithana et al., 2016). Biochar derived from nutrient-rich feedstocks (e.g., manure) contains more available nutrients compared to biochar derived from nutrient-limited feedstocks (e.g., hardwood) (Gaskin et al., 2008; Spokas et al., 2011). The number of available nutrients in biochar can influence the soil nutrients cycling, priming effect, and plant growth (Kuzyakov et al., 2009; Xu et al., 2014). The methods used to determine the available nutrients in biochar are very similar to those used in soil science. Most of the available nutrients are associated with biochar-exchangeable sites. Biochar shows similar properties in its CEC to those of humus in soil (Lee et al., 2010).

Lee et al. (2010) used a modified barium chloride ($BaCl_2$) compulsive exchange method to determine the CEC of biochar. Biochar samples were washed with distilled water to remove the impurities. Several pre-treatment steps were carried out prior to loading $BaCl_2$ into the biochar samples, where magnesium sulfate ($MgSO_4$) was used to displace barium (Ba) from biochar. Conductivity titration was used to calculate the $MgSO_4$ weight and the CEC of biochar, although this method is intricate and time-consuming. Wu et al. (2012) determined the exchangeable cations (K^+, calcium (Ca^{2+}), sodium (Na^+), and magnesium (Mg^{2+})) and CEC of biochar using ammonium acetate (1 M, pH 7). This method was very similar to that used to determine the exchangeable cations and CEC in the soil. Brewer et al. (2011) used a similar method with a lower concentration of ammonium acetate (0.5 M, pH 7) to determine the biochar CEC. Carrier et al. (2013) determined the CEC using ion chromatography; however, the method used to calculate the exchangeable cations was not provided. Researchers have typically determined the number of exchangeable cations and CEC before using biochar for soil amendment.

Surface area and pore size

Determining the surface area of biochar is very important because the biochar surface area has shown positive correlations with contaminant removal from soil and aqueous solutions (Ahmad et al., 2014b; Mohan et al., 2014; Rizwan et al., 2016), the microbial community size and activity in soil (Lehmann et al., 2011), and its performance as a supercapacitor (Jiang et al., 2013). The biochar surface area is a function of the biochar production feedstock and production conditions and is principally related to the production temperature (Figure 8) (Ahmad et al., 2014b; UC Davis Biochar Database, 2015).

Brunauer-Emmett-Teller (BET) is a method commonly used to calculate the biochar surface area (Brewer et al., 2014, 2009; Keiluweit et al., 2010). In the BET method, the amount of liquid N_2 adsorption on the surface is measured at a low temperature (77 K) to calculate the surface area. However, this method has limitations in determining the biochar surface area because N_2 shows kinetic limitations when diffusing through the very small pores of biochar (de Jonge and Mittelmeijer-Hazeleger, 1996). Hence, the N_2 adsorption method can underestimate the

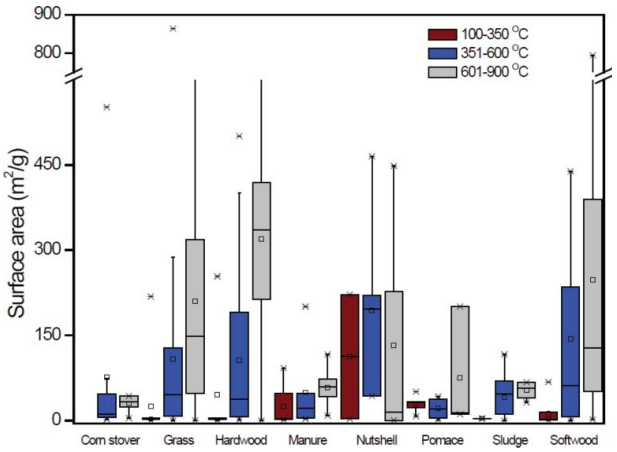

Figure 8. Relationship between biochar surface area, pyrolysis temperature, and feedstock. Data obtained from UC Davis Biochar Database, 2015.

surface area, especially in biochars that contain a large number of small pores. The CO_2 adsorption method at a relatively high temperature (273 K) has been shown to be more sensitive and provide more accurate biochar surface area measurements (Kasozi et al., 2010; Kwon and Pignatello, 2005). Biochar surface area measurements were collected by Yao et al. (2011) with both of these methods; a larger value for the biochar surface was obtained with the CO_2 adsorption method than with the N_2 adsorption method. In another study, Wang et al. (2013) determined the biochar surface area with both the N_2 and CO_2 adsorption methods and reported significantly higher values for the CO_2 adsorption method. Xue et al. (2012) also used N_2 and CO_2 adsorption methods to analyze the micropores and macropores in biochar, respectively. CO_2 adsorption is commonly used to characterize biochar pores smaller than 2 nm (micropores) and N_2 adsorption is commonly used to characterize pores ranging from 2 to 50 nm (micropores and mesopores, respectively) (Brewer et al., 2014). Recent findings by Sigmund et al. (2016) explained that the degassing temperature in both the N_2 and CO_2 methods is also important to accurately measure the surface area of biochar. They found that degassing temperatures >105 °C can lead to the volatilization of labile biochar components, increasing the surface area by opening up the pores that do not represent the actual surface area of the biochar. In addition to the surface area determination, both N_2 and CO_2 methods can be used to determine the pore volume and pore diameter in biochar. For that purpose, the Barrett-Joiner-Halenda (BJH) equation is commonly adapted (Ahmad et al., 2012b; Igalavithana et al., 2017)

The BET surface area method used for biochar was originally adopted from the standard test method for black carbon (ASTM D6556-10, 2010; Uchimiya, 2015). The IBI (2015) included ASTM D6556-10 in the "standard product definition and product testing guidelines for biochar that is used in soil" as the standard method for determining the surface area of biochar. The IBI recommended this method for

the quantification of total surface area and external surface area of biochar. Other than the N_2 and CO_2 methods, the iodine absorption method has very rarely been used to determine the biochar surface area (Chintala et al., 2013), possibly be due to the inapplicability of iodine absorption method for biochar surface determination. The method was originally developed for surface area determination in organic matter-rich soils and activated C (ASTM D4607-94, 2006). The test method of ASTM D4607-94 (ASTM D4607-94, 2006) explains the detailed methodology of iodine absorption.

Density and porosity

Biochar bulk density is a crucial physical property that has attracted significant attention. Lehmann and Joseph (2009) discussed two important biochar densities: the solid density and the bulk density. Biochar densities are also expressed as skeletal density (solid density) and envelope density (bulk density) (Brewer et al., 2014). Solid density is the ratio of solid volume to weight and it is related to the biochar C structure and its degree of packing. Brewer et al. (2009) reported a solid density of 1.5–1.7 g cm^{-3} in biochar produced from slow and fast pyrolysis at 500 °C and gasification at 730–760 °C. This is lower than the solid density value for graphite, which was recorded to be 2.25 g cm^{-3} (Brewer et al., 2014). Bulk density is the ratio of total volume to weight. Biochar bulk density decreases as the pyrolysis temperature increases because of the development of new pores (Brewer et al., 2014; Lehmann and Joseph, 2009) and has very low values due to the high porosity and low particle weight. Brewer et al. (2011) observed a bulk density of 0.22–0.65 g cm^{-3} in biochar produced from grass and wood by slow pyrolysis at a temperature range of 350–700 °C. Wood biochar showed higher bulk density than grass biochar due to its higher particle weight (Brewer et al., 2011). Biochar bulk density and solid density can also be determined using mercury and helium displacement methods with mercury porosimetry and quantachrome stereopycnometry, respectively (Brewer et al., 2014; Pastor-Villegas et al., 2006, 1993). Both of these measurements can be used to determine the porosity of biochar (Brewer et al., 2014).

