SIMPLE LAB METHOD FOR DETERMINING CARBON CONTENT OF BIOCHAR

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Introduction

Biochar is most accurately characterized by the set of analytical methods that have been proposed by the International Biochar Initiative[1] and the European Biochar Certificate[2]. However, the full suite of these tests can be very expensive. For purposes of carbon removal verification in small, artisanal biochar projects, it is preferable to draw conservative values from a database of characterized biochar made from known feedstocks and production methods. However, to minimize reliance on conservative default values, it is beneficial to include a regular program of sampling and testing to verify the mass fractions of biochar moisture, carbon and mineral ash components. For instance, if a project takes place at a certain location where kilns will be operated for several days or weeks processing essentially the same feedstock, this basic analysis could be completed on a mixed sample sometime during the project to provide a carbon content value that can be applied less conservatively across the whole project.

To facilitate access to testing like this, it is possible to employ students, using comparatively simple lab equipment available at schools to conduct these analyses. In this paper, we describe a methodology for simple biochar component analysis that was tested by students at Butte Community College in California.

The analytical data on biochar presented here are the result of laboratory studies at Butte College in Oroville, California, under an EPA-P3 (People-Planet-Prosperity) Phase II Grant (SU839370), Biochar Research and Development, with grant period March 2018 to February 2020. The Report Final was submitted to EPA in February 2022, available on request from authors[3].

The project was conducted by community college student interns enrolled in the MESA (Math Engineering Science Achievement) Program under the supervision of John Dahlgren (Principal Investigator) and Stephen Feher (Technical Director). The students received hands on training in laboratory procedures by Hugh McLaughlin, PhD, PE & CTO of Next Char, LLC, based on his published guide: Basic Biochar Metrics, Version X, November 8th, 2016[4].

Laboratory testing and characterization of biochar's stability

The focus of the applied research on biochar by MESA interns at Butte College was carbon sequestration and agricultural applications for soils improvement to enhance water retention and plant growth. These applications require the biochar to remain stable over time and under various conditions. To determine the carbon and ash content of various biochars produced during the project we used simplified laboratory analysis methods developed by Dr. Hugh McLaughlin. Four different biochars were tested:

a) Rice Hulls Biochar made in Olivier Pyrolizer at Butte College
b) Walnut Shell Biochar made in NextChar 1X Pyrolizer
c) Hard Wood Biochar made at Berkeley Olive Grove by On-Site Biochar Production
d) Oregon Softwood Biochar made by Rogue Biochar[5]

Biochars made from Rice Hulls, Walnut Shells and Olive Wood were of early interest in the Butte College project due to the abundance and accumulation of these agricultural waste products in the Sacramento Valley, where the college is located. Later in the program, after the 2018 Camp Fire, focus of the interest shifted to biochar made from forest waste.

The procedure was carried out by first filling 30 mL crucibles about half full of each type of Biochar and covering the crucible with aluminum foil with a small hole punched in the top, and then, cooking the samples overnight at 150°C. Mass was measured before and after cooking and the weight loss is attributed to water present in the biochar prior to drying at 150°. Samples were then held for an hour at 250°C, 350°C, 450°C, and 550°C each, and mass measurements taken after the end of each temperature ramp. Mass loss was graphed as a function of temperature in order to compare the thermal stability of the different biochars as shown in Figure 1.

