BBM on a Budget

Baseline Biochar Metrics (BBM) measurements on a Budget

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Introduction: A strategy for characterizing raw biochar (as created and prior to any post-pyrolysis modifications) called the "Baseline Biochar Metrics" was recently developed and is currently being evaluated by the "Stakeholders of the Biochar World" as to its merit. The BBM protocol involves a set of fairly simple tests that measure fundamental biochar properties. Because Biochar is a unique material that has a corresponding unique set of impacts when introduced into agricultural and remediation applications, most of the tests are specific to biochar and have been developed carte blanche to allow the most accurate measurement of the target metrics. As such, the individual tests tend to be fairly labor intensive and less instrument intensive, since biochar has not been around long enough for the instrument marketplace to develop and promote the adoption of expensive instruments over more labor-intensive methods.

Background: The current version of the Baseline Biochar Metrics is Version X – Nov 18, 2016. That version provides some guidance on the specific equipment requirements, with that section reproduced below:

> **Recommended Dedicated Equipment:** Laboratory Analytical weighing capabilities or (via ebay) 500+ gram balance, accurate to 0.01 gm, 50 gram balance, accurate to 0.001 gm, 12 oz canning jars with lids, 4 oz canning jars with lids, filter paper (Whatman No. 5, paper towels as alternative), 30 ml porcelain crucibles, regular weight aluminum foil, pH paper or pH meter (calibrated with fresh buffers), TDS and/or EC meter. Apparatus for adsorption testing (GACS recommended, propane or butane activity as alternative). Standard laboratory capabilities (drying ovens, muffle furnace, etc) and additional small items as specified in procedures.

What is presumed in the above equipment are some standard laboratory capabilities, such as drying ovens and the unusually demanding muffle furnace requirement (medium temperature for a muffle furnace but accurate temperature control), in addition to the overhead of typical analytical balances. **The purpose of this document is to provide additional guidance for sourcing inexpensive options for these specific equipment requirements.**

The goal is to provide the ability to accomplish the requirements of the Baseline Biochar Metrics procedures with reasonable accuracy and minimal cost, starting from essentially no equipment, but assuming a clean dry environment where accurate analytical work can be conducted. As such, an unheated garage may be a bit rough, but can be pressed into service if the door can be closed and the technician has good attention to detail. What is not required is a traditional chemistry laboratory with bench tops and hoods, etc. The biggest constraint is safety, since some pieces of equipment run hot and have to be positioned such that passers-by do not brush up against them. As a rule, excess foot traffic does not help the quest for accurate measurements, but practice and repetition will make up for any situational shortcomings. Furthermore, slightly sub-optimal data is vastly superior to inadequate and/or insufficient data.

Skills needed to attempt this: While the skill set required assembling the specific apparatus are not exhaustive, it is a task not recommended for everyone. Some power tools will make the tasks easier, but what is most important is that the builder can read these instructions and fill in the gaps in the guidance based on understanding the intent of the design. If these instructions make "no sense" to you, then don't attempt this without additional help from others that can read between the lines and ferret out the functional intent of specific details.

In addition to fabrication challenges, there is a "scientific method" being applied to running the individual BBM procedures. The BBM procedures measure specific biochar properties, and one has to make sure that the desired measurements are both precise and accurate – and repeatable. The rigor and attention-to-detail utilized in passing an undergraduate science lab course is the right set of skills. So, if you hated science labs or flunked several, this guidance may not be for you. Sometimes it takes one kind of person to make the equipment and another type to execute the analytical procedures – so look for help and learn from someone who has mastered the specific challenge you are up against.

