# 1

# Carbonaceous Material Characterization

## 1.1 Material Characterization

The thermochemical conversion of biomass by means of processes such as torrefaction, pyrolysis, or gasification, produces a char-like material with properties that differ considerably from the original feedstock. Further processing in the form of chemical or physical activation continues to modify the properties of the carbonaceous materials produced. The following Sections (1.1.1–1.1.4) provide a description of the main properties needed to characterize carbon-based materials for applications of biochar production and energy conversion.

### 1.1.1 Thermophysical Properties

Thermophysical properties are directly related to the structure and composition of the carbon-based materials. There is a large volume of studies that have analyzed their variation due to the effects of thermochemical conversion processes (Balogun et al., 2018). These properties not only have an impact in the operating conditions of processing equipment but also affect transportation costs and pollutant emissions. The thermal and physical properties have a strong dependence on parameters such as moisture content and temperature (James, 1975; Skaar, 1988; Dietenberger et al., 1999; Zelinka et al., 2007); therefore, various models have been developed to represent them as a function of these factors. The most relevant properties include thermal conductivity, density, and specific heat. However, a more detailed description of properties for biomass can be found in Ross (2010), De Jong (2014), Goss and Miller (1992), and Gaur and Reed (1995).

*Voltage-Enhanced Processing of Biomass and Biochar*, First Edition. Gerardo Diaz.

### 1.1.2 Moisture Content

The moisture content is defined as the mass of moisture (water) divided by the ovendry mass of the sample (Goss and Miller, 1992). The moisture content can be obtained by placing the sample in an oven filled with inert gas at a temperature of 105 °C for 24 hours, and then applying Eq. (1.1) to calculate the fraction of moisture in the sample.

$$MC = \frac{m_{wet} - m_{dry}}{m_{dry}} \times 100\ (\%) \tag{1.1}$$

where $m_{wet}$ is the mass of the specimen at a given moisture content and $m_{dry}$ is the mass of the ovendry specimen.

### 1.1.3 Ultimate and Proximate Analysis

The description of the composition of carbonaceous materials is usually obtained by performing an *ultimate* and *proximate* analysis. The ultimate analysis provides the chemical composition of a material, providing information about the contents of carbon, hydrogen, nitrogen, oxygen (by difference), and sulfur of a dry sample on a weight basis (CHNOS analysis). In addition, the analysis provides the heating value of the sample, which provides the energy released due to combustion of the material. The high heating value (HHV) considers that water present in the sample as well as water formed during the combustion process are in liquid state. On the other hand, the low heating value (LHV) considers that water has not been condensed. The proximate analysis provides the information about moisture, volatile matter, fixed carbon, and ash content of a particular sample. A detailed description of these analyses can be found in De Jong (2014).

### 1.1.4 Dielectric and Electrical Properties

Dielectric properties are important for the study of storage and dissipation of electric energy in materials (Bain and Chand, 2017). A dielectric is an insulating material that is a very poor conductor of electric current. Wood and other types of biomass behave as dielectrics, but as high voltages are applied, breakdown can occur, which constitutes an irreversible change of the material that allows current to flow through it. The main dielectric properties for applications of thermochemical conversion are the relative permittivity and loss tangent. The relative permittivity (also called dielectric constant) describes the ability of a material to absorb and store energy from

an applied electric field (James, 1975), and it is defined as (Barker-Jarvis et al., 2001):

$$\epsilon_r = \frac{\epsilon}{\epsilon_0} = \epsilon_r' - j\epsilon_r'' \tag{1.2}$$

where $\epsilon_r$ is the relative permittivity, $\epsilon$ is the absolute permittivity of the material, $\epsilon_0 = 8.854 \times 10^{-12}$ (F/m) is the permittivity of vacuum, and $\epsilon_r'$ and $\epsilon_r''$ are the real and imaginary parts of the relative permittivity. The relative permittivity can be represented by a scalar for a perfectly insulating material subject to DC voltage, but for AC voltage it is represented as a complex number given by Eq. (1.2), that varies significantly with respect to the applied frequency. The loss tangent denotes the dissipation of electric energy of a material and is defined as:

$$\tan(\delta) = \frac{\epsilon_r''}{\epsilon_r'} \tag{1.3}$$

In addition, as biomass is subject to thermochemical conversion, the char-like material produced has a higher electrical conductivity (an electrical property) than the original feedstock. When an electric field is applied to a material and a flow of current is established, part of the energy is dissipated as heat. As the resistivity of the material decreases due to thermal decomposition, the electrical conductivity characterizes the ability of the material to conduct electricity.