Particle size distribution

Biochar particle size distribution plays a major role in biochar physicochemical properties. It is a key factor affecting the soil structure, water holding capacity, water movement, soil fertility, and microbial communities. Biochar particle size distribution is also an important factor in contaminant adsorption and building construction.

The IBI (2015) provided sieve size details to determine the particle size distribution of biochar when it is used for soil amendment; that is, progressive dry sieving of biochar with 50, 25, 16, 8, 4, 1, and 0.5 mm sieves. Moreover, it provided instructions about the percentage calculations of nine different particle sizes based on these seven sieves. Yargicoglu et al. (2015) used the ASTM D422 (ASTM D422-

63, 1998) method to determine the particle size in biochar; this method was originally developed for determining soil particle size. ASTM D422 (ASTM D422-63, 1998) recommends specific sieve sizes of 75, 50, 37.5, 25, 19, 9.5, 4.75, and 2 mm. Since ASTM D422-63 was specifically developed for soil, many pre-treatment steps are involved in separating bound soil particles, but those steps are not relevant in biochar analysis. ASTM D422-63 also provides a hydrometer procedure to determine the particle size distribution in soil, which is not suitable for biochar because it does not involve apparent downward movement through a water column. In addition, some authors have used their own methods for particle size distribution analysis based on their specific objectives. For example, Jaafar et al. (2015) used 4, 2, 1, and 0.5 mm sieves for the analysis of woody biochars to compare the effects of particle size on soil microbial colonization and biomass. The sieving method can have some errors because some particles can be lost due to retention in the sieve and/or by being blown away during the sieve motion. A laser technique (laser diffraction particle size analyzer) has also been used to determine the biochar particle size distribution (Abdullah and Wu, 2009; Ibrahim et al., 2017), which is more accurate and can provide more detailed results about the particle size distribution.

Biochar surface analysis

The surface properties of biochar are dictated by its functional group chemistry (e.g., phenolic, carboxylic, aliphatic, amine, and aromatic functional groups) and directly influence its interactions with other surfaces and chemicals (Li et al., 2013). Characterizing the surface functional groups of biochar is imperative to understand the mechanisms that occur during pyrolysis. Moreover, understanding the surface functional groups of biochar is important for the application of biochar for reducing greenhouse gas (GHG) emissions and contaminants in soil and water (Mandal et al., 2016b). Surface properties of biochar greatly depend on the feedstocks and production conditions (Meyer et al., 2011). Some of the more common surface analysis techniques for biochar are discussed in the next sections.

Scanning electron microscopy (SEM)

SEM is used to identify the morphological differences in biochar surfaces. It provides information about the mesopore and micropore distributions as well as the arrangement of pores (Yan et al., 2013). SEM equipped with energy dispersive X-ray spectroscopy (EDX) is commonly used to estimate the elemental composition of biochar surfaces (Rajapaksha et al., 2015). The abundant elements on the biochar surface can be identified and mapped by SEM-EDX analysis (Inyang et al., 2012; Yao et al., 2011; Zhang and Gao, 2013). Therefore, SEM-EDX analysis has been employed to characterize the distribution of elements on biochar surfaces prior to biochar applications (Chia et al., 2012; Fellet et al., 2014). Many researchers have used SEM-EDX to map the elements on the biochar surface after inorganic contaminant adsorption studies (Inyang et al., 2012; Yao et al., 2014, 2013a),

SEM-EDX is not applicable for organic contaminants. The efficiency of biochar for specific element adsorption and the involvement of biochar surface elements in adsorption processes can be determined via elemental mapping with SEM-EDX. As an example, Moon et al. (2013) reported lead (Pb) association with Al, silicon (Si), and O on the biochar surface after a contaminated soil sample was incubated with soybean stover biochar at 700 °C. Similarly, metal (e.g., Pb, cadmium (Cd), arsenic (As), and zinc (Zn)) adsorption on biochar in aqueous systems was evaluated by SEM-EDX analysis (Beesley and Marmiroli, 2011; Lu et al., 2012). Michálešková-Richveisová et al. (2017) modified biochar produced from different feedstocks (i.e., corn cobs, garden wood waste, and wood chips) at 500 °C by a post-treatment with aqueous ferric nitrate ($Fe(NO_3)_3$). They observed the Fe distribution on the biochar surface by SEM-EDX analysis.

X-ray photoelectron spectroscopy (XPS)

The surface analysis of biochar elements can also be performed with XPS. XPS analysis provides information about the chemical compounds and chemical bonds on the biochar surface to a surface depth <10 nm (Lin et al., 2013; Yao et al., 2010; Zhang et al., 2013). Moreover, XPS can be used to determine the relative abundance of different species of defined elements on the surface (Castle, 1990; Vithanage et al., 2015). Lin et al. (2013) used XPS to analyze the bonding states of C and N in biochar enhanced with chicken litter, clay, and minerals. They identified different C (i.e., C-C, C-H, C = O, and -COOH) and N (i.e., N-C, amino acid N, and ammonium-N) bonds on the biochar surface and assessed the soil N availability following biochar application. Sorrenti et al. (2016) analyzed the surface elemental composition of biochar aged in the soil for four years by XPS. Biochar association with organic and inorganic compounds in the soil (i.e., the percentages of O, N, Na, Al, Si, manganese (Mn), and iron (Fe)) was higher on the aged biochar surface compared to fresh biochar. XPS is a powerful technique that can be used to characterize the biochar surface before and after application in different disciplines (Lin et al., 2013; Manyà, 2012; Yao et al., 2013b; Zhou et al., 2013). However, in XPS, data acquisition is performed at a particular point of the sample. Since biochar is a heterogeneous mixture of materials, the results of XPS analyses always rely on the detection point (Castle, 1990; Chia et al., 2012).

Böehm titration

Böehm titration is used to evaluate the acidic and basic functional groups present on the biochar surface. Böehm titration is primarily used to identify the oxygen-containing surface groups of C materials (Böehm, 1994; Goertzen et al., 2010; Salame and Bandosz, 2001). These oxygen-containing functional groups have different acidities. Hence, bases with different strengths are used to neutralize and quantify each acidic group. Sodium hydroxide (NaOH) is used to neutralize all acidic groups, including phenols, lactonic groups, and carboxylic acids, whereas

sodium carbonate (Na_2CO_3) and sodium bicarbonate ($NaHCO_3$) are used to neutralize carboxylic and lactonic groups and carboxylic acids, respectively (Böehm, 1994). Moreover, hydrochloric acid (HCl) is used to determine the surface basicity of biochar during Böehm titration (Ahmad et al., 2013b). Böehm titration induces several types of interference during the analysis (Goertzen et al., 2010). Pre-treatment is essential if biochar samples contain a high amount of inorganic acidic and basic compounds because these compounds can dissolve, resulting in an overestimation of the functional groups (Tsechansky and Graber, 2014). Moreover, to avoid the CO_2 dissolving effect, titration needs to be performed after degassing with N_2 or argon (Ar) for 2 hr; degassing should be continued during titration (Goertzen et al., 2010).

