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**Objective:** The goal of these analytical procedures is to provide a standardized set of simple, affordable and accessible tests, which will generate metrics that characterize a given biochar and will facilitate the comparison of biochars. The suite of tests and the associated biochar metrics are intended for testing raw unmodified biochar, if such material is available. The metrics are also targeted at biochars that are being commercially transacted (bought and sold). In all cases, the biomass raw material, pyrolysis method, and any exceptional properties of the finished biochar, relative to other commercially available materials, should be clearly identified.

**Summary of “As Is Biochar” Procedure:** Using a representative free-flowing sample, measure the bulk density. Wet weight of “As Is” material in a weight of clean water in an appropriate container, seal and shake periodically for one hour, note As Is Contact Ratio. Settle or filter a portion of clear liquid and measure the pH and TDS/EC of the clear liquid phase; note any significant coloration. Calculate metrics for: **As Is Bulk Density, As Is Contact Ratio, Contact pH, Contact TDS/EC, and Contact Coloration of the Contact filtered water.**

**Summary of “Dried Biochar” Procedure:** Using a representative free-flowing sample, dry sufficient material at 150°C to perform the all the tests; Weight loss on Drying (at 150°C), Dry Bulk Density, Volatile Dry Weight Fraction (covered sample at 450°C), Inorganic Dry Weight Fraction (uncovered sample at 550°C), and measure Adsorption Capacity (either Gravimetric Adsorption Capacity Scan (GACS) or Propane Activity – see individual procedures). Calculate metrics for: **Weight loss on Drying, Dry Bulk Density, Volatile Dry Weight Fraction, Inorganic Dry Weight Fraction and Adsorption Capacity by (specify method).**

**Summary of “Wetted Biochar” Procedure:** Using a representative free-flowing sample, dry sufficient material at 150C to perform the all the tests. Measure weight loss on Drying (at 150°C) and Dry Bulk Density. Wet weight of dried biochar by boiling and cooling twice to fully wet biochar internal micropores with a weight of clean water (see procedure), note Extraction Ratio. Measure Wetted Biochar Density; settle or filter a portion of clear liquid and measure the pH and TDS/EC of the clear liquid phase; note any significant coloration. Weigh a sample of drained wetted biochar; dry at 150°C, weigh dried biochar and calculate Water Holding Ratio. Calculate metrics for: **Weight loss on Drying, Dry Bulk Density, Extraction Ratio, Wetted Biochar Density, Water Holding Ratio, Extract pH, Extract TDS/EC, and Extract Coloration of the Extraction filtered Extract.**

**Background:** The intent is to create a starting point that may evolve over time, but will start a database of biochar metrics that will allow producers, consumers and researchers to measure the materials with the same set of rulers. For many high quality biochars, this set of metrics may be all that is needed to satisfy the purchasing public. For biochars that have outlying metrics, the tests will highlight which aspects of the biochar merit additional consideration prior to utilization as a soil amendment or remediation treatment. Each core metric may be augmented by additional measurements, which will similarly evolve as the science and utilization of biochar matures.

Because biochar is a unique material, many of the test methods are unique to biochar, although standard analytical practices have been utilized to create the methods. It is believed that any commercial analytical lab can perform this core set of measurements, in addition to any college-level science/engineering practitioner, by carefully following the procedures provided. The rationale for the methods is briefly noted where appropriate, but the evaluation of the utility of this approach will occur subsequently in the published literature and the open marketplace.

**Recommended 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). Additional small items as specified in procedures.

**Overview of Procedures:** The procedures are broken into three sets of assays that are performed on a representative sample of “As Is” (not additionally dried), “Dried” or “Wetted” Biochar. The “As Is” 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, focusing on pH and TDS/EC concerns. The “Dried” tests dissect the biochar sample, after proper drying, into inorganics, volatiles and the calculated residual of resident matter, representing an estimate of stable graphitic biochar. The “Wetted” tests measure properties of the biochar relevant to how it will exist once it is mixed in the soil or in some other application where the biochar is saturated with water.