## 1.2 Biomass

Biomass is composed of renewable organic material that comes from plants and animals.[1] The most common types of biomass used for power or heat generation come from forest or agricultural waste. Municipal solid waste (MSW) is not considered in this chapter due to the mixing of organic materials with other types of waste. The description of the dependency of biomass properties with respect to temperature and moisture is described in this section.

*Thermal conductivity*: The thermal conductivity corresponds to the intrinsic ability of a material to conduct heat. According to the *Handbook of Wood* (Ross, 2010), for wood with moisture contents below 25%, the thermal conductivity can be obtained with the expression:

$$k = G_x(B + Cx) + A \text{ (W/(m K))} \tag{1.4}$$

1 https://www.eia.gov/energyexplained/biomass/.

where $G_x$ is the specific gravity at moisture content $x$ (in %), and where $A = 0.018\,64$, $B = 0.1941$, and $C = 0.004\,064$, for $G_x > 0.3$, and a temperature around 24 °C, with $x < 25\%$.

*Specific heat*: The specific heat can be interpreted as the amount of heat required per unit of mass to raise the temperature of a sample by one degree (Sonntag et al., 1998). Its value as a function of temperature for *dry wood* can be approximated by the expression:

$$c_{p_{dry}} = 0.0131 + 0.003\,867T \text{ (kJ/(kg K))} \tag{1.5}$$

where the temperature $T$ is in Kelvin. For *wood* that contains a percentage $x$ of moisture, the specific heat can be calculated as:

$$c_{p_x} = \frac{c_{p_{dry}} + c_{p_w}\frac{x}{100}}{1 + \frac{x}{100}} + A_c \tag{1.6}$$

where $c_{p_w} = 4.18$ kJ/(kg K) is the specific heat of water and $A_c$ is a correction factor equal to:

$$A_c = x(b_1 + b_2T + b_3x) \tag{1.7}$$

where $b_1 = -0.061\,91$, $b_2 = 2.36 \times 10^{-4}$ and $b_3 = -1.33 \times 10^{-4}$.

*Electrical conductivity*: The electrical conductivity of wood depends on temperature and it can be approximated as (Skaar, 1988):

$$\sigma(T_L) = 371\,535.2 \times 10^{-3660/T_L} \tag{1.8}$$

where $\sigma$ has units of $(\Omega\,\text{m})^{-1}$. The dielectric constant can be estimated by a constant value of 800 (James, 1975; Dietenberger et al., 1999).

Tables 1.1–1.4 provide a list of properties for a number of biomass types.

## 1.3 Biochar

When biomass is heated to temperatures in the range between $200 < T < 800$ °C in conditions of limited or no oxygen supply, char formation exists (White and Dietenberger, 2001), and its properties change dramatically with respect to the original biomass feedstock. Biochar is a material produced with the intention of using it as soil amendment. However, in many published works, this material is still referred to as biochar even if it is produced for other purposes such as liquid or gas filtration or as construction material.

In this chapter, average properties of biochar produced at $T_{carb} = 800$ °C are summarized in Table 1.1. A more detailed summary of biochar properties can be found (Yang et al., 2017).

