



## Guidelines for Specifications of Biochars - Meeting Minutes

Meeting Title:	Working Group meeting to review comments from Oct 15 – Nov 15, 2011 comment period
Date:	December 6-21, 2011
Location:	Email discussion

### Attendees:

Debbie Reed, United States  
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Stephen Joseph, Australia  
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Franco Miglietta, Italy  
Amran Salleh, Malaysia  
Balwant Singh, Australia  
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### Agenda:

1. Carbon and Ash comment summary and discussion
2. Toxin Assessment comment summary and discussion
3. Enhancement Properties comment summary and discussion
4. Determining Feedstock Changes comment summary and discussion



# Issues Group 1: Carbon and Ash

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## Issues Summary of Comments Received During Oct 15 – Nov 15 Comment Period

### 1 Carbon and Ash

Recommendation: Based on the comments below, we are proposing the following approach to carbon and ash content measurements and reporting within the Guidelines:

- i. Declaration of Ash Content
- ii. 20% Carbon Recovery recommended minimum
- iii. H:C ratio threshold of <0.6
  - Molar mass, metric mass or a volume ratio? Which should be used?
- iv. Corg (necessary for item iii) declaration
- v. Cinorg (necessary for item iv) declaration

The above recommendations are based on comments received during this most recent comment period. Scientific rationale and justification for these recommendations have been provided. Proposed changes to this approach should be accompanied by justification and scientific support. We also request assistance in determining the appropriate methodologies and reporting metrics to use for analyzing these parameters, if they are new.

#### 1.1 Ash Content

- a. Many feedstocks likely to not meet max. ash threshold of 50% (including rice husk, wood waste, log yard waste (soil & dirt), dirty corn stover)
- b. Ash has agronomic benefit, why not declaration rather than threshold?
- c. 50% max ash content prevents selling gasifier ash as biochar, incensing pyrolysis over gasification.
- d. High-ash feedstocks can be blended with lower-ash feedstocks to reduce total ash.
- e. Need to CLEARLY distinguish ash from soils (diluent) in a testable manner
- f. Disagreement over feedstock blending (i.e. adding wood to poultry litter to reduce ash content)
- g. 90% ash is not biochar
- h. Revise max. limit to 60% on an oven-dry basis, eliminate carbon correction and use total ash instead. Nominal total carbon content then expected to be ~35%.
- i. Add specification for minimum carbon recovery % (Organic C% of biochar x biochar yield/ organic C% of feedstock) where biochar yield = dry biochar mass/dry feedstock mass.
- j. Ash definition should be made more clear. Ash and mineral content are not the same thing, and must be clearly delineated/identified as such.
- k. Inclusion of an energy balance within biochar production process to help identify high-ash-producing processes & feedstocks and control ash content in biochar – To be added to Sustainability Guideline criteria.

#### SUMMARY OF ISSUE:

- 50% seems too stringent as a maximum ash content
- Raise maximum to 60% or higher, or to make ash reporting a declaration rather than a threshold
- Definition of ash should be clearly laid out so as to identify what does and does not constitute ash (chemically or otherwise) including diluents, mineral content, etc. Refer to definition section within level methodology table.



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### 1.2 Minimum C content vs maximum Ash content

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- a. Parameter should be C content based on dry-weight (oven-dried, must provide an appropriate temperature/time for oven-dried wt). This should be used instead of Ash, Oxygen and Hydrogen content. Carbonates (CO<sub>3</sub>) should be included in C content reporting. Instead of 50% Minimum, C content could indicate a sliding scale of quality from high at 50%+ to low with lower percentages.
- b. Minimum carbon % should be set, b/c max ash% doesn't guarantee a set minimum C content (diluent etc.). Need a more direct measurement of C content & allow for easier communication.
- c. Minimum carbon recovery of 20% from original feedstock as additional requirement. (feedstock-biochar yield ratio). Requiring feedstock carbon content analysis.
- d. Must better-define and -distinguish terminology between different forms of C (inorganic or mineral C like in carbonates, ash-C, "functional" organic C, etc.) if reporting C content. Or, is there a benefit to knowing different C forms? What terms should be used? (inorganic vs mineral vs carbonate; organic vs bio-available, etc.)
- e. Minimum C is more easily understood/recognized vs ash and H:C etc.
- f. %ash (or non-char fraction???) declaration necessary. %C org measure might provide sufficient information for end-users & carbon markets.