Density, the relationship between the volume of a sample and how much that volume weighs, is a fairly quick and simple measurement, but difficult to make to great accuracy. Since most biochars can be crushed and also fluffed up, the volume can change with handling and shipping. In addition, since biochar has a large amount of internal space (voidage, porosity), water can be

added to or evaporate from biochar without changing the volume. The combination makes density a difficult measure to make once and have it apply to all situations. For this reason, several density metrics are measured and they can be applied to the appropriate situations, as will be seen.

One final important point: drying biochar is not like drying most solids because of the micro-porous nature, a property biochar shares with activated carbons. For this reason, biochar is dried in accordance with ASTM D2867, "Standard Test Methods for Moisture in Activated Carbon". The principal feature for oven drying samples is to raise the oven temperature from the typical 105°C to the higher temperature window of 145°C to 155°C and to allow ample time to achieve stable dry weights. In addition, once the biochar sample is "dry", additional precautions have to be taken to prevent moisture from being taken back up by the dry biochar. The procedures are specific when these additional precautions are appropriate.

### ***DETAILED PROCEDURES***

**"As Is Biochar" Procedure** – 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 significant amounts of the biochar to an existing soil.

Note: This procedure assumes that the biochar will pass through a ¼" Hardware Cloth (coarse screen). If the biochar contains larger particles, such as chips, then the material needs to be reduced in size to pass through a suitable sieve or screen (virtually any screen, colander, flour sieve, etc. will do). The results should be reported as "Ground to pass through an approximately "x.x" mm screen size – original material was (brief description – chips, 2" briquettes, lump charcoal 1" to 3" nominal, etc)

- 1) Dry and tare a 4 oz canning jar (without the lid)
- 2) Fill a 4 oz canning jar with clean tap water to the top so the water level is even with the jar edge, weigh. **Calculate the volume of the 4 oz canning jar in cubic centimeters.**
- 3) Dry and tare the calibrated 4 oz canning jar (without the lid)
- 4) Measure "as is" biochar density by over-filling a 4 oz canning jar, settling the material by tapping the side of the jar, and sweeping the crown with a straight edge (or leveling to the top of the jar visually) and weighing in grams. **Calculate the "As Is Bulk Density" in grams per cm<sup>3</sup>.**
- 5) Dry and tare a 12 oz canning jar with lid.

- 6) Pour the contents of the 4 oz jar from step 4 into the 12 oz jar. Add sufficient clean water to fill the 12 oz jar to approximately  $\frac{3}{4}$  full. Note weight of clean water and calculate Contact Ratio (weight of water/weight of “as is biochar”).
- 7) Seal the 12 oz jar and shake the contents to completely wet the char. Vent the jar and let the contents settle. Repeat shaking/venting/settling every 15 minutes for 1 hour (a total of 5 shake/vent/settle cycles).
- 8) Filter the liquid from the top of the 12 oz jar through filter paper or paper towel to create at least 4 oz of clear liquid. Note any coloration in the Contact water.
- 9) Measure pH and TDS/EC of the filtered liquid. Note: TDS/EC measurements are not accurate if liquid contains suspended solids or exhibits significant turbidity
- 10) **Report the results of “As Is Biochar”** as:

Sample ID/Description: ID # or unique sample description

As Is Bulk Density = xx gr/cm<sup>3</sup>

Contact Ratio = yy (optional units of grams of water/gram of “as is biochar”)

Contact pH = zz (pH measurement)

Contact TDS/EC = zz reported in the instrument units of TDS/EC

Contact Coloration of the Contact filtered water = comment.

Report Contact Coloration as “Clear” if no obvious additional coloration is detected.

**“Dried Biochar” Procedure** – performed on dried biochar, including the proper drying method. These tests are focused on the biochar properties as manufactured, representing the impact of variable feedstocks and pyrolysis conditions. It is intended to assist the manufacturer in operating the production process and the consumer in comparing properties of available biochars.

Note: This procedure assumes that the biochar will pass through a  $\frac{1}{4}$ ” Hardware Cloth (coarse screen). If the biochar contains larger particles, follow the “As Is Biochar” guidance.