**Table 1.1** List of thermophysical properties

| Material | Specific gravity | $k$ W/(m K) (12% MC) | Specific heat[a] kJ/(kg K) |
|---|---|---|---|
| **Hardwoods**[b] | | | 1.63 |
| Ash (white) | 0.63 | 0.17 | |
| Birch (yellow) | 0.66 | 0.18 | |
| Elm (rock) | 0.67 | 0.18 | |
| Maple (sugar) | 0.66 | 0.18 | |
| Oak (red) | 0.65 | 0.18 | |
| Oak (white) | 0.72 | 0.19 | |
| **Softwoods**[b] | | | 1.63 |
| Cedar (Western red) | 0.33 | 0.10 | |
| Douglas-fir (coast) | 0.51 | 0.14 | |
| Pine (ponderosa) | 0.42 | 0.12 | |
| Reedwood (old growth) | 0.41 | 0.12 | |
| **Biochar (peach pits)** | 0.5 | 0.38 | 1.1 |
| **Graphite** | 1.78 | 113 | 0.72 |

a) Goss and Miller (1992).
b) Ross (2010).

**Table 1.2** List of dielectric and electrical properties

| Material | $\epsilon_r$ | $\tan(\delta)$ | $\sigma$ $(\Omega\ \mathrm{m})^{-1}$ |
|---|---|---|---|
| Douglas-fir[a] | 4.3 at 20 Hz | 0.033 at 20 Hz | |
| Douglas-fir[a] | 3.8 at 1 kHz | 0.024 at 1 kHz | |
| Douglas-fir[b] | 1.9 at 1 MHz | 0.023 at 1 MHz | |
| Oak[a] | 4.1 at 20 Hz | 0.028 at 20 Hz | |
| Oak[a] | 3.5 at 1 kHz | 0.028 at 1 kHz | |
| Oak[a] | 3.4 at 1 GHz | 0.076 at 1 MHz | |
| Wood (damp)[c] | | | $10^{-4}$ to $10^{-3}$ |
| Wood (ovendry)[c] | | | $10^{-16}$ to $10^{-14}$ |
| Biochar[d] | 42 | | $2.6 \times 10^{1}$ |
| Carbon (graphite)[c] | 10–15 at 1 kHz | | 2 to $3 \times 10^{5}$ |

a) Goss and Miller (1992).
b) https://www.rfcafe.com/references/electrical/dielectric-constants-strengths.htm.
c) https://www.thoughtco.com/table-of-electrical-resistivity-conductivity-608499.
d) Win and Nang (2017).

**Table 1.3** Ultimate analysis (weight fractions) and heating value of biomass analyzed in this work

| Material | C (%) | H (%) | O (%) | N (%) | LHV (kJ/kg) |
|---|---|---|---|---|---|
| **Biomass**[a] | | | | | |
| Hard wood shaving | 48.41 | 6.28 | 41.1 | 0.13 | 18 854.5 |
| Peach pits | 52.52 | 6.18 | 39.74 | 0.38 | 20 754.9 |
| Almond hulls | 44.31 | 5.64 | 40.13 | 1.06 | 18 040.5 |
| Grape pomace | 52.74 | 6.23 | 33.48 | 2.14 | 21 883.0 |
| Coffee ground | 56.13 | 7.16 | 27.03 | 2.53 | 23 771.7 |
| **Biochar** | | | | | |
| Ponderosa pine | 77.73 | 2.43 | 9.5 | 0.55 | — |
| Walnut shell | 78.86 | 3.48 | 13.7 | 0.64 | — |
| Peach pits[b] | 65.62 | 2.33 | 10.2 | 0.57 | — |
| Almond shell[c] | 50.5 | 6.6 | — | 0.21 | 18 200 (HHV) |
| Almond pruning[c] | 51.3 | 5.7 | — | 0.77 | 18 200 (HHV) |
| **Activated carbon** | | | | | |
| Paulownia wood[d] | 70.83 | 3.41 | 25.76 | — | 23 300 |
| Peach pits[b] | 63.17 | 0.10 | 1.44 | 0.32 | — |

a) Diaz et al. (2015).
b) Munoz-Hernandez (2018).
c) Chen et al. (2010).
d) Yorgun and Yildiz (2015).