Molecular/structural characterization of biochar

Structural characterization of biochar has been extended to investigate and characterize the structural stability and structural arrangement.

Thermal gravimetric analysis (TGA)

Biochar structural stability assessment is mainly performed using TGA (Mimmo et al., 2014). TGA is used to detect the temperature-induced weight loss patterns of biochar at the desired heating rate under controlled atmospheric conditions (Yan et al., 2013; Zhang et al., 2012). For example, helium (He) (Mimmo et al., 2014), N_2 (Yan et al., 2013; Zhang et al., 2012), and air (Inyang et al., 2014; Yao et al., 2013b; Zhang et al., 2012) can be used as heating atmospheres. Biochar heating normally starts at room temperature and increases to 1,000 °C (Mimmo et al., 2014); however, some researchers have heated biochar to less than 1,000 °C (Yao et al., 2013b). Commonly used heating rates are 10 K/min, 10 °C/min, and 20 °C/min (Ghani et al., 2013; Inyang et al., 2014; Yan et al., 2013; Zhang et al., 2012). The weight loss patterns of different biochars differ according to their structural stability. Biochar contains hygroscopic moisture, released from the biochar matrix at temperatures between 100 and 200 °C. Volatile matter degradation can occur from 200 to 600 °C or higher, depending on the structural stability of biochar (Mimmo et al., 2014). A lower temperature (<400 °C) produces a lower biochar structural stability than a higher temperature (>400 °C) because, as the temperature increases, the organic C contained in the biochar is arranged more systematically with high aromatic structures (Ahmad et al., 2014b).

X-ray diffraction (XRD)

XRD is usually implemented for biochar composition analysis (Inyang et al., 2012; Zhou et al., 2014). XRD can be applied to determine the crystalline C and other materials in biochar. Moreover, XRD spectra provide details about organic compounds such as lignin, cellulose, hemicelluloses, and inorganic compounds (e.g.,

carbonates, oxides, sulfides) (Nanda et al., 2013; Usman et al., 2015; Yan et al., 2013; Zhang et al., 2013). Yao et al. (2014) used XRD to analyze engineered biochars produced from three different feedstocks (i.e., bamboo, bagasse, and hickory chips) by the incorporation of montmorillonite and kaolinite. The presence of montmorillonite in the biochar matrix was identified by peaks at 6.4° (d = 13.840 Å), 6.9° (d = 12.803 Å), 19.9° (d = 4.449 Å), and 35.1° (d = 2.555 Å) in the XRD spectra; however, they did not observe kaolinite in the biochar. Yao et al. (2013a) also used XRD to analyze the P sorption of Mg-enriched engineered biochar. XRD spectra showed the precipitation of P on biochar surface associated with Mg. Although XRD is not usually used for quantitative analysis of compounds, it is an excellent tool to investigate the biochar structure.

X-ray fluorescence spectroscopy (XRF)

Researchers have also used XRF to quantify the compounds in biochar (Abdel-Fattah et al., 2015; Carrier et al., 2013). Compared to XRD, XRF is more powerful for identifying inorganic materials in the biochar structure and particularly useful for analyzing the ash composition of biochar (Bjeoumikhov et al., 2004). For instance, Abdel-Fattah et al. (2015) reported the inorganic composition of biochar by XRF analysis and showed the highest percentages of calcium oxide (CaO), potassium oxide (K_2O), and ferric oxide (Fe_2O_3) (i.e., 27.7, 1.87, and 1.60%) among the 18 inorganic compounds identified. However, XRF is seldom used in biochar studies, possibly because of its relatively high analysis cost (Table 3).

Fourier transform infrared (FTIR) spectroscopy

FTIR spectroscopy is a widespread vibrational technique used to investigate the chemical functional groups (e.g., aliphatic or aromatic nature) and mineralogy of biochar (Ascough et al., 2010; Inyang et al., 2012; Mukome et al., 2013; Nanda et al., 2013; Usman et al., 2015; Wu et al., 2012; Yan et al., 2013; Zhou et al., 2013). FTIR analysis with different modes and methods has been used by many researchers to reveal the changes and the degree of carbonization of biochar (Chia et al., 2012). Fourier transform mid-infrared photoacoustic (FTIR-PAS) spectroscopy is one such FTIR method, where the sample is placed in a photoacoustic cell and purged with He before analysis (Brewer et al., 2009). In diffuse reflectance infrared Fourier transform (DRIFTS) spectroscopy, the absorbance mode is generally used and the biochar sample is mixed with potassium bromide (KBr) to form a pellet (Novak et al., 2010). The biochar sample is placed in direct contact with the attenuated total reflectance crystal (ATR) in attenuated total reflectance-Fourier transform infrared (ATR-FTIR) spectroscopy (Keiluweit et al., 2010). One advantage of ATR-FTIR is that it is non-destructive. Synchrotron-based Fourier transform infrared (SR-FTIR) spectroscopy is an advanced FTIR method that is frequently used in the transmittance mode with a KBr pellet (Chia et al., 2012; Lehmann et al., 2005). SR-FTIR has also been used to study the interactions between biochar

Table 3. Biochar surface and molecular characterization methods used by researchers in different disciplines.

Applications	Surface and functional group characterization	Molecular characterization	Reference
Contaminant removal in aqueous solutions			
Inorganic and organic contaminants	SEM, XPS	XRD	(Zhou et al., 2014)
Methylene blue	SEM-EDX, XPS	XRD	(Yao et al., 2014)
Cadmium	FTIR KBr method	–	(Kim et al., 2013)
Phosphate	SEM-EDX, XPS	XRD	(Yao et al., 2013a)
Atrazine	FTIR KBr method	–	(Zhao et al., 2013)
Arsenic, methylene blue, and phosphate	SEM-EDX	XRD	(Zhang and Gao, 2013)
Heavy metals	SEM-EDX, FTIR-ATR	XRD	(Inyang et al., 2012)
Heavy metals	FTIR (method not mentioned)	–	(Park et al., 2016)
Phosphate	SEM-EDX, FTIR KBr method	XRD	(Yao et al., 2011)
Heavy metals	SEM, FTIR KBr method, XPS	–	(Zhou et al., 2013)
Methylene blue	Tapping mode AFM, SEM-EDX	XRD	(Zhang et al., 2012)
Cationic methylene blue	SEM, FTIR (method not mentioned)	XRD	(Sun et al., 2013)
Fe(II)	SEM, FTIR	XRD	(Usman et al., 2015)
Trichloroethylene	SEM, FTIR (method not mentioned)	–	(Ahmad et al., 2012a)
Trichloroethylene	SEM, FTIR (method not mentioned)	–	(Ahmad et al., 2013b)
Sulfamethazine	SEM-EDX, FTIR (method not mentioned)	–	(Rajapaksha et al., 2014)
Sulfamethazine	FTIR (method not mentioned)	–	(Rajapaksha et al., 2015)
Iodine	–	XANES, solid-state NMR	(Choung et al., 2013)
Biochar characterization and application in other disciplines			
Characterization of biochar after acid hydrolysis	SEM	Solid-state NMR	(Melligan et al., 2012)
Characterization of enhanced biochar	SEM-EDX, TEM, XPS	–	(Lin et al., 2013)
Characterization of biochar	FTIR KBr method	Solid-state NMR	(Rutherford et al., 2012)
Characterization of biochar	FTIR-ATR	–	(Mimmo et al., 2014)
Catalyst for the conversion of syngas to hydrocarbon	SEM, TEM, FTIR (method not mentioned)	XRD	(Yan et al., 2013)
Catalyst for the ozonation of water	FTIR KBr method	–	(Moussavi and Khosravi, 2012)
Characterization of magnetic biochar	SEM-EDX, TEM, XPS	XRD	(Zhang et al., 2013)
Assessment of geochemical weathering of a mineral ash-rich biochar	XPS	XRD	(Yao et al., 2010)
Characterization of biochar produced from corn stover	SEM, FTIR KBr method	–	(Lee et al., 2010)
Characterization of rice straw-derived biochar	FTIR KBr method	XRD, solid-state NMR	(Wu et al., 2012)
Characterization of biochar for field studies	FTIR equipped with photoacoustic detector	Solid-state NMR	(Brewer et al., 2011)
Characterization of fast pyrolysis biochar	–	XRF	(Carrier et al., 2013)
Characterization of biochar produced from rubber wood saw dust	SEM, FTIR (method not mentioned)	XRD	(Ghani et al., 2013)