- 1) NOTE: If the Propane Activity method is being used for measuring the Adsorption Capacity (step 15), then sufficient material can be dried for that assay to provide dried biochar for the Volatile and Inorganic measurements – modify Step 2 to 5 as needed.
- 2) Dry and tare two 30 ml porcelain crucibles, including the crucible covers, for each biochar sample
- 3) Fill each crucible approximately one half full with biochar; record the weight of added material. Note: Biochar is damp or “as is” at this time. Calculate weight of “as is” biochar, referred to as the **Initial Biochar Sample Weight (IBSw<sub>t</sub>)**.
- 4) Cut an oversize square of aluminum foil and cover the top of the 30 ml crucible. Seat the foil on the top of the crucible with the heel area of the palm of the hand; punch a single

small hole in the center of the depression formed in the foil (a needle or straightened paper clip is recommended for punching the hole). Cover the crucible with the porcelain cover, compressing the aluminum foil over the edge of the crucible to form a uniform seal. Note: crucible is covered but not vapor tight to allow excess vapors to exit.

- 5) Heat the crucibles assemblies to a constant weight in a lab drying oven (a toaster oven is an acceptable alternative, due to wide temperature window for drying) controlled to an internal temperatures of 145°C to 155°C. Depending on how much moisture the damp biochar contains, the drying may take from between 1 hour and 4 hours. A minimum of two hours of drying is recommended unless the sample is known to contain minimal moisture. Any sample can be weighed periodically after the initial hour to determine acceptable dryness (less than 5% wt loss per hour); samples can be weighed hot to establish rate of weight loss, and then allowed to cool.
- 6) Cool the crucible assemblies to room temperature and weigh complete assemblies, recording this weight as the **Dried Crucible Assembly Weight (DCAwt)**.
- 7) Store the crucible assemblies in a desiccator until Volatiles Dry Weight Fraction assay and Inorganics Dry Weight Fraction assay can be performed.
- 8) **Volatiles Dry Weight Fraction (VOLdwf)**: Heat Dried Crucible Assembly for 2 hours in a preheated muffle furnace controlled to 450°C (+/- 10°C). Remove the crucible assembly after two hours and allow crucible assembly to cool to room temperature. Weigh the crucible assembly, recorded as **Exit Crucible Assembly weight (ECAwt)**, and then weigh the emptied crucible with aluminum foil cover and lid = **Crucible Assembly Tare weight (CATwt)**.
- 9) Calculate Drying Weight Loss weight fraction (DWLwf) =  $[1 - (DCAwt - CATwt) / IBSwt]$
- 10) Calculate Volatiles Dry Wt Fraction (VOLdwf) =  $[1 - (ECAwt - CATwt) / (DCAwt - CATwt)]$
- 11) **Inorganics Dry Weight Fraction (INORGdwf)**: Weigh lid of 30 ml crucible and invert the lid to make a shallow pan. Add a layer of dried biochar from a *Dry Crucible Assembly* to fill the crucible lid about  $\frac{3}{4}$  full and weigh the crucible lid plus biochar. Save the remaining dried biochar for the GACS Assay (see step 14). Calculate the weight of dried biochar in the crucible lid = **Dry Weight Biochar wt (DWBwt)**. Heat the crucible lid plus biochar in a preheated muffle furnace controlled to 550°C (+/- 10°C) until fully converted to pale gray ash (no salt and pepper appearance). Note: Inorganics Assay is performed at a different temperature than the Volatiles Assay. Note: the time required to fully ash the sample depends on the level of ash in the biochar sample, with low-ash biochars often taking longer to completely convert to ash. The progress can be monitored by opening the muffle furnace periodically and confirming the absence of any residual salt and pepper appearance of the residual ash samples.
- 12) After completely ashing the dried samples, remove them from the muffle furnace and allow them to cool to room temperature in a draft-free area to prevent any loss of ash

from the crucible lids. It is recommended to cover the ash samples with aluminum foil during cooling to prevent loss of dried material.

- 13) Weigh ash and lid, then discard the ash and weigh the crucible lid (dry wipe the lid to clean it). Calculate the weight of ash removed from the crucible lid = Dry Weight Ash weight (DWAwt).