*Thermal conductivity*: The thermal conductivity of biochar depends on temperature approximately as follows (Ragland et al., 1991; Parfen'eva et al., 2011):

$$k_L(T_L) = 0.0013T_L - 0.01 \text{ (W/(m K))} \tag{1.9}$$

*Specific heat*: The specific heat of biochar as a function of temperature can be found in Dupont et al. (2014).

*Electrical conductivity*: It has been reported that the electrical conductivity of wood has a strong dependence on temperature (Dietenberger et al., 1999). However, the electrical conductivity of dry wood is so low that it still remains a good electrical insulator at moderate temperatures. Nonetheless, when wood is heated, it becomes charcoal and the electrical conductivity increases by several orders of magnitude (Kwon et al., 2013; Parfen'eva et al., 2011; Kumar and Gupta, 1993). For instance, measured

**Table 1.4** Proximate analysis of biomass analyzed (weight fractions)

| Material | Ash (%) | Fixed carbon (%) | Moisture (%) | Sulfur (%) | Volatile matter (%) |
|---|---|---|---|---|---|
| **Biomass**[a)] | | | | | |
| Hard wood shaving | 4.03 | 7.53 | 5.72 | <0.01 | 7.53 |
| Peach pits | 0.72 | 11.9 | 36.73 | 0.46 | 50.7 |
| Almond hulls | 8.86 | 18.9 | 8.01 | <0.01 | 64.3 |
| Grape pomace | 5.29 | 18.5 | 8.97 | 0.12 | 67.2 |
| Coffee ground | 1.14 | 7.07 | 54.69 | 0.08 | 37.1 |
| Almond shell[b)] | 0.6 | 15.8 | 3.3 | — | 80.3 |
| Almond pruning[b)] | 1.2 | 15.9 | 10.6 | — | 72.2 |
| **Biochar** | | | | | |
| Peach pits[c)] | 21.24 | 54.98 | Dry basis | — | 23.78 |
| **Activated carbon** | | | | | |
| Paulownia wood[d)] | 2.63 | 77.27 | 2.30 | — | 17.80 |
| Peach pits[c)] | 35.0 | 59.96 | Dry basis | — | 5.04 |

a) Diaz et al. (2015).
b) Chen et al. (2010).
c) Munoz-Hernandez (2018).
d) Yorgun and Yildiz (2015).

at room temperature, the electrical conductivity of oven dry wood is of the order of $10^{-15}$ $(\Omega\ \mathrm{m})^{-1}$ (Dietenberger et al., 1999), whereas the electrical conductivity of biochar produced at $T_{carb} = 600\ ^\circ\mathrm{C}$ is roughly $7 \times 10^{-4}$ $(\Omega\ \mathrm{m})^{-1}$ (Kwon et al., 2013). The electrical conductivity of biochar depends on temperature as (Sugimoto and Norimoto, 2004; Kwon et al., 2013):

$$\sigma(T_L) = 64\ 565 \times 10^{-1000/T_L} \tag{1.10}$$

where the units of $\sigma$ are $(\Omega\ \mathrm{m})^{-1}$.

Tables 1.1–1.4 provide a list of properties for a number of biochar types.

### 1.3.1 Surface Area, Cation Exchange Capacity, and pH

As mentioned above, biochar is a material produced with the intention of using it as soil amendment. The ability of biochar to increase nutrient retention for plants is related to the *cation exchange capacity* (CEC) property. In

addition, as biomass is thermally decomposed, a higher fraction of hydrogen and oxygen are removed from the material as compared to carbon. Due to this, the overall carbon concentration increases and it is referred to as *carbon recovery*. From initial carbon fractions around 39%, it can be increased to values in the range between 60% and 85%.