Characterization of biochar related to nitrous oxide emission from soil	FTIR KBr method	–	(Wang et al., 2013)
Characterization of biochar from fast pyrolysis and gasification	FTIR equipped with photoacoustic detector, SEM	Solid-state NMR	(Brewer et al., 2009)
Molecular characterization of plant biomass-derived biochar	FTIR-ATR	NEXAFS	(Keiluweit et al., 2010)
Characterization of biochar	FTIR (method not mentioned)	–	(Angın, 2013)
Characterization of black carbon	Synchrotron-based FTIR (method not mentioned)	NEXAFS	(Lehmann et al., 2005; Mukome et al., 2014)
Stabilization of Cr in leather wastes by biochar	–	XANES	(Wells et al., 2014)

and minerals to evaluate the potential mechanisms of biochar binding and stabilization in soils (Mukome et al., 2014). The use of FTIR, including SR-FTIR, to evaluate biochar is discussed in detail in a review by Parikh et al. (2014).

Raman spectroscopy

Raman spectroscopy is another method used to evaluate the structural characteristics of biochar (Jamieson et al., 2014; Mukome et al., 2013). However, the utility of Raman spectra is diminished due to the influence of fluorescence that occurs during the analysis, especially by graphitic and polycyclic aromatic compounds in biochar (Chia et al., 2012). Various researchers have used Raman spectroscopy to evaluate the biochar functional groups and amorphous and graphite structures (Inyang et al., 2012; Nanda et al., 2013; Vithanage et al., 2015). Jorio et al. (2012) studied the C nanostructures in Terra Preta de Indio soils and in biochar with Raman spectroscopic analysis to evaluate crystalline C structures. The in-plane crystallite size distribution (La) was estimated using the ratio of D and G band intensities (I_D/I_G) (Cançado et al., 2007) of C nanostructures in Terra Preta de Indio soils and biochar. Their findings revealed that the distribution of La in charcoal from the Terra Preta de Indio soils was 3–8 nm, while that from biochar was 8–12 nm. The D and G bands of the Raman spectrum represent the breathing mode sp^3 C in disordered graphite rings, and sp^2 C in ring structures and C = C bonds, respectively (Parikh et al., 2014). Hence, the ratio of I_D/I_G can be used to evaluate the amorphous and crystalline C structures in biochar (Mukome et al., 2013; Vithanage et al., 2015; Wu et al., 2009). The use of Raman spectroscopy to evaluate biochar structures was discussed in detail by Wu et al. (2009).

Raman spectroscopy is a powerful vibration technique that can be used to investigate biochar. It has higher sensitivity, lower interference, and minimal sample preparation requirements compared to FTIR. However, Raman spectroscopy is not as widely used as FTIR, likely because of its higher instrument cost.

Solid-state nuclear magnetic resonance (NMR)

NMR is another spectroscopic technique commonly utilized to investigate the structural composition of biochar (Choung et al., 2013; Melligan et al., 2012; Wu et al., 2012). NMR spectroscopy is used to determine the concentrations of aliphatic and aromatic hydrocarbons in a biochar matrix. Hence, the degree of carbonization and stability in different biochars can be compared using the NMR results (Jindo et al., 2012; Melligan et al., 2012). Moreover, NMR spectroscopy provides details on the functional groups (e.g., aliphatic, aromatic, phenolic, and methoxyl) and hydrocarbons, similar to FTIR spectroscopy. However, NMR spectroscopy is more suitable for analyzing biochar containing C compounds than it is for determining functional groups. Hence, researchers have used both NMR and FTIR in their studies to examine the biochar structure and functional groups (Brewer et al., 2011; Rutherford et al., 2012; Wu et al., 2012). NMR spectroscopy

has limitations in high-temperature pyrolyzed biochar analysis owing to the low signal/noise ratio. Another limitation is that if ferromagnetic minerals are present, the NMR signals can be masked (Begaudeau et al., 2012).

Near-edge X-ray absorption fine structure (NEXAFS) spectroscopy

NEXAFS is a synchrotron-based method. Very few laboratories (~70) around the world support this type of spectroscopy (lightsources.org, 2016). Consequently, NEXAFS is not commonly applied and its capabilities in biochar characterization remain largely unknown. NEXAFS is an element-specific approach, which has been effectively used to characterize highly variable and complex types of C materials such as charcoal (black carbon) particles (Lehmann et al., 2005; Liang et al., 2010). NEXAFS has been successfully used for the identification of C species in biochars with various structures produced at different pyrolysis temperatures (100 and 700 °C) (Heymann et al., 2011; Keiluweit et al., 2010). Biochar stability (aromatic and aliphatic C) and biochar associations in soil have been effectively studied using NEXAFS (Heymann et al., 2011; Keiluweit et al., 2010). For example, NEXAFS data of biochars (walnut shell, softwood) that were incubated with and without a silt loam soil showed increased aromaticity with aging, as evident by the change in the ratio of the aromatic peak (285.3 eV) to the aliphatic peak (287.1 eV) and the presence of a C = O peak (288.6 eV) after incubation (Mukome et al., 2014). These data reflect the changes in the biochar structure resulting from degradation and interactions with soil minerals. NEXAFS has also been used to determine the surface chemistry of black C materials (Heymann et al., 2011).

