

Perspective on Baseline Biochar Metrics – Past, Present & Future Version VI – Nov 18, 2016

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Motivation: The first drafts of the "Baseline Biochar Metrics" were issued in October, 2016. A respected colleague acknowledged that he could understand the underlying drivers for the individual tests, based on his familiarity with previous writings on the subject and the past body of work on biochar by the author, but feared that others may lack this historical perspective. The colleague recommended that the effort be supported by two things, as quoted below;

"1) Give the reader a bit more background on why you recommend each of these measurements. You could do so by giving a brief reason why and referencing one of your previous publications, or just cut and paste in some of that discussion.

Provide a table listing the parameters measured and perhaps what that parameter tells you. That would give a quick reference for future users."

Summary of Past Efforts: There are basically two concurrent threads that *diverge* to create the current situation; a sequence of efforts conducted by IBI that created the IBI Biochar Certification Program & associated Biochar Standards and a series of early works by Frank Shields and Hugh McLaughlin that proposed a characterization approach known as the "McShields Method". Other than timing, there is little in common with these programs, but the thread on the each still exists in the catacombs of the web and serves to set the stage for the evolution of the current Baseline Biochar Metrics.

The history of the IBI program is documented at the IBI website and provides a convenient compendium of documents. The earliest foundations appear to be a document called "Guidelines for a safe Biochar Industry", which aspires to address a void described as "The IBI and Australia New Zealand Biochar Researcher network recognize that there are currently no guidelines for manufacturers, retailers or purchasers of biochar to describe the material." Over the course of three years, that effort ballooned to a 47-page manifesto, which was voted on by the eligible IBI membership and adopted, with 90% of the only 100 votes cast advocating adoption. The latest revision of the IBI Standards, IBI Biochar Standards Version 2.1, weighs in at 61 pages.

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The McShields method had very different origins, being developed as part of the analytical efforts utilized to generate the data in a paper titled "All Biochars are Not Created Equal, and How to Tell Them Apart" prepared for the 2009 North American Biochar Conference, Boulder, CO. To be honest, at the time, we were measuring everything we could about the chars using the instruments and methods available to the authors, lacking any budget for "outside analyticals". The methods were utilized in a sequel paper for the 2010 biochar event in Iowa titled " Schenkel and Shenxue revisited - implications on char production and biochar properties", with the McShields procedure delineated in Appendix D.

Subsequently, many of the McShields analyses were offered by an established commercial soil testing business, Soil Control Labs of Watsonville, CA, as a series of cost-effective biochar characterization packages. Initial bundled biochar characterization packages included additional affordable soil tests, such as NPK and liming metrics. As the IBI Characterization effort advanced, additional measurements were added to address IBI Characterization requirements, such as metals and sieve analysis. Unfortunately, the current IBI includes thousands of dollars per sample of toxicity testing for PAHs, PCBs and Dioxin/Furans. Due to the excessive cost and limited utility for predicting biochar efficacy in real-world applications, marketplace adoption of the current IBI protocol has ranged from minimal to abysmal.

Concurrent with the IBI and McShields approaches was the ongoing maturation of the "science of biochar", with academic publications addressing issues such as the dynamics of biochar in the soil and the challenge of predicting the stability of biochar-sequestered carbon contained in a diversity of growing systems. As is often the case of a new area of research, there are more questions being raised than answers being provided. What has emerged is a lack of consensus on how to predict the performance of biochar from the feedstock and how it was made, with no consensus on how to characterize a sample once it is produced.

Furthermore, the predictions of biochar properties and consequences based on atomic ratios, such as H/C and O/C, and sophisticated instruments like magic angle spinning NMR have proven inconclusive and lacking in sound scientific foundations. De facto, the carbon sequestration impact of most biochar additions is represented by the response of the receiving soil/growing system to a much greater extent than the "recalcitrant" properties of the original biochar. As such, measuring an intrinsic biochar property, and using it as the basis for carbon sequestration

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accounting, is grossly undervaluing the biochar and stunting its potential in future climate discussions and programs.

Summary of Recent Efforts: There was an acknowledgement heading into the August USBI2016 Biochar Conference in Corvallis that "biochar characterization" was an industry-wide critical-path issue, driven by the diversity and disparity of products and representations in the current biochar marketplace. Initially, there was envisioned a process involving a round-robin of ideas, followed by consensus by the principal stakeholders, culminating in an announcement of the resulting work products and/or voluntary standards at the USBI2016 event.