**14) Calculate the Inorganics Dry Weight Fraction (INORGdwf) = DWAwt/DWBwt**

- 15) Gravimetric Adsorption Capacity Scan (GACS) can be measured on the remaining dried biochar. Alternately, the Propane Activity Uptake can be measured on larger sample of dried biochar – see **Appendix A on Adsorption Capacity Assay Options**.

- 16) (Optional) If Wetted Biochar Procedure will not be performed on a biochar sample, then steps 1 – 3 of the Wetted Biochar Procedure can be used to measure the “**Dried Bulk Density**” in grams per cm<sup>3</sup>.

- 17) **Report the results of “Dried Biochar”** as:

Sample ID/Description: ID # or unique sample description

Weight Loss on Drying (DWLwf) =  $[1 - (DCAwt-CATwt)/IBSw]$  wt fraction wet basis

Dried Bulk Density – from Step 16 or Wetted Biochar Procedure

Volatiles Dry Wt Fraction (VOLdwf) =  $[1 - (ECAwt-CATwt)/(DCAwt-CATwt)]$  reported on a weight fraction dry basis (dwf)

Inorganics Dry Wt Fraction (INORGdwf)= DWAwt/DWBwt wt fraction dry basis

Adsorption Capacity: GACS = R134a wt % Uptake at 100C *and/or* Butane Activity (C4A) by ASTM D5742 *and/or* Propane Activity (C3A) = wt % uptake at Ambient Temperature.

Note: Report Propane Activity Uptake at ambient temperature (near 25°C) or actual room temperature (if significantly different than 25°C).

**“Wetted Biochar” Procedure** – performed on wetted biochar, including the proper wetting method. 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 biochar and the wetting water in that environment. It is intended to assist the soil scientist and the consumer in comparing properties of saturated biochars and predicting subsequent soil impacts.

Note: This procedure assumes that the biochar will pass through a ¼” Hardware Cloth (coarse screen). If the biochar contains larger particles, follow the “As Is Biochar” guidance.

- 1) Dry sufficient biochar sample to constant weight in a drying oven or toaster oven at 145°C to 155°C to fill a calibrated 4 oz canning jar (see “As Is Biochar” Procedure, steps 1 & 2 for guidance how to calibrate a canning jar)

- 2) Dry and tare the calibrated 4 oz canning jar (without the lid)
- 3) Measure dried biochar density by over-filling a 4 oz canning jar, settling the material by tapping the side of the jar, and sweeping the crown with a straight edge and weighing in grams. **Calculate the “Dried Bulk Density” in grams per cm<sup>3</sup>.**
- 4) Dry and tare a 12 oz canning jar with lid.
- 5) Pour the contents of the 4 oz jar from step 3 into the 12 oz jar and record the weight of char added as **Dry Biochar weight (DryBwt)**. Add sufficient clean water to fill the 12 oz jar to approximately  $\frac{3}{4}$  full. Record total weight; calculate the weight of water added = (H<sub>2</sub>Owt). Calculate the **Extraction Ratio = (H<sub>2</sub>Owt) / (DryBwt)**.
- 6) Seal the 12 oz jar and heat in a covered pan with a shallow water layer to boiling. Hold for at least 15 minutes at a gentle boil. Remove the 12 oz jar from the pan, swirl the jar to wet any floating biochar, vent the jar to relieve any excess pressure, reseal.
- 7) Allow to cool in air to below 40°C (warm to the touch, no longer hot). Periodically swirl the jar to wet any floating biochar.
- 8) Refill the covered pan with cold water and allow the once-heated 12 oz jars to equilibrate with the water in the covered pan prior to reheating.
- 9) Heat the 12 oz jar a second time in a covered pan with a shallow water layer to boiling. Hold at for at least 15 minutes at a gentle boil. Remove the 12 oz jar from the pan, swirl the jar to wet any floating biochar, vent the jar to relieve any excess pressure, reseal.
- 10) Allow 12 oz jar with wetted biochar to cool in air to below 40°C (warm to the touch, no longer hot). There should be minimal floating biochar after the second heating, although small amounts are allowed. Weigh cooled container to determine if the amount of extraction water (step 5) has changed; if total weight (step 5) has changed, recalculate the **Extraction Ratio = (H<sub>2</sub>Owt) / (DryBwt)**.
- 11) Once cooled below 40°C, shake the 12 oz jar to fully mix the wetted biochar into a slurry, then allow the contents to settle without moving the jar. Allow the 12 oz jar to settle for at least one hour and until a clear partitioning between settled solids and relatively solid-free liquid is formed. Mark the level of the solids-liquid interface on the side of the 12 oz jar or measure the location of the level from either the bottom or top of the jar (see step 13).
- 12) Prepare a paint filter assembly to allow the filtering of the wetted biochar. Slurry the contents of the 12 oz jar and pour the majority of the water-biochar slurry into the paint filter. Allow the slurry to drain for one hour at room temperature. Save the filtrate (see step 21)
- 13) Clean, dry and tare the jar from step 11. Fill the jar with clean tap water to the level recorded in step 11 and weigh. Calculate the settled wetted biochar volume (SWBml) and calculate the **Wetted Biochar Density (WBD)** in gms/cm<sup>3</sup> = (DryBwt)/ (SWBml)
- 14) Dry and tare a 30 ml porcelain crucible, including the crucible cover.