Another important aspect is that low-pH organic matter in soil shows very low values of CEC (Lehmann, 2007). Therefore, biochar pH is another important property that can improve soil health. Finally, climate change increases the odds of worsening drought conditions in several parts of the world. Therefore, a material with adequate surface area can help to increase water-holding capacity. Depending on how it was produced, biochar can have a relatively large surface area that can potentially help in reducing the amount of irrigation water used for plants. One of the most common methods used to determine surface area of biochar is by determining its BET *surface area* utilizing the equation and methodology developed by Brunauer, Emmett, and Teller that utilizes $N_2$ at 77 K, where results are provided in $m^2/g$ of biochar. It is important to indicate that the surface area determined is related to the adsorption of the nitrogen molecule in the material, and thus, depends on the adsorbate utilized. For nitrogen at 77 K, the activation energy of adsorption is very high (greater than 40 kJ/mol) and thus there is restricted adsorption into narrow porous (diameter, $d < 0.5$ nm) requiring very long equilibrium times (weeks to years) (Marsh and Reinoso, 2006). Therefore, many studies use adsorption of carbon dioxide at 298 K based on the equation by Dubinin–Radushkevich (DR) to determine surface area ($m^2/g$) of highly porous biochar and activated carbon. These materials with high levels of microporosity are usually utilized for applications that require removing pollutants from liquid or gas streams.

There are other methods that refer to the ability of porous material, such as biochar or activated carbon, to adsorb a gas. For instance the ASTM D5742 standard is used to determine *butane activity* and provides the ratio (in %) between the mass of butane adsorbed and the mass of the original sample of biochar or activated carbon.

Very common in the industrial sector, the *iodine number* measures the micropore content of biochar or activated carbon (0–20 Å, or up to 2 nm) by adsorption of iodine, where results are often reported in mg/g (Mianowski et al., 2007).

Finally, adsorption of R134a at ambient temperature has also been proposed to characterize carbonaceous materials, where results are provided in percentage of weight gained.

Other ways of characterizing biochar include moisture content, ash, mobile matter, and resident matter (McLaughlin, 2010), in addition to

physical attributes such as weight, hardness, greasiness to the touch, and morphological descriptions such as size, characteristic length, and shape.

## 1.4 Activated Carbon

For applications related to pollutant absorption from liquid or gas streams, a material with very high surface area and microporosity (porous diameter, $d < 2\,\text{nm}$) is desired. In addition, the inclusion of functional groups that enhance adsorption properties would improve filtration performance. Biochar does not have a very high surface area and it tends to have a large fraction of macro ($d > 50\,\text{nm}$) and meso ($2 < d < 50\,\text{nm}$) porous as opposed to microporosity, making it good for soil applications but not for filter material.

Biochar can be post-processed by chemical or physical activation in order to modify its porosity distribution and increase surface area and adsorption properties. Physical activation involves subjecting biochar to high temperature steam or carbon dioxide (around 800 °C), while chemical activation involves soaking biochar with KOH or NaOH and then heating to temperatures around 400–500 °C. Activated carbon has a higher fraction of fixed carbon and its surface area and porous size distribution vary with burn-off percentage. An excellent source of information for activated carbon can be found in Marsh and Reinoso (2006).

## 1.5 Pyrolytic Graphite

As biomass is carbonized, its electrical conductivity increases by several orders of magnitude. In a similar manner, graphite is made when a mixture of hydrocarbons are heated to graphitization temperatures (2000 °C or higher). As opposed to biochar and activated carbon, graphite is a crystalline material with a comparatively low porosity. These attributes enhance the electrical conductivity of graphite, which is about two to three orders of magnitude higher than that of biochar obtained at temperatures above 700 °C. However, compared to metals, the electrical conductivity of graphite is more than two orders of magnitude lower. For example, the electrical conductivity of POCO graphite is $7.8 \times 10^4\ (\Omega\ \text{m})^{-1}$ compared to copper, which is $3.58 \times 10^7\ (\Omega\ \text{m})^{-1}$ (Entegris, 2015). In this chapter, properties of (polycrystalline) POCO® Graphite AXF-5Q are used for models shown in Chapter 5, so when future reference is made to graphite without a qualifier,

it means POCO graphite. The thermophysical properties of POCO and other commercial polycrystalline graphites are nearly isotropic.

Tables 1.1–1.4 provide a list of properties for a graphite as well as activated carbon.

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