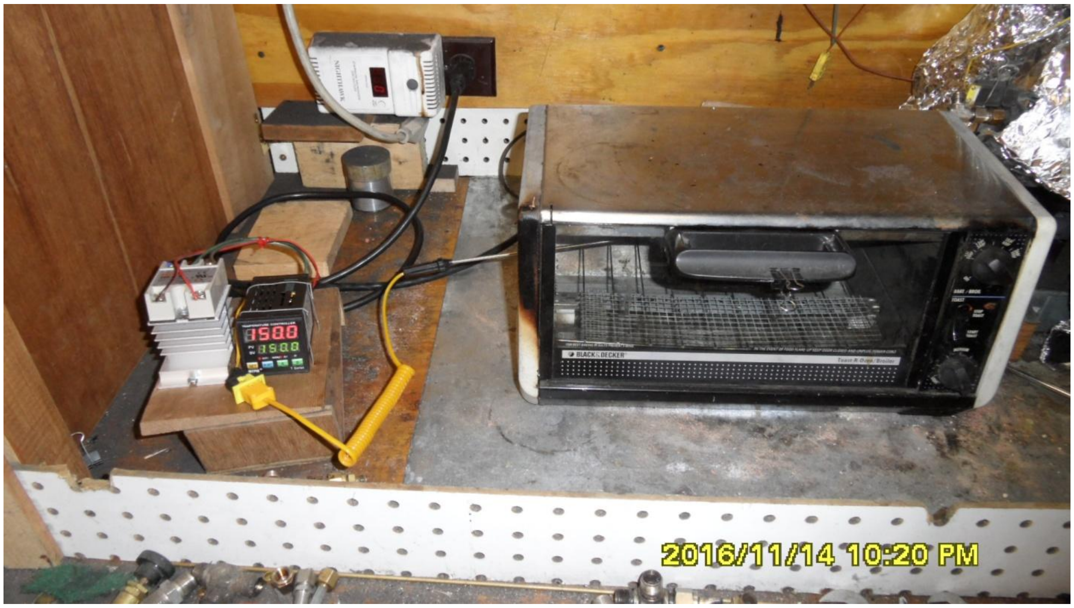
Option for the Drying Oven: Every analytical lab has one or more drying ovens, set at 105°C and used to dry glassware after it is cleaned and before it is used for the next assay. The problem is biochar samples are dried at 150°C to remove adsorbed moisture, with the accepted range for the drying oven being 145°C to 155°C. The end result is trying to share a drying oven is not practical, since it will always be at the wrong temperature for some of the users.

Fortunately, the lowly toaster oven serves nicely as an alternate option for drying biochar samples for the BBM procedures. 150°C equals 302°F, which is comfortably in the range of any toaster oven if all the glow bars still work. The challenge is getting the temperature controlled within the desired range of 145°C to 155°C (293°F to 311°F) called for by the ASTM method for drying activated carbon samples (ASTM D2867). To be honest, the temperature control is not critical, but it can be a source of error when drying high volatile biochars, so it is worth avoiding if possible.

In order to calibrate the toaster oven, it is typical to measure the internal temperature of the toaster oven under the control of the built-in dial thermostat. This allows one to assess if the internal thermostat is in error and how wide is the temperature "dead-band", which is the interval between when the thermostat turns on to heat and then turns off after exceeding the temperature setpoint. This is done by drilling a 1/8" hole through the non-controller side of the toaster oven to allow a 1/8" by 4 to 8 inch thermocouple to be inserted and actual temperature of the inside of the toaster oven to be measured at any time using a digital thermocouple thermometer.

However, it may be easier to solve the problem than to measure it when it comes to accurate temperature control of a lowly toaster oven. If one goes to ebay.com and searches on the terms “digital temperature controller SSR heat sink”, one will find for $15 to $35 a number of suppliers from the Far East selling packages including a 1/16th DIN (that’s the size of the unit) digital PID temperature controllers with a solid state relay (SSR) and the heat sink for the SSR. Then search “3mm K thermocouple” and find a unit between 140mm and 190mm in length – buy a couple (recommend having a spare on hand). Then search on “K thermocouple female connector” to get the mate to the molded plug on the thermocouples. The last pieces are a short piece of K thermocouple wire and an extension cord – cut in half to make the pigtails to supply 120V to the controller and the controller power outlet that the toaster oven plugs into.

The whole system gets mounted together to cut down on wear and tear on the wiring. A completed system is shown in the picture below. The nice feature of the Digital Controllers is a function called “autotune” – which optimizes the response of the controller to the dynamics of the load that is being controlled. Once autotuned, the control of the temperature is excellent, although whenever the oven door is opened it introduces a load change that requires the controller to regain control once the door is shut again – it usually take just a few minutes.



One last modification of the toaster oven is to attach a piece of ¼” hardware cloth (another name for screen with big openings) on top of the tray of the toaster oven (as shown above) so items will slide smoothly and not get caught in the rails of the sliding shelf. The shelf was designed for toast, which has a whole different set of performance criteria – so the shelf needs a bit of modification for its new lifestyle.

Option for weighing to the required precision: One of the fundamental truths of analytical work is "You can't measure what you can't see", meaning the precision of the measurements has to be much better than the differences in the materials you are testing. For this reason, many analytical methods strive to measure the differences in materials by measuring the differences in weight, since weight is one of the easier measurements to make with precision and repeatability. Just as the bathroom scale is measuring differences in people's weigh to appropriate levels of precision, the scale used to sell sliced meat at the deli has a smaller unit of resolution to establish the accurate charge for the sale. Analytical scales are differentiated by the resolution and the range of weighing, which is combined by calculating how many discernible increments the ranges of the scale is divided into. In general, the higher the partitioning, the more expensive the analytical scale.