Well, that didn't happen (best laid plans of mice and men.....). The author was motivated to write up his thoughts and present at USBI2016 on a potential approach, titled "NextChar Characterization Matrix - Measuring biochar properties to establish Valuation". Notably, this presentation represented essentially all the work product devoted to resolution of the characterization vacuum; however, the desperate cries for clarity, consensus and resolution were genuine and widespread at the USBI2016 gathering.

Which brings us to Occam's (Ockham's) Razor; the author went into the lab and stripped the analytical options for biochar to the minimum necessary steps, and built up what could logically be concluded from each exercise. It took a few weeks, but coupled with many years of experience working with myriads of biochar and a diversity of analytical methods, the "Baseline Biochar Metrics" approach was assembled upon the lessons derived from past efforts. In the process, it was determined that several of the metrics being sought were, in fact, not accurately being measured, but rather quantified with the inclusion of potentially significant sources of error. Such interactions, and the limits they place on the insights into the biochar being tested, are discussed for the individual metrics.

Summary of "As is Biochar" Metrics: - performed on "as is" or "as received" or "damp" biochar: These tests are intended for quick field evaluation of biochar for calculating shipping weights and check for concerns relating to the addition of the biochar to an existing soil.

As Is Bulk Density: "as is" test; used to estimate of shipping weights As Is Contact Ratio: The ratio of weight of "Contact water" to the weight of 'As Is" biochar Contact pH: check to see if there is excessive liming effect that could affect alkaline soils

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Contact TDS/EC: check to see if excessively brackish (salty) conditions result if soil is wetted **Contact Coloration of the filtered water**: most filtrates are clear; any significant color is something soluble and colored leaching off the biochar, which is rarely observed in practice.

Summary of "Dried Biochar" Metrics: By drying the biochar, the measurements dissect the solid biochar itself using gravimetric methods (doing things to the biochar sample and quantifying the impacts by the change in weight). These tests provide insights into the biochar production conditions and the intrinsic properties of the biochar at the time of purchase. This procedure follows the strategy of McShields Method, where the biochar is split into Water, Mobile Matter, Resident Matter and Ash – however, it turns out it is hard to get exact partitions in biochar, so the expectations are lowered to reflect what is actually being measured in the assay, and how this tempers insights into the biochar dynamics in the soil.

Drying Weight Loss: Quality biochars have bound or adsorbed water, which cannot be removed at typical laboratory drying conditions of 105°C. In order to remove the bound water, higher temperatures are needed (150°C to 200°C), but biochar will also lose some portion of VOCs, if present, when dried at these higher temperatures. In addition, once biochar is completely desiccated at the elevated temperatures, it rapidly takes up moisture from the air – which introduces another source of error in the measurements. In order to minimize these interferences, the experimental set up is designed to allow drying with controlled loss of volatiles and minimal contact of the dried sample with air before the next property is measured (usually the Volatiles or Inorganics assay).

Volatiles Dry Wt Fraction: This measures the weight loss when the dried sample is heated to 450°C in the covered container. The temperature is high enough that any VOCs and SVOCs (tars) are driven off, but low enough that the graphitic carbon in the sample does not oxidize to carbon dioxide. This measurement is the closest measure of the "Mobile Matter", but it is not exact because some mobile matter is pyrolyzed into additional resident matter at the assay temperature - and therefore not fully represented in the weight loss. In addition, some biochars have surface oxides on the graphitic internal surfaces and carboxylic acid groups that decompose at the 450°C temperature, which is a weight loss that is not associated with the vaporization of Mobile Matter. Furthermore, if any VOCs were lost during drying, they are not counted as Mobile Matter, but should have been. Overall, the Volatiles Dry Wt Fraction is a

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temperature-induced "cut", where the more weight loss means more of something is coming off the sample, without exactly knowing what makes up that something. As such, it is a broad indicator of what portion of the biochar is not tightly bound and not likely to be long term sequestered carbon. Excessive volatiles are often an indicator of leachable soluble organics, which can cause available plant nitrogen deprivation if the biochar is directly put in the soil without composting, which biodegrades the VOCs, or charging with excess plant nutrients.