- 15) Fill the 30 ml crucible approximately one half full with drained biochar from step 12; use granular material, freely drained, avoiding any surface “mud layer” that represents finely divided biochar capable of trapping interstitial free liquid; record the weight of added material. Note: These conditions reflect the (Maximum Drained) Water Holding Limit of the biochar. Calculate weight of wet biochar, referred to as the **Wetted Biochar Weight** (WetBwt).
- 16) Cut an oversize square of aluminum foil and cover the top of the 30 ml crucible. Seat the foil on the top of the crucible with the heel area of the palm of the hand; punch a single small hole in the center of the depression formed in the foil ((a needle or straightened paper clip is recommended for punching the hole). Cover the crucible with the porcelain cover, compressing the aluminum foil over the edge of the crucible to form a uniform seal. Note: crucible is covered but not vapor tight to allow excess vapors to exit.
- 17) Heat the crucibles assemblies to a constant weight in a lab drying oven (acceptable alternative, due to wide temperature window, is a toaster oven) controlled to an internal temperatures of 145°C to 155°C. The recommended drying time is 4 hours. A minimum of two hours of drying is recommended. Any sample can be weighed every one-half hour to a constant weight; samples can be weighed hot to establish constant weight, and then allowed to cool.
- 18) Calculate the weight of dry biochar by weighing the dry crucible assembly, then removing the biochar (save the dry biochar sample), dry cleaning the crucible and weighing crucible, aluminum foil and lid together. The weight difference is the **Dry Biochar Weight** (DryBwt).
- 19) Calculate the **Water Holding Ratio (WHRwt) = (WetBwt - DryBwt)/(DryBwt) = (WetBwt)/(DryBwt) - 1**. This metric represents a quantitative measure of the biochar’s impact on soil water holding capacity by measuring the maximum weight of water contained within the biochar particles in a given weight of dry biochar. Note: Due to the unique mechanical and physical properties of biochar (microporous solid that is much less compressible than typical soil components), the impact of biochar is poorly and inaccurately quantified by traditional soil pressure plate methods.
- 20) (Optional) The dry biochar sample from step 18 can be tested for GACS adsorption. This GACS assay can be compared to the GACS assay of step 14 of Procedure B to gain insight to the change in adsorption capacity, if any, upon the biochar being wetted in the soil.
- 21) Filter the filtrate from step 12 through filter paper or paper towel to create at least 4 oz of clear liquid. Note any coloration in the Extraction filtered water.
- 22) Measure pH and TDS/EC of the filtered liquid from step 21. Note: TDS/EC measurements are not accurate if liquid contains suspended solids or exhibits significant turbidity.
- 23) (Optional) Measure the brix, TOC, COD or BOD5 of the filtered liquid from step 21 as a relative measure of leachable soluble organics present in the original biochar.

24) Report the results of “Wetted Biochar” as:

Sample ID/Description: ID # or unique sample description

Weight Loss on Drying, as described in step 1.