As a point of reference, medium quality analytical balances typically break the measurement range into one or two million pieces – which is pretty incredible, but costs a lot of money to provide the very stable electronics, precision moving parts and a working life of years to decades in the lab. In addition, the finer the resolution, the higher the price for an analytical balance. Suffice to say, if money is limited, the place to conserve it is by not buying traditional analytical balances.

In the design of the BBM, care has been taken to make the measuring differences as large as practical and hopefully make the cost of the balances affordable. Furthermore, owing to the dramatic improvement in microprocessor-based devices and the booming illicit drug trade, there is a class of "portable battery-powered scales" that combine low-cost with reasonable accuracy, subject to one restriction: they are not real stable over protracted periods of time – meaning they have to be zeroed (tared), then the item weighed immediately to get the most accurate measurements. Many scales feature the ability to tare a weight and measure an incremental weight – and these scales have this feature also – but the longer the time between the tare and the weighing, the greater the chances of the balance drifting and introducing measurement error.

Which, finally, brings us to the recommended low-cost analytical scales for the BBM: on ebay.com, shipped directly from China and taking up to three weeks to arrive, one can buy a 600 gram capacity scale with 0.01 gram resolution for under $15 and a 50 gram capacity scale with 0.001 gram resolution for under $20. These two scales can cover all the weighing requirements of all three BBM procedures. The units are shown below - they are quite the bargain.

Option for the Muffle Furnace: Along with weight, temperature is another tightly measured and/or controlled variable in analytical measurements, as was seen for the toaster oven when pressed into duty as a drying oven. The accurate measurements of the Volatiles, removed from a dry sample at 450°C, and Inorganics, the ash remaining at 550°C, are the core of the Dried Biochar procedure and two of the most significant metrics of a biochar's quality.

In a typical lab, a unique piece of equipment called a Muffle Furnace would be used for these conditions because it is usually the only piece of equipment that can provide the necessary elevated temperatures in a safe and controlled manner. Unfortunately, Muffle Furnaces are expensive, with very low quality units from India being about $500 and the smallest reasonable quality domestic units starting north of $1000. To make things even more challenging, non-digitally controller unit (with analog or sweeping needle-type readout of temperature) cannot be controlled to the tight temperature limits necessary to get acceptable reproducibility of the analytical results.

Fortunately, the BBM procedures have been designed to NOT require control of the atmosphere inside the muffle furnace for the Volatiles measurement, which would have put the method beyond the capabilities of most commercial analytical labs. In addition, the upper temperature of 550°C (just over 1000°F) is hot, but not so hot that special materials are necessary. With these requirements in mind, a much lower cost alternative has been designed, built and tested. The major design features of this approach are discussed in detail in what follows, but the sole responsibility for construction and safe operation of any unit resides with the end-user.

In operation, the inner surfaces of the unit will be dangerously hot; especially the refractory bricks used to cover the furnace opening and that may be removed during operation to check on the samples inside. Appropriate lab tongs and protective gloves should be used in addition to utmost care whenever a hot furnace is accessed. Fortunately, the exposure to the hot surfaces is limited and any mistake will likely result in a painful encounter, but will heal and leave a burn mark as a constant reminder.

The core of the “Alternative Muffle Furnace” (AMF) is on the next page. The starting point is a 6” long section of 4” x 6” Rectangular Steel Tubing, with a wall thickness of 1/8” to 3/16” (strength is not an issue, but thermal properties and ease of fabrication are). Rectangular Steel Tubing is a common building material and should be available from most fabricated steel supply houses, although they will not be excited about selling 5 pounds of steel and having to provide clean cuts on both ends. An available but more expensive option is McMaster-Carr (www.mcmaster.com), where item number 89825K74 will get you the exact piece in 304SS for under $70 plus shipping. 304SS is not necessary and makes the fabrication more challenging (it has to be drilled and tapped), but it will work if a short piece cannot be sourced locally. Ebay had a 12” piece for $37 by searching on “4x6 rectangular steel tubing” with free shipping. If you buy a longer piece and have to cut it, be sure to have a machine shop cut it on a mechanical band saw intended for steel – you will want a clean flat face to provide a reasonable seal on the ends of the AMF.