Inorganics Dry Wt Fraction: This assay reacts the dried biochar with oxygen in air and removes the organic portion of the biochar, representing the combination of the Mobile Matter and the Resident Matter. The weight left behind represents the total inorganics, often called "ash", since the process of burning off all the organics and seeing what is left is a laboratory procedure called "ashing". The temperature of the ashing is 550°C, which is chosen to oxidize away the organic portion, but not convert the inorganic carbonates present into the corresponding oxides (thereby driving off carbon dioxide and resulting in a reduced weigh of ash. This error would be quantified as part of the stable organic Resident Matter, which the carbonates are not). The class of inorganics includes many of the plant fertilizers (potassium, phosphorus, calcium and micronutrients), but also can include less welcome components such as salt (sodium chloride) and inert materials such as sand (silicon dioxide). As such, if the Inorganics Dry Wt Fraction is elevated (above 0.10 dry weight fraction or 10 weight percent), then it is recommended that additional information be provided on the breakdown of the Inorganics in the biochar. Note that Inorganics assay is not an effective method for the detection of Heavy Metal contamination in biochars. Those considerations and analytical options are discussed further in the section on "Beyond Baseline Metric Options".

Adsorption Capacity Assay: Adsorption is the unique property of biochars that is pivotal in some situations in the soil, specifically providing detoxification, ameliorating desiccating conditions, and promoting microbial populations. Unfortunately, adsorption is hard to measure and cannot be inferred from the presence of other properties. There are several options for determining adsorption capacity of a biochar, and the adsorption capacity of a biochar needs to be accurately quantified. Furthermore, the presence of significant adsorption capacity implies several other beneficial attributes to the biochar, such as the inhibition of SVOCs (tars) leaching. The options for measuring Adsorption Capacity are included as an appendix to the Baseline Biochar Metrics Procedures.

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Summary of "Wetted Biochar" Metrics: These tests are focused on the biochar properties and interactions once the biochar is fully wetted in the soil environment. As such, it does not directly measure intrinsic biochar properties, but rather subjects the biochar to complete wetting and measures the properties of the wetted biochar and the resulting wetting water. It is intended to assist the soil scientist and the consumer in comparing properties of saturated biochars and predicting associated soil impacts.

Dried Settled Density: Density is the measure that relates the weight of a material with the corresponding volume, so it is a critical relationship. Unfortunately, density is hard to measure for biochar because biochar is crushable (technically referred to as friable), so the total bulk volume of a given weight can change as the material breaks down in particle size. For this reason, we measure several densities and take them into consideration when comparing biochars and predicting future properties.

Extraction Ratio: This is the ratio of the weight of water used to wet the biochar to the weight of biochar being wetted. It is captured to allow other researchers to reproduce the extraction conditions and assure the lab does not bias the subsequent liquid measurements by using a gross excess of extraction water.

Wetted Biochar Density: This is a second density metric that provides insight into how the biochar behaves when fully mixed and wetted in the soil. It is measured because it may prove to be a better predictor of future biochar properties than the Dry Settled Density, although the relative analytical merits of each density metric are not known at this time. In addition, it is easily measured during the execution of the steps in the "Wetted Biochar" Procedure.

Biochar Water Holding Ratio: This is one attempt to create a reproducible and facile metric of the water holding ability of a biochar and allow comparison with other biochars. The range of this metric is quite significant, from as low as 0.5 for dense biochars to 5 or more for low density biochars.

(Optional) Wetted GACS Adsorption Capacity: As discussed in the procedure, this is a convenient sample to check to see if a biochar adsorption capacity improves or deteriorates

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upon wetting in the soil. Many biochar manufacturers speculate that their biochars will develop additional adsorption capacity as the mobile matter leaches out. This sample provides a potential insight into that hypothesis, although that prediction remains to be validated by additional research (by others).

Extract pH: check to see if there is excessive liming effect that could affect alkaline soils

Extract TDS/EC: This metric yields an indicator of the level of extractable compounds that dissolve into the soil water over time. The specific impact of a elevated measured level likely requires additional partitioning of the makeup of the TDS/EC constituents, however a low reading indicates reduced potential for any impacts, both negative (brackishness) or positive (a source of plant nutrients). Suffice to say, high quality low-ash biochars have essentially no risk of adverse TDS/EC effects. Note that the sample for TDS/EC can also be used to assay for other soluble and/or extractible compounds on the biochar, such as TOC and/or PAHs. The aqueous extract is a much better indicator of bio-available impacts of the biochar, since many potentially toxic organic compounds are tightly adsorbed on biochar and unlikely to be bio-available at concentrations of consequence in the soil.

Extract Coloration of the filtered water: most filtrates are clear; any significant color is something soluble and colored leaching off the biochar, which is rarely observed in practice.

Beyond Baseline Biochar Metric Options: The Baseline Biochar Metrics are intended to be the starting point for the characterization of a biochar sample. The primary audiences are researchers, to characterize their biochars in a manner to allow other researchers to compare and contrast core biochar properties, and the marketplace, providing a consistent set of characterizations of available commercial offerings. Depending on the specific attributes measured by the baseline metrics, there are logical additional assays to augment the understanding of the biochar properties and anticipated impacts in growing systems.