X-ray absorption fine structure (XAFS) spectroscopy

XAFS is also a synchrotron-based method. Application of XAFS spectroscopy has partially transformed our understanding of delineating the metal(loid)-biochar interactions in soil, sediments, and water settings. XAFS spectroscopy is divided into X-ray absorption near-edge structure (XANES) spectroscopy and extended X-ray absorption fine structure (EXAFS) spectroscopy. XANES spectroscopy has been used as a fundamentally important tool to differentiate between the oxidation states of redox-sensitive heavy metal(loid) species, such as arsenic (arsenic(III)/arsenic(V)), chromium (chromium(III)/chromium(VI)), and selenium (selenium(III)/ selenium(IV)), within a wide range of environmentally-relevant situations (i.e., the pre-edge region of the XAFS spectra is used in XANES analysis). Alternatively, EXAFS spectroscopy has been applied to delineate symmetry and identify the coordinating ligand environment. It can also provide information about more distant neighboring atoms in environmental media (soil, sediments, or biochar). For example, in a sample where Zn is absorbed in an iron oxide mineral, the Zn K-edge EXAFS spectrum can be used to determine whether Zn is four- or six-fold coordinated. It can also show whether zinc is absorbed as an outer-sphere complex or is directly coordinated at the mineral surface as an inner-sphere complex (i.e.,

the post-edge region of the XAFS spectrum is considered for EXAFS analysis). The following paragraphs summarize the research conducted on the application of XAFS spectroscopy for examining biochar-metal(loid) interactions.

Recently, Ahmad et al. (2016) used XAFS spectroscopy to investigate the solid-phase speciation of Pb and copper (Cu) in shooting range soils amended with different biochars (i.e., soybean stover, pine needle). They found that Pb was mainly present in the form of Pb adsorbed to humic acid (22%), hydrocerussite (19%), and gibbsite (17%) in the soybean biochar-amended soil. Similar species were observed in the pine needle biochar-amended soil with different proportions of Pb adsorbed on gibbsite (37.4%), hydrocerussite (24.3%), and humic acid (28.3%). Rajapaksha et al. (2015) applied EXAFS spectroscopy to elucidate the different solid phases of Pb, antimony (Sb), and Cu in contaminated soils from a shooting range amended with biochars, nanomaterials, and natural Fe oxides. They revealed that Pb was bound to phosphate (to form stable chloropyromorphite ($Pb_5(PO_4)_3Cl$)) in the biochar-amended soils and such species of Pb were not found in the soils amended with nanomaterials or natural Fe oxides. Additionally, the XANES data of Cu and Sb did not show the formation of stable species after biochar amendment, indicating the significance of solid-phase speciation in determining the potential fate and risk of these metal(loid)s in amended soils. In a laboratory batch-type study, the efficiency of meat and bone meal (MBM) biochar was evaluated for the immobilization of Zn, whereby Zn speciation was carried out using synchrotron-based Zn K-edge EXAFS spectroscopy (Betts et al., 2013). These researchers revealed that Zn was associated with phosphate groups in a monodentate inner-sphere fashion (via surface complexation) under tested conditions. Thus, phosphate in the biochar enabled stable Zn-phosphate precipitates. Wagner et al. (2015) provided direct evidence of precipitation of Zn with phosphate as Zn-phosphate ($Zn_3(PO_4)_2$) species using phosphorus K-edge XANES spectroscopy in field soils amended with biochar. They also highlighted the significant role of XANES analysis to delineate the sequestration mechanism of Zn in biochar-amended soils and to predict the long-term fate of Zn in such soils.

Choung et al. (2013) compared the uptake of emerging contaminants, i.e., iodine species, by black C and commercial humic acid in batch sorption experiments in aqueous solutions; they also determined the solid-state speciation by applying XAFS spectroscopy. Their XAFS data demonstrated that iodide was transformed into electrophilic species that were chemically (covalently) associated with C atoms in polycyclic aromatic hydrocarbons made up of black carbon biomass. Wells et al. (2014) determined the chromium (Cr) speciation in biochar produced from chrome-tanned leather (i.e., waste from the tanning industry). XANES analysis showed that leather and biochar contain Cr as a mixture of Cr sulfate and Cr carbide. Interestingly, the proportion of Cr as carbide increased from 0% for untreated leather to 88% for biochar formed at 1,000 °C. Such information about the mechanism of biochar-metal interactions is vitally important for determining the actual fate of toxic metal(loid) species in biochar-amended soils and sediments, as well as for developing suitable and metal(loid)-specific remediation strategies.

XANES and EXAFS (as discussed above) have been applied as valuable tools for examining biochar-metal(loid) interactions in soil, sediment, and aquatic conditions. Future research is warranted to resolve the solid-phase speciation of different metal(loid) species in environmentally relevant multi-contaminated soil and water settings, where biochars can be applied for amendment. Micro-XANES and micro-EXAFS imaging techniques have been relatively unexplored and should be considered in the future to determine the micro-scale spatial distribution of metal(loid)s in biochar-amended soils or at the biochar-metal(loid) interface in aqueous solutions.

Possible applications of biochar properties

Soil application for agronomic benefits represents the primary use of biochar. However, the fundamental mechanisms by which biochar can provide a significant beneficial function to soil and the environment are poorly described in terms of providing the required predictive capacity. Biochar can act as a remediation agent by reducing the pollutant (GHG, heavy metals) concentrations in soils and the environment. Biochar can also be used in other areas such as electrical appliances, construction materials, and microbial carrier materials (Qian et al., 2015). A greater understanding of biochar properties is needed to utilize biochar in areas described in the following sections.

Reducing greenhouse gas (GHG) emissions

Scientists have tested biochars for reducing GHG emissions from soils and sediments (Awad et al., 2012; Clough et al., 2010; Yanai et al., 2007; Zhang et al., 2010). The porous structure, surface area, and selective surface functional groups of biochar can change the water retention capacity in soil to modify GHG emissions, especially non-CO_2 species, by altering the O_2 availability in soil (Ayodele et al., 2009; Busch et al., 2013; Singh et al., 2010; van Zwieten et al., 2010). The changes in the soil microbial community structure and abundance following biochar incorporation also influence the GHG emissions from soils (Lehmann et al., 2011; Liang et al., 2010; Mandal et al., 2016a). However, the mechanisms of biochar for the reduction of GHG emissions are still under speculation; they may be related to the inherent properties of biochar or to changes in the soil microbial community (Martin et al., 2015). On the other hand, production of biochar from feedstock reduces the biodegradation of organic materials and mean residence time can be extended >1,000 yr, which help to reduce the CO_2 emissions to the atmosphere (Glaser et al., 2009).

Potential interactions with the soil microbial community

Biochar can influence the activity, dynamics, and population of a soil microbial community (Spokas and Reicosky, 2009). Microbial responses to biochar depend

on the feedstocks and pyrolysis temperature (Pietikainen et al., 2000; Steiner et al., 2004). Applying biochar to soil can modify the symbiotic relationships that exist in the rhizosphere zone, which is between plants and microbes, located in close proximity to the root zone (Anderson et al., 2011). The roles of microbial and rhizosphere secretions in the presence of biochar materials have not been established (Anderson et al., 2011). The microbial community size and activity increased in different soils with more biochar applied (Wardle et al., 2008). Pietikäinen et al. (2000) observed microbial colonization on biochar surface and Hamer et al. (2004) reported biochar mineralization after the samples were incubated with bacteria. Shneour (1966) also reported microbial-mediated biochar mineralization. However, there is contradicting evidence concerning biochar stability in soils (Bruun et al., 2008; Kaal et al., 2012; Zimmerman, 2010). The conflicts on high stability, soil organic matter accumulation, and enhanced soil microbial activity can be resolved by surface characterized biochar applications. In addition, likely indirect impacts on soil microbial populations are caused by porosity, habitats, and labile C and nutrients provided by the biochar.