#### SUMMARY OF ISSUE:

- Opinions seem to indicate need for some form of C reporting
  - Average customer/ carbon market operation will want easy-to-understand metrics – explanations should be provided.
  - Clear justification of chosen parameters is necessary
  - If interim constituent reporting (H:C, O, H, ash) remains in document, reasoning/explanation should be provided.
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### 1.3 Stable C

- a. Stable C would be more useful than Inorganic C (which is seen as irrelevant to usefulness of biochar as C credit and ag application)
- b. Labile C instead of H:Corg
- c. "organic" and "inorganic" need clear definitions and must be clearly (unambiguously) used within the document. Alternative words could be used: fixed vs labile, stable vs mobile, charred vs uncharred, mineral vs bio-available, etc. (see similar comment (d) in Min. C vs Max. Ash)
- d. Carbonate determination needs a separate analysis than ASTM D1762 – analytical & cost burden consequences from this change?
- e. Carbon analysis for carbon credit/capture necessary. Will such a measure require exclusion of carbonates, or are they ok to be left in?
- f. Semantics of using organic/inorganic. Could "carbonate carbon" or "mineral carbon" be used instead?

#### SUMMARY of ISSUE

- Word use will need some clarification – with appropriate definitions emphasized within the document
- Should Stable C address inorganic, organic or a combination of the two types of carbon?



- Analytical cost burden of multiple C analyses should be considered when deciding on this parameter (and the other C measurements).
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#### 1.4 H:C Threshold

- Should be a declaration, not a threshold – stable C is insufficient for reporting to carbon markets because whole lifecycle analysis is needed to determine C offset value.
- Following Spokas' approach is good – straightforward & simple. As a mass-ratio, H:C should be <0.6.
- For time-being H:C is sufficient, following Spokas. May want to consider thermogravimetric analysis of resistance to thermal oxidation in the future, but needs more proving first.
- The average consumer is not going to understand H:C – what it's purpose is, nor how to interpret it. Should this parameter come with some further explanation in an appendix?

#### SUMMARY OF ISSUE

- Inclusion of H:C is sufficient for the time-being
- Ratio should be specified as mass ratio or molar or volumetric so that consistent values are reported.
- Greater explanation of the purpose of H:C ratios within biochar analysis is required.

#### 1.5 Accuracy of H:C when ash content is high

- Following logic of H:C and O:C ratios being closely related with low-ash biochars <50% ash, will different analysis be needed to assess C stability of high-ash biochars?
- Modified dry ashing with nitric acid digestion and hydrogen peroxide oxidation holds promise for assessing total element content, but there is no currently available document. What documentation is currently available on such analyses?

#### SUMMARY OF ISSUE

- Justification or more detailed explanation would be beneficial
- Use currently-available, published, peer-reviewed and easily-accessed information for all methodologies.

### **DISCUSSION:**

#### Minimum C recovery:

A recovery minimum is not consistent with a material properties guideline. Better stick to parameters that can be measured in the biochar. One way to combine these two approaches is to choose a minimum Corg content in a way that the C recovery is high enough for even low-ash feedstocks (since there is a relationship between the two). Inclusion of an energy balance within biochar production process to help identify high-ash-producing processes & feedstocks and control ash content in biochar is not appropriate either for these Guidelines – Has to be in the sustainability guidelines, not here.

#### H:C ratio:

H:C ratio correlates well with C stability – it is the best analysis we currently have. It should always be a molar ratio. Threshold of 0.7 molar ratio is the correct level. If we base on H:Corg, that removes concern about H:C accuracy for high-ash biochars.



Minimum Corg content: A lower limit of Corg makes more sense than an upper limit of ash. ~~Should be organic C and not total C to avoid misrepresentation of carbonates. If there is a~~ lower limit of Corg, then we do not need a limit of ash, just declaration. Threshold for minimum Corg could be 10%, 20% or 30%.

Ash:

With Corg minimum content, ash can be declaration only.

Stable vs. labile carbon:

No good way to measure long-term stability in a short-term analysis. Volatile C from proximate analysis is lost by heating, but it has not been correlated with degradability in soil.