Dried Bulk Density in grams per cm<sup>3</sup>, as described in step 3.

Extraction Ratio, as described in step 5, (optional units of grams of water/gram of “wetted biochar”)

Wetted Biochar Density (WBD) in gms/cm<sup>3</sup>, as described in step 13.

Biochar Water Holding Ratio, as described in step 19.

(Optional) Wetted GACS Adsorption Capacity, as described in step 20.

Extract pH, as described in step 22.

Extract TDS/EC in instrument units of TDS/EC, as described in step 22.

Extract Coloration of the Extraction filtered Extract = comment.

Report Extract Coloration as “Clear” if no obvious additional coloration is detected.

(Optional) Additional Extract properties, as described in step 23.

## Appendix A on Adsorption Capacity Options

**Adsorption** is the physical phenomenon where biochar emulates the distinguishing property of activated carbon, which is a non-ionic property whereby soluble organics and chemicals in the soil water are preferentially attracted to the internal surfaces of the biochar. The energy of adsorption is highly dependent on the specific chemical being adsorbed and the local characteristics of the solid surface where the adsorption occurs. Overall, adsorption is a highly dynamic and complicated process, but a very important and unique one in predicting the impact of biochar in soils. The property of adsorption is usually quantified by measuring how much of a particular adsorbate is taken up by the adsorbent under the controlled conditions, expressed as an amount of adsorbate uptake per unit of adsorbent in appropriate units.

The recommended test method is the weight increase due to the adsorption of pure R134a, a fluorocarbon refrigerant, on dry biochar at 100°C at a pressure of one atmosphere, known as the Gravimetric Adsorption Capacity Scan (GACS) assay. Unfortunately, the GACS instrument is not commercially sold, but can be constructed for a reasonable budget and modest effort. Instructions for constructing the instrument are available by searching the phrase “*GACS on a Budget*” or at [www.drtylud.com/?resource=prt16730](http://www.drtylud.com/?resource=prt16730).

Another adsorption assay is the uptake of n-butane on dry biochar at 25°C at a pressure of one atmosphere, called the “Biochar Butane Activity” after the ASTM D-5742 Standard Test Method

for Determination of Butane Activity of Activated Carbon. This method is available at many commercial analytical labs for fee per assay. An acceptable alternative, less expensive and more accessible, is to perform the Butane Activity method using Propane (resulting in a method called the "Biochar Propane Activity, based on ASTM D-5742"). A propane torch or camping burner is modified by removing the burner head to create a controlled source of propane. The propane source is adapted with  $\frac{1}{4}$ " ID flexible tubing (Tygon or equal) that terminates in a inflation needle used to pressurize sports balls (soccer balls, basketballs, etc).

The biochar sample container is made from a 6 oz tomato paste can, with one end removed with a "Safe Cut" can opener, which creates a close fitting lid. A hole slightly larger than the inflation needle is drilled in the side of the can at the closed end and a similar size hole is made in the center of the removable lid (see figures). The dried and weighed 6 oz can is filled with a biochar sample to approximately  $\frac{3}{4}$  full, then the entire assembly is dried in the same manner as the 30 ml crucibles (oven at 145°C to 155°C to constant weight). The dried container assembly is then allowed to cool, weighed, and the weight of dry biochar calculated.

The inflation needle is inserted in the bottom of the biochar container and the assembly is purged with room temperature propane for 30 minutes, with the excess propane exiting the center opening of the top. The exiting propane can be ignited to allow a minimal but constant flow of propane to exit the container (see figures below). The flame is extinguished for the last 10 minutes to avoid heating the top of the propane container. The propane equilibrated assembly is weighed and the weight gain due to the propane adsorption calculated and expressed as Propane Activity in wt C<sub>3</sub>/wt of dry biochar, expressed as weight percent. Note: covering the lower opening in the container, where the inflation needle was removed, with electrical tape will inhibit the loss of propane from the container and facilitate accurate weight measurement of the saturated container. The weight of the calculated propane weight gain should be adjusted for the impact of any additional tape. Typical propane uptake is 1 to 10 weight percent of the dry biochar weight, depending on the quality of the biochar with respect to adsorption capacity.