For any biochar sample, there is merit in subjecting it to available routine soil tests for a few reasons; the material will likely be mixed with soils and the soils testing readily identifies the non-biochar specific merits of the sample, such as the levels of plant nutrients (NPK, etc.) Most

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biochar samples can be treated as analogs to compost samples for the purpose of soil testing – and if the biochar meets the purity requirements for compost, that speaks volumes about the appropriateness of adding it to soils. This approach is especially appropriate when looking at heavy metals – which is a class of materials that spans from things to be rigorously avoided (mercury) to a wide range of beneficial plant micronutrients. Current analytical methods can basically detect some level of anything in any sample, which is why it is important to put the measured reading in perspective. Thus, the regulated and acceptable levels of heavy metal levels in compost products provides a reasonable set of reference target levels for the same elements in biochars.

The individual supplemental analytical directions are briefly summarized here.

Beyond "As-Is Biochars" Measurements: The focus of the "As Is Biochar" Procedure is a quick and convenient check of a few basic biochar metrics – relevant considerations prior to shipping and prior to mixing the biochar directly into soil. It is not recommended that this procedure be expanded beyond the current limited utility, since the subsequent dried and wetted biochar procedures have much better analytical methods and accuracy, thus providing a better starting point for analytical improvements. As such, the path beyond the "As Is Biochar" Procedure is e basically to go to the "Dried Biochar" Procedure and/or the "Wetted Biochar" Procedure.

Beyond "Dried Biochars" Measurements: This procedure generates a few basic metrics that are focused on the properties of the solid biochar, providing insights to how it was manufactured and what the purchaser is getting. As such, this is the most productive opportunity for improving the understanding of the biochar, the material itself. The individual metrics have the following room for improvement:

Drying Weight Loss: As discussed, drying a biochar is a challenge to remove the water, including the tightly adsorbed moisture, while leaving the volatiles (VOCs and SVOCs) behind. Alternate drying conditions, such as partial vacuum and even freeze-drying (technically called lyophilization) may improve the separation. Freeze-drying is a fairly routine procedure for labs dealing with foods and perishables, but it is an area that academic researchers should sort out before the marketplace switches to a different method.

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Volatiles Dry Wt Fraction: There are several directions the Volatiles assay could go, and they all point to the academic researchers initially dissecting the options and impacts. When the Mobile Matter assay was first developed, its limitations were identified, as discussed in Schenkel and Shenxue revisited – Version 1 (June 2010) - page 6: "The mobile matter is postulated to represent that portion of the biochar that is unstable over time in the soil. The principal mechanism for loss of the mobile matter in the soil is believed to be the migration from the biochar into the soil pore water and subsequent metabolization by soil microbes. At this point, and pending further research by the soil scientists and vetting this assay against other available analytical options such as Soxhlet Extraction with a water miscible solvent, the mobile-resident partition is just a practical and convenient analytical procedure that generates a metric for comparing chars. At this juncture, perhaps it should be treated as a premise, awaiting validation by others and based on future research results." Unfortunately, so far, the academics have not taken the bait.

In addition to improving the method of isolating the short-lived organic portion of various biochars, there is merit in further studying the exiting volatiles, perhaps by a thermally controlled desorption with coupled GC/MS characterization. Knowledge of the specific chemicals desorbing into the soil water would provide significant insights into the observed and anticipated impacts.

Inorganics Dry Wt Fraction: This assay does a very efficient job of isolating the non-organic fraction of the biochar. Standard analytical partitions, such as acid-soluble and acid-insoluble fractions, along with standard soil tests for fertilizer/compost properties, are recommended, as discussed above and if justified by the level of Inorganics present in the biochar.

Adsorption Capacity Assay: Adsorption is a very difficult area to study analytically, especially for a system as complex as any actual soil-biochar-soluble organics equilibrium. The current GACS assay provides the current state-of-the-art characterization from the activated carbon industries and it is recommended that the insights provided by that approach be exhausted before we start looking for a better mouse trap.

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Beyond "Wetted Biochars" Measurements: This procedure is performed on wetted biochar and provides important insights into both the physical and chemical interactions that await the introduction of the biochar into soil systems.

Dried Settled Density: Dry density is quite sensitive to the particle size distribution; because that dictates how the individual particles pack within the settled bed. Academics could dissect the interactions of particle size distributions and packing densities, but any such efforts should be coupled with additional studies to predict how the friable biochar will break down in specific situations in the soil, etc. (otherwise, the value of the results will really be "purely academic").