Microbial inoculant carrier

Biochar has been effectively used as an inoculant carrier for many microbial species, including *Azotobacter, Bacillus, Clostridium, Frankia, Pseudomonas*, and *Rhizobium* (Lehmann et al., 2011), and has shown a high survival rate similar to that of the conventionally used material, peat. The biochar chemical properties (i.e., N content (low C:N ratio and high N content) and pH (∼6-10)) are critical for initial inoculum survival; however, after incorporation into soils, the physical properties of biochar (e.g., surface area, pore diameter (macropores, pore opening ∼26–46 μm), and water-filled pore spaces) were more important than the chemical properties (Hale et al., 2015). Hence, biochar produced at relatively high temperatures (∼600 °C) serves as a more effective microbial inoculum carrier after incorporation of readily available nutrients (e.g., compost extract or supplemental nutrients) (Hale et al., 2015, 2014). Biochar is also considered to be a competitive inoculant carrier owing to its low cost, easy production, sustainability, and high stability (Lehmann et al., 2011).

Supercapacitor

A supercapacitor is an electrical energy storage device with capacitance values much higher than those of other capacitors. It can accept and deliver a charge faster than batteries (Winter and Brodd, 2004). Nanostructured C can be used to achieve highly stable, reversible electrical energy storage capacity and high power density in a supercapacitor (Gupta et al., 2015). Biochar showed promising results as an electrode in supercapacitors. Jiang et al.(Jiang et al., 2013) produced a highly-ordered biochar with a high carbon content (i.e., 98%) and large macro-porosity from cedar wood at 750 °C; this material showed a very high surface area

(i.e., >400 m^2 g^{-1}). They observed very fast charging-discharging behavior in the biochar electrode with a capacitance of ~14 F g^{-1}. Modification with diluted HNO_3 at room temperature increased the capacitance seven-fold (i.e., 115 F g^{-1}) owing to the increased number of –O containing functional groups on biochar surface. The modified cedar wood electrode processed at 750 °C was stable for >5,000 cycles without a decrease in capacitance. Similarly, Jin et al. (2013) also evaluated the use of biochar for supercapacitors. They produced biochar from a co-product of corn ethanol production (i.e., distiller's dried grains with solubles) at 950 °C in an N_2 atmosphere. Biochar was activated with potassium hydroxide (KOH; 1 g biochar and 0.075 mol KOH) during pyrolysis and with 4 mol L^{-1} HNO_3 (1 g biochar and 20 mL HNO_3) after pyrolysis. The produced biochar consisted of a highly systematic arrangement of C nanostructures with a very high surface area (2,959 m^2 g^{-1}) and pore volume (1.65 cm^3 g^{-1}). After activation with HNO_3, it showed a surface area, pore volume, and specific capacitance of 3,310 m^2 g^{-1}, 1.85 cm^3 g^{-1}, and 260 F g^{-1}, respectively. The capacitance performance of the biochar was higher than that of activated C. Hence, biochar has great potential as a low-cost supercapacitor electrode (W. J. Liu et al., 2015).

Dye-synthesized solar cell

Dye-synthesized solar cells are a group of thin-film solar cells based on the semiconductor formed between a photosensitized anode and an electrolyte. Electrodes used for this are very expensive, but they can be replaced with nanostructured C materials. Biochar with a high surface area, wider mesopores, and high conductivity can be used instead of expensive electrodes (Dincer et al., 2014). However, more studies are needed to determine the suitability of biochar in dye-synthesized solar cells.

Building sector

Biochar has also been used as a construction material. Biochar produced at low temperatures with low heating rates (e.g., 400 °C at 15 °C/min) and with small particle sizes (75 μm) have shown the highest performance as a binding material in asphalt mixtures (Zhao et al., 2014). Biochar reduces the high-temperature susceptibility of asphalt binders and enhances their corrosion resistivity. Biochar can also be used as a material for insulating buildings and regulating humidity owing to its very low thermal conductivity (Das et al., 2016) and its ability to absorb water six times its weight (Zhao et al., 2014). Zhao et al. (2014) analyzed asphalt mixtures incorporated with biochar produced from switchgrass at 400 and 500 °C. They observed reduced temperature susceptibility and increased resistance to rusting, moisture, and cracking in the binder. Biochar also showed better performances than C black or C fiber. This is one of the emerging disciplines of biochar applications; thus, future studies in this area are still needed.

Animal farming

When biochar is used in feed, litter, or in slurry treatments, less odor is observed compared to when additives are not used (McHenry, 2010). Biochar can also be used as a feed supplement to increase animal health. Hale et al. (2011) reported reduced bioaccumulation of organic pollutants in cows when biochar was mixed with feeds such as silage and hay. In addition, biochar helped incorporate animal dung into the soil, which led to improved nutrient cycling in soil (Joseph et al., 2015), increased production, and reduced the cost of production (Joseph et al., 2015). Biochar has the potential to (a) improve feed intake ability, (b) decrease allergies, (c) reduce stress in animals, (d) improve cattle health and appearance, e) increase milk protein and fat, (f) reduce the mortality rate, and (g) improve udder health (Gerlach and Schmidt, 2012).

Catalyst

Biochar can also be used as a catalyst in syngas cleaning. Usually, syngas cleaning for tars is performed with dolomite, zeolites, and metal-based catalysts (Bhandari et al., 2014). Recent studies revealed biochar's efficacy at removing tar from syngas, indicating that it is a promising alternative (Abu El-Rub et al., 2008; Bhandari et al., 2014; Wang et al., 2011). More interestingly, biochar can be used directly in syngas cleaning without active metal loading. The catalytic activity of biochar for tar removal in the syngas is mainly reliant on the pore size, surface area, and mineral content (Qian et al., 2015). Biochar has also been demonstrated to be effective as a catalyst in liquid fuel production from biomass. The Fischer-Tropsch synthesis clearly showed that biochar-based iron (Fe) nanoparticles were successful as a catalyst for conversion of syngas into liquid hydrocarbons (Yan et al., 2013). Biochar has also been used as a solid acid catalyst for biodiesel production. Moreover, biochar has been successfully used for transesterification of canola oil with alcohol and oleic acid (Dehkhoda et al., 2010). Observations by Dehkhoda et al. (2010) clearly indicate that the efficiency of biochar catalytic activity and reusability depends on the surface area and acid density of biochar in biodiesel production. The particle strength, hydrophobicity, and density of the sulfonic acid groups also govern the efficacy of biochar-based solid acid catalysts (Dehkhoda et al., 2010; Kastner et al., 2012).