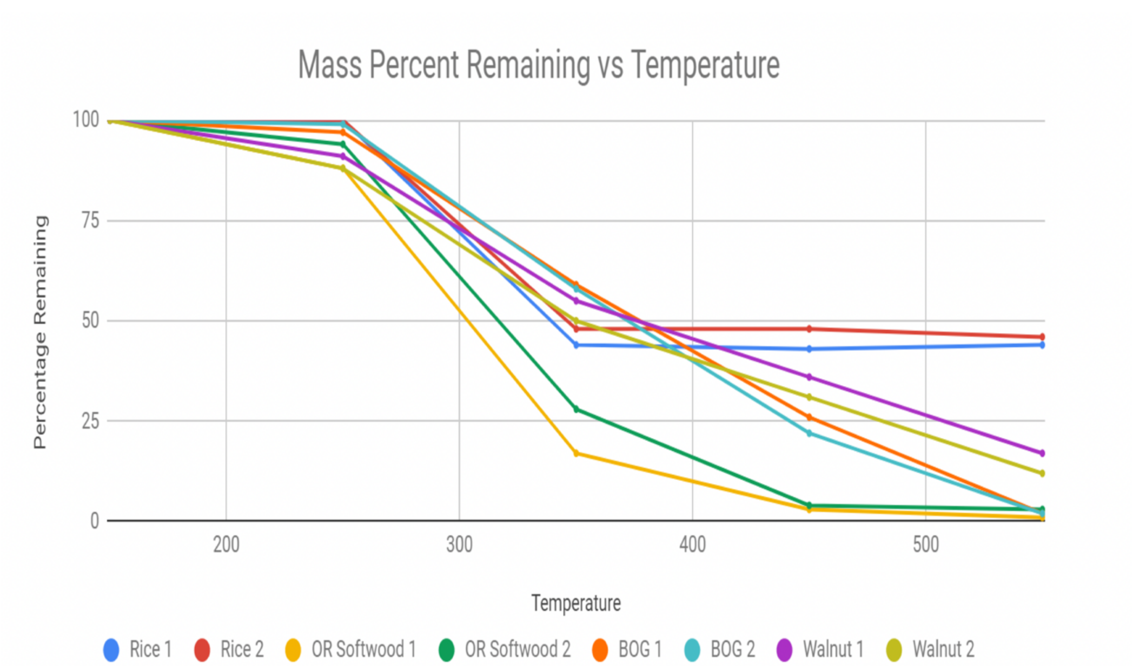

Figure 1: Graph shows the mass percent remaining of the different biochars heated to increasing temperature levels (250, 350, 450 & 500 Degrees C) and held for 1 hour at each level in a vented air oven.

Analysis

Close analysis of this data provides valuable indications as to the composition of various biochars, as follows:

The weight-loss in the temperature range of 150°C to 250°C represents approximately 10-15% of the bone-dry weight (after drying at 150°C overnight) of the samples, may be attributed to some moisture absorbed by the samples after the overnight drying, during handling in carrying out the rest of the procedure. We have observed many times in our experiments that “dry biochar” picks up moisture very fast despite careful procedures to prevent it. It is fair to say that it is almost impossible to prevent biochar from absorbing moisture. In practicality, there is no such thing as “bone dry biochar”!

It is interesting to observe from the data, that the biochar made from rice hulls had no further weight loss during heating to higher temperatures. That indicates that the remaining material is inert to further oxidation, thus it is considered to be ash. The nearly 50% ash content by weight indicated by the test data correlates well with other tests of rice hulls biochar composition (Ref. 2).

Upon further heating at 450°C the Oregon softwood biochar samples appear to have also reached ash status with no further weight loss. The ash content in this case is much lower percentage than in the case of rice hulls biochar, only 3-5% by weight.

The BOG 1&2 biochar samples (pyrolyzed in “open kiln burn” from olive wood local orchard cuttings) reached the ash stage at the 550°C heating stage at very low weight percentage, in the range of less than 5%.

The walnut-shell biochar samples have seemingly never reached “ash stage” based on the test results. After an hour at 550°C, the weight-loss curve is still showing a straight line down with 10-15% of the original weight remaining. This sample indicates that some biochars will need either a longer time or a higher temperature to reach the ashing point.

While it is difficult to interpret the meaning of the weight loss curves in these samples, it is valid to use the data to determine mass fractions of water content and ash. The organic carbon content is determined by subtracting the weight of water and ash from the original sample weight before heating.

To facilitate access to testing like this, it is possible to employ students, using comparatively simple lab equipment available at schools, to conduct these analyses. The main requirement is an electric furnace with the capacity to heat to 550°C and an analytic balance capable to measure with the accuracy of milligrams. A set of a dozen 50-100 mL porcelain crucibles are also required, which must be handled by metal tongs when hot.

[1] https://biochar-international.org/certification-program/
[2] https://www.european-biochar.org/en
[3] Final Report by John Dahlgren and Stephen Feher, of Butte College, Oroville CA on EPA-P3 Grant SUB39370, BIOCHAR R&D, February 2022.
[4] McLaughlin, Hugh. Baseline Biochar Metrics Series, https://www.nextchar.com/baseline-biochar-metrics-series/, Accessed on November 8th, 2016.
[5] Rogue Biochar (https://www.chardirect.com).