As shown below, the rectangular steel tubing is fitted with a small electric burner from a single burner hot plate (the kind every college dorm bans), by cutting a slot in the bottom of the tubing and sliding the burner in from one end. The tubing is also tapped with 4 x ¼-20 threads one inch in from each corner to allow bolts to be used to locate the tubing inside the insulation.

There are a few recommendations about the individual components. On ebay, there are 120V single burner hot plates with 750 watt elements and with 1100 watt elements. The larger unit is recommended because it has been successfully used in the prototype assembled to test out the concept. The particular unit was manufactured by "IMUSA" (search "imusa single hot plate" on ebay – under $20 delivered). The hot plate is disassembled (destroyed) and the burner extracted. The wiring can be reused after cutting off any extraneous bits.

The temperature measurement, used to control the AMF in a manner seen previously for the toaster oven, is via the thermocouple located in the middle of the back unheated wall of the AMF, which is made from a single refractory brick. A ¼" hole is drilled in the appropriate location, then a short piece of ¼" tubing inserted (recommend SS tubing – 0.035" wall) and a 1/8" thermocouple slid through the SS tubing, as shown. Refractory brick, also called fire brick, is the light yellow brick used for inside fireplaces and inside wood stoves. It is harder to find and more expensive than typical bricks, but typical bricks will not stand the heat and may crack or disintegrate over time (but you are welcome to try regular bricks – they will not explode, just fail over time). Some Lowes locations sell fire brick and it is available on ebay or outlets that sell wood stove components. The AMF uses a total of six standard fire bricks, which are 9" x 4.5" x 1.25", two for the base, two for the back wall and two for the front door.

The recommended insulation for the AMF is a medium temperature (max temp 650°C) industrial insulation sold under the brand name “Roxul” (http://www.roxul-rti.com/) and specifically the ProRox SL960 product line, which is available in a number of thicknesses. Just as the steel supplier was thrilled to sell you 6” of steel tubing, the industrial insulator is going to be crestfallen about selling a single panel of 2’ x 4’ x 2” ProRox SL960 – but they will for about $10. If the ProRox SL960 is not available, then “Ceramic Fiber Blanket” insulation from ebay will suffice at a higher cost and lower mechanical integrity.

The picture above shows the partially insulated prototype, with additional insulation needed on the right side. When completed, 2” to 3” of insulation should be on all sides of the AMF, including 2” on the back side – outside the fire brick that is the back wall of the AMF. The easiest way to cut the insulation is with a bare hacksaw blade and a straight edge – just slices right through it with a few passes. The sides of the AMF can then be covered with galvanized sheet metal, with a single piece of residential hot air ductwork, shown below and from Home Depot, being cut up to cover the sides and top.

The front of the AMF is constructed from two standing fire bricks, side by side, with another 2” of insulation outside the fire brick, which will get very hot during operation of the AMF. It is up to the individual fabricator as to whether the front panel is hinged or removable as a unit to access the interior. Fully insulated, the AMF does not cool off quickly, so some provision needs to be made to vent the unit to cool it and/or access the contents. As noted earlier, this memo is guidance and recommendations, with “the sole responsibility for construction and safe operation of any unit resides with the end-user”, so build and operate the AMF as you see fit.

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Not shown, but recommended, is a piece of ¼" hardware cloth above the burner to stabilize the crucibles in the AMF and keep the bottoms of the crucibles away from direct contact with the electric heating element. Alternately, a wire carrier can be made to facilitate inserting and removing the crucibles, typically in a group of four at once. Using the AMF, or any muffle furnace for that matter, takes a bit of practice due to the high temperatures and radiant heat emitted when the unit is opened. A nice long extension piece to hold the crucibles at length is recommended, and it is not uncommon to drop the occasional sample – just repeat it.

One little detail: The temperature control is exactly the same as the strategy for the toaster oven, except the temperature controller has to be able to control at higher temperatures (450°C & 550°C). Some controllers on ebay only allow 0°C to 400°C (REX-C*00 family and others) and will not work for the AMF. The current acceptable models have the brand name "mypin" and are available with the SSR output. The autotune function will dial in the control parameters – the settings do not have to be changed for the 450°C versus 550°C setpoints.

Finally, this guidance is my gift to you, but it does not include a seasons-pass on technical assistance. You are welcome to contact me for assistance, but be prepared to compensate me for the time involved. This is particularly relevant to what Garp called "Gradual Students": You are in Gradual School, where you gradually figure out you no longer want to be a student anymore. As such, suck it up and figure it out – the perseverance and techniques will serve your experimental career well in the future. Trust me, I have "been there and done that".

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