Biogas production

Biogas is generally produced from the anaerobic digestion of raw bagasse, municipal wastes, and household wastes under batch conditions as a renewable energy production method (Kothari et al., 2010). Shen et al. (2016) used pine wood and white oak wood biochar as additives in biogas production from sludge under anaerobic digestion. Biochar additives increased the methane content in biogas by 92.3% and 79.0% for pine wood and white oak wood biochar, respectively. Those

two biochars also increased the CO_2 sequestration in biogas by 66.2% and 32.4% and inhibited NH_3 evolution during biogas production. Sunyoto et al. (2016) observed increased H_2 and CH_4 production by 31.0% and 10.0%, respectively, caused by the addition of biochar during anaerobic digestion of aqueous carbohydrates. Biochar also increased volatile fatty acid generation during H_2 and CH_4 production. Biochar acts as a temporary substrate for microbial growth and buffers the pH in the digester to enhance H_2 and CH_4 production (Sunyoto et al. (2016). Proper biochar characterization would be helpful in developing the use of biochar in biogas production.

Direct C and microbial fuel cells

The application of biochar leads to significant improvements and reduce production costs of direct C and microbial fuel cells (Ahn et al., 2013; Ganesh and Jambeck, 2013). Direct C fuel cells exhibited a power density of 60–70% that of coal-based fuels for the same cathode area and reactor volume; stirring at 200–500 rpm led to further improvements. Biochar has been used in microbial fuel cells as the anode (Ganesh and Jambeck, 2013), the cathode (Huggins et al., 2015), and both cathode and anode (Huggins et al., 2016). Biochar porosity, surface area, and pore size have been reported as important factors in both direct C and microbial fuel cells (Ahn et al., 2013; Qian et al., 2015). Biochar-based supercapacitors are also an emerging technology (Genovese et al., 2015; Jiang et al., 2013). Carbon-based materials with high surface area and porosity are used to produce supercapacitors; consequently, biochar produced from several feedstocks is used as a raw material in the development of supercapacitors (Basri et al., 2013; Farma et al., 2013; Goodman et al., 2013; Liu et al., 2012). Biochar showed increased capacity owing to the increased number of –O-containing functional groups (e.g., phenolic, carboxylic, ketone etc.) after surface modification with nitric acid (HNO_3) (Basri et al., 2013; Farma et al., 2013).

Slow-release fertilizer

Biochar can be applied in the fertilizer industry to produce slow-releasing fertilizers. The biochar produced from Mg-enriched biomass or by Mg-impregnation can be used to control the release of phosphate by precipitating phosphates through a chemical reaction with Mg and by surface deposition of phosphate on Mg crystals (Yao et al., 2013a). Manikandan and Subramanian (2013) developed a slow-releasing N fertilizer with biochar produced from *Prosopis juliflora* wood and urea and observed higher N availability in topsoils when biochar was incorporated into the soils. Future research in fertilizer industry should be conducted to incorporate biochar into fertilizer to increase the efficiency of the products.

Water treatment plants

Biochar can also be applied to wastewater treatment plants. Wastewater is generally composed of organic pollutants, micro-pollutants, and trace elements. Biochar applied to wastewater can adsorb trace elements and pollutants on its surface. Previous research has also reported that biochar with a high surface area and adsorptive capacity can reduce pollutants from wastewater and render the water acceptable for reuse (Hossain et al., 2010). The oxygen-containing surface functional groups (carboxyl, phenolic) of biochar can also adsorb heavy metals from wastewater and reduce contamination (Hossain et al., 2011, 2010). Inorganic contaminants such as nitrate and phosphate can be removed from water using biochar (Li et al., 2014; Rajapaksha et al., 2016).

Management of biochar properties

Biochars can be modified to obtain well-characterized surface properties for effective use. Researchers have advanced the designer/engineered/tailored biochar concept by modifying the biochar surface using physical, chemical, and pyrolysis programs to produce micro- to nano-sized materials. The purpose of this strategy is to improve the physicochemical and sorptive properties of biochars for novel uses such as heavy metal and organic pollutant sorption for water purification and soil remediation. Prior to modification, proper characterization is needed to identify the optimal properties for a particular application. Correct and appropriate evaluation of surface functional groups is critical for selecting biochars that will maximize the immobilization of heavy metals in soil and water (Inyang et al., 2016; Mandal et al., 2017). Simple physical methods, such as the selection of production technologies (e.g., slow pyrolysis, fast pyrolysis, and gasification), adjustment of pyrolysis temperature, modification of holding time, and selection of feedstocks, should be the first steps of modification before attempting any chemical methods. Figure 9 shows the differences in biochar ash, mobile matter, and fixed C in wood biochar produced with two different holding times. The wood biochar shows a significant difference in the contents of ash, mobile matter, and fixed C at a low temperature of 300 °C. However, there was no significant difference between the contents at temperatures >300 °C. Ronsse et al. (2013) also reported similar behaviors between straw, green wastes, and dry algae at the same pyrolysis conditions. Hence, biochar properties can be modified by changing the holding time at production temperatures as low as 300 °C. In addition, the production temperature is a crucial factor that needs to be considered when modifying the properties of biochar.

The feedstock can be treated either pre- or post-pyrolysis to gain desirable properties. Biochars can be treated to have a higher capacity for specific interactions with organic and inorganic chemicals because they have a high surface area, a high degree of physicochemical attraction between the adsorbate and C surface, and an appropriate pore size distribution. Biochar can be modified in different ways such as impregnation (guest elements are usually incorporated into biochar materials), oxidation

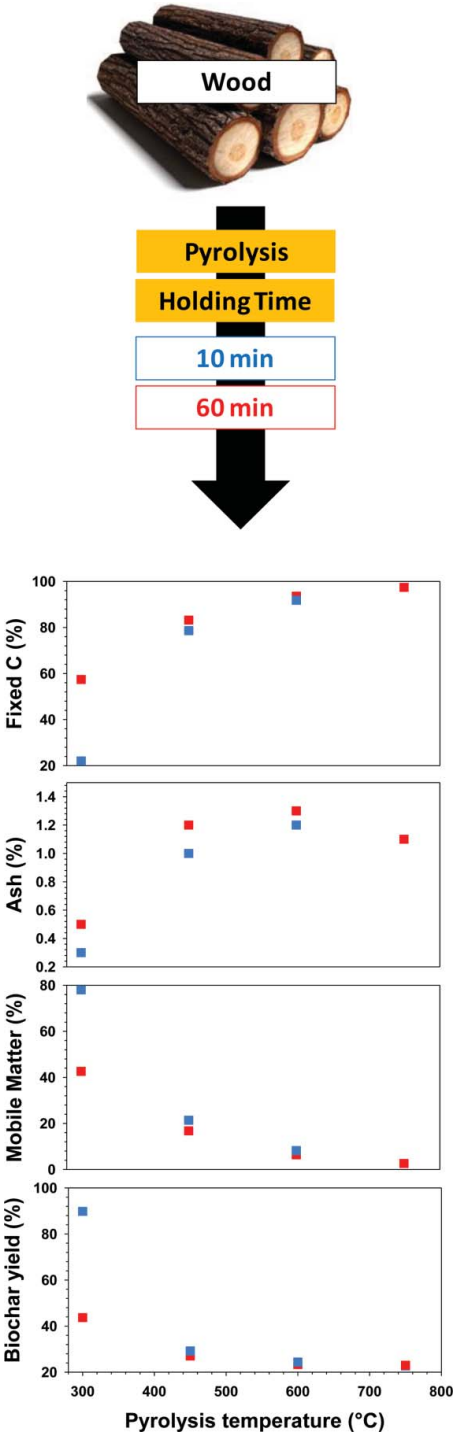

Figure 9. Differences in fixed C, ash, mobile matter/volatile matter, and biochar yields at two different holding times with different pyrolysis temperatures. Data obtained from Ronsse et al., 2013.