Wetted Biochar Density: This assay actually has great potential because it accesses the properties of fully wetted biochars, the condition encountered in saturated soils. The assay can be modified to provide a measure of the skeletal density of the solid biochar itself, which allows insights into the relative portions of solid versus voidage in the biochar. It is recommended that a body of comparative data be accumulated on dried versus wetted densities for a wide range of biochars before any additional embellishment of the current density offerings is pursued.

Biochar Water Holding Ratio: This is an area that needs additional validation, since it is one simple procedure that generates a comparative metric between biochars, but the metric has not been further correlated and/or validated as to the impacts in actual soil systems. It is an area where the academics should provide the heavy lifting (since studying core phenomena, ferreting out the underlying science, and writing about it is what they are all about) and the industry can adopt improved water-biochar metrics as they are validated in the reviewed scientific literature.

Extract pH: pH is a convenient but often misleading and misinterpreted metric of biochar. The pH impact of a biochar is really dictated by the pH buffering of the receiving soils, and most soils with significant microbiology have local pH levels dictated by the microbes by the excretion or metabolization of local soluble organic acids. As such, one could generate a titration curve to measure the buffering of the raw biochar, but then one would have to know or measure the corresponding buffering properties of the receiving soil matrix. At the end of the effort, it is unlikely any conclusive insights will be gained. As such, Extract pH is a flag, but not a hard and fast differentiator of a core biochar property. It does serve to identify a class of

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gasifier biochars that have any salts present in the oxide form, such as Calcium Oxide, that may result in pH levels above 9 if introduced to soils in large quantities. This consideration is relevant in drier climates and alkaline soils, such as encountered in the Western USA.

Extract TDS/EC: This procedure yields an equilibrated and concentrated aqueous extract of the original biochar and provides a unique predictor of short-term effects of a biochar when it is added to the soil. TDS/EC are looking at short term ionic level effects, but the aqueous sample can be queried for a number of soluble constituents, including VOC, SVOCs, PAHs, etc. Quantification of the magnitude (the "Equivalency" calculation piece) of the specific extractable chemicals allows projection of anticipated soil-plant interactions based on the addition of a quantity of a biochar to a specific growing system, but will require significant controlled experiments to provide the basis for developing accurate guidelines and projections.

Extract Coloration of the filtered water: most filtrates are clear; any significant color is something soluble and colored leaching off the biochar, which is rarely observed in practice. The presence of significant coloration in the filtered Extract Aqueous Phase should raise the issue of "what kind of tricks is the biochar producer trying to pull this time".

As a final note, America just elected a climate-denier as the next President, which will shift the emphasis of biochar away from carbon impacts and towards soil and remediation benefits.

Citations and Recommended References

IBI Biochar Standards Document Archive of past documents and steps in the development of the IBI Certification: <u>http://www.biochar-international.org/node/3340#documents</u>

IBI Biochar Standards Version 2.1 can be downloaded from <u>http://www.biochar-international.org/characterizationstandard</u>.

McShields Method: downloadable at http://acfox.com/index_files/Page509.htm

All Biochars are Not Created Equal, and How to Tell Them Apart. Version 2 (October 2009), which supersedes the digital reprint issued at the North American Biochar Conference, Boulder, CO – August 2009. Hugh McLaughlin, PhD, PE, Paul S. Anderson, PhD, Frank E. Shields and Thomas B. Reed, PhD

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Schenkel and Shenxue revisited - implications on char production and biochar properties. Version 1 (June 2010) issued at the Biochar2010 Conference, Ames, Iowa – June 2010. Hugh McLaughlin, PhD, PE and Frank E. Shields

Appendix D: McShields Biochar Characterization Procedure, contained in Appendices A through E of Schenkel and Shenxue revisited - implications on char production and biochar properties. Version 1 (June 2010) issued at the Biochar2010 Conference, Ames, Iowa – June 2010

NextChar Characterization Matrix - Measuring biochar properties to establish Valuation. Hugh McLaughlin,NextChar, LLC (downloadable at http://biochar-us.org/presentation/nextcharcharacterization-matrix-measuring-biochar-properties-establish-valuation)

Occam's (Ockham's) Razor is defined at https://en.wikipedia.org/wiki/Occam's_razor

Chapter 8 of "The Biochar Revolution", copyright 2010 Global Publishing Group, titled Characterizing Biochars: Attributes, Indicators, and At-Home Tests. Hugh McLaughlin, PhD PE Director of Biocarbon Research, Alterna Biocarbon Inc. (mail order via http://biocharbooks.com/TBRDetails and web sites like ebay.com and Amazon.com)