(liquid and gas phase oxidation where liquid or gaseous compounds are used to modify/purify chars), grafting (grafting of organic molecules onto the biochar surface), and with chemicals such as acids (HNO_3, HCl, phosphoric acid (H_3PO_4), and sulfuric acid (H_2SO_4)), amino acids, and potassium permanganate ($KMnO_4$) (Rajapaksha et al., 2016). All of these modifications can enhance the biochar surface properties, including surface area, pore volume, surface charge, pH, CEC, EC, nutrient-retention capacity, and effective surface functional groups (e.g., phenolic, hydroxyl, carboxyl, amine). A range of compounds/materials have been used for these modifications such as chitosan (Zhou et al., 2013), nano-sized zero-valent iron (Zhou et al., 2014), hydrogen peroxide (H_2O_2) (Xue et al., 2012), HNO_3, and nanocrystals (such as zinc oxide (ZnO), CaO, and ferric chloride ($FeCl_3.6H_2O$)) (Martinez-Hernandez et al., 2010).

Summary—Current status of biochar characterization

The chemical and physical properties of biochar can vary significantly, even when they are created from the same feedstock. The final product differs according to the production conditions (e.g., temperature, heating rate, holding time, and gas regents). Biochar production conditions can be controlled in the laboratory and on a commercial scale with advanced heating equipment to produce consistent materials. However, it can be difficult to control the production conditions with simple biochar production apparatus utilized by farmers. Potential applications of biochar are broad and rely on the specific properties of a given biochar. Not all types of biochars work well in any single discipline; for example, biochars produced at low temperatures (200–400 °C) commonly perform better as a soil amendment in agricultural applications than those produced at higher temperatures (>400 °C) because they increase the availability of plant nutrients and beneficial microbial populations in some soils. Biochar properties maintain constant values in consecutive productions from the same feedstock, although the production conditions are not foolproof, even with advanced instruments. Moreover, biochar weathering can occur during storage if the materials are not stored under airtight conditions. Hence, the characterization of biochar pre- and post-application is essential to yield the maximum benefits. Biochar characterization objectives and methods are not standardized and vary between studies. The IBI has provided some assessment methods for different biochar properties when biochar is employed as a soil amendment; however, researchers seldom apply the same methods and there is no published standard document from any other organization for the characterization of biochar in other disciplines. Moreover, biochar characterization methods have many weak points, which have not been appropriately evaluated and modified. Hence, the observed biochar properties seldom meet their potential and the comparison of biochar properties in different studies is not straightforward or logical, even when the production methods are similar. In addition, researchers seldom provide all of the details for the methods that they employ.

Table 4 summarizes the current perspective of the importance of biochar properties in different applications. The significance of biochar characterization is

Table 4. Summary of the important focal points of biochar characterization in different applications according to the existing knowledge.

	Biochar properties																
	1			2		3	4	5	6	7	8	9	10	11	12	13	14	15
Biochar applications	a	b	c	d	e													
Soil quality improvement	+	+++	+++	+++	+++	+++	+++	+	++	+	(+)	(+)	+++	(+)	+	(+)	+	+
Soil carbon sequestration	+++	+++	+++	+++	++	+++	+++	+	+	+++	(+)	(+)	++	(+)	+++	(+)	+++	+++
Soil and water remediation	+++	+++	+++	+++	++	+++	++	(+)	+++	+++	(+)	(+)	+++	++	+++	++	+++	+++
Gas adsorbent	nk	nk	nk	nk	nk	nk	nk	nk	nk	+++	+	+	nk	+++	+++	++	+++	nk
Catalyst in biofuel production	++	+++	+++	+++	+++	+	(+)	(+)	(+)	+++	+	+++	+	(+)	+++	++	+++	+++
Supper capacitor production	nk	+++	+++	+++	nk	(+)	(+)	nk	nk	+++	(+)	+	nk	(+)	+++	+++	+++	nk
Activated carbon production	+++	+++	+++	+++	++	(+)	(+)	(+)	(+)	+++	(+)	(+)	+++	(+)	+++	+	+++	+
Material in fuel cells	++	+++	+++	+++	(+)	(+)	(+)	+	(+)	+++	(+)	+	+	+++	+++	++	+++	+++
Microbial carrier material	+++	+++	+++	+	+	+++	+++	(+)	(+)	+++	+	(+)	++	(+)	(+)	(+)	(+)	(+)
Management of greenhouse gas emission	+	+++	+	+++	+	+++	nk	(+)	+	+++	(+)	(+)	nk	(+)	+++	nk	+	nk
Construction material	++	+++	+++	+++	nk	nk	nk	nk	nk	+++	+++	+++	+++	++	nk	nk	+++	+++

+, ++, and +++; analysis requirements are low, medium, and high, respectively.
Parentheses; not essential to analyze.
nk; not known.
1. Proximate analysis.
   a. Moisture.
   b. Mobile/volatile matter.
   c. Ash.
2. Ultimate analysis.
   d. C, H, O, N, S.
   e. Elemental analysis in ash.
3. pH.
4. Electrical conductivity (EC).
5. Exchangeable cations.
6. Cation-exchange capacity (CEC).
7. Surface area and pore size.
8. Bulk density.
9. Porosity.
10. Particle size distribution.
11. Surface morphology.
12. Surface functional groups.
13. Surface elements.
14. Structural stability.
15. Structural composition.

different for various applications. The level of importance of each biochar property is not comparable between disciplines and can even vary within the same discipline. Hence, biochar characterization must be performed based on the identified mode of action of the biochar in a specific discipline. Some biochar properties have been insufficiently clarified, have attracted inadequate attention, and/or have not been evaluated because of the analytical limitations in the applied fields. For example, the effects of biochar on heavy metal(loid) immobilization in soil have been studied in depth, but simultaneous changes in soil biological factors and nutrient status have not been considered in many studies. In addition, a large knowledge gap exists in the criticality of certain biochar properties in research areas such as gas adsorbents, supercapacitor production, direct and microbial fuel cells, management of greenhouse gas emissions, and construction materials.

Biochar characterization approaches have become more advanced due to new technologies and knowledge. However, the use of advanced technologies is always influenced by accessibility and economic viability. The management of biochar properties and biochar modification is emerging as an objective for the optimization of biochar applications. Hence, the accuracy of the quantified properties is very important for modification techniques. Furthermore, the selection of more sensitive techniques is essential for critical applications of biochar such as medicinal purposes, catalysts, supercapacitors, and microbial carrier materials. Results must be published with the exact biochar characterization methods.

Biochar is emerging as a very promising, environmentally-friendly material in a broad range of disciplines. Hence, it is necessary to develop standardized biochar characterization methods that contain the inputs of all scientists who have worked with biochar. A global forum that discusses biochar characterization would be helpful as an initial step.

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