Carbonates:

The methodology currently proposed by the guidelines says as follows: "apply loss on ignition (ASTM D1762-84) to ascertain total non-carbonate ash, then back inorganic carbon as carbonate" As not all carbonates will be eliminated with this methodology, it would be better to stick to the standard methodology for the determination of ashes in wood charcoal (ASTM D1762-84) and eliminate the addition of inorganic C as carbonates. Also no need to report inorganic C -- it may confuse the consumer to have inorganic C, lime equivalence and pH.

## Issues Group 2 – Toxins

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### Issues Summary of Comments Received During Oct 15 – Nov 15 Comment Period

#### 2 Toxin Analysis

##### 2.1 Worm Avoidance

- a. Earthworms will always avoid new environments, and thus makes the earthworm avoidance test unreliable.
- b. Concerns over universality and accessibility of OECD standard soil types.
- c. Concerns with variability of biochar moisture and influence on substrate avoidance by earthworms
- d. Is the earthworm avoidance test useful? What information does it actually provide? What other tests can provide similar information? (Repeat of similar comment under Dioxins/Furans and PAHs)

#### SUMMARY OF ISSUE

- Need to determine whether earthworm avoidance test needs to be adapted for specific conditions (e.g. moisture, particle size) relevant to biochar
- Determine availability of test methodology materials/ingredients.
- Information needed on whether or not biochar can be submitted to Earthworm Avoidance test to an equivalent degree as other materials for which the test was designed.

##### 2.2 PAH

- a. Many low-quality chars, regardless of feedstock, contain PAHs, why not require PAH analysis at all levels?



b. Bad practices can result in PAH with ~~every kind of~~ feedstock. PAH should apply to at least Level 2 if not lower (unless earthworm/germination analysis is adequate to cover this concern & enough explanation is provided to that effect)

c. The selected methodology should include a distinction between high and low molecular weight components (e.g. naphthalene vs benzopyrene) to ensure appropriate materials are being analysed. Methods should follow existing soil amendment regulations in US, UK etc.

#### SUMMARY OF ISSUE

- Determine if PAH testing should be moved to earlier levels.
- Selected methodology should include thorough analysis of soil-relevant PAH components, and follow other soil amendment regulations.
- Do earthworm avoidance and plant germination tests provide an effective analysis for PAHs of concern?

#### 2.3 Dioxins & Furans

a. Combine dioxin & furan thresholds into one value, expressed as TEQ for dioxins, furans and dioxin-like PCBx, expressed as pictograms TEQ/g of dry matter. Threshold should be set at 5pg TEQ/g.

b. Rebuttal to point a: soil threshold needs to be higher than individual amendment regulations, as amendments are likely to be added year after year, while soil thresholds relate to total soil toxicity in perpetuity.

c. Should Dioxin/Furan analysis be necessary for MSW? All feedstock types including manure?

#### SUMMARY OF ISSUE

- Can/should analysis of dioxins and furans be combined?
- Threshold level should be based on typical soil amendment regulatory thresholds (not total soil content thresholds)
- Revisit level- placement of dioxin/furan analysis relevant to feedstock type.

#### 2.4 Feedstocks

a. Consider adjusting feedstock placement within unprocessed and processed categories. Do manures and poultry litters require the same level of stringent assessment as MSWs and industrial sludges?

b. The IBI would like to avoid pushing manures to a higher test level category unless appropriate research, and justification is provided.

#### SUMMARY OF ISSUE

- Consider moving manure – feedstocks to the unprocessed feedstock category.

#### **DISCUSSION:**

New research results show that the Earthworm Avoidance Test is reliable and that concerns about worms reacting to a new material are probably worms reacting to inadequate wetting of the substrate. Need to cite new results to give guidance about adequate moisture in the test.



## Issues Group 3 - Biochar Enhancement Properties

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### Issues Summary of Comments Received During Oct 15 – Nov 15 Comment Period

#### 3.1 EC Electroconductivity

- a. EC is critical for seed germination and should be included in Level 1
- b. EC soil:water ratios of 1:5 are commonly used in soil testing. Will values be higher because of the difference in water content or water-holding-capacity in biochars?
- c. EC can vary with particle size. Grinding and sieving to a specified particle size should accompany EC analysis if reproducible results are to be obtained.
- d. Method should provide a recommended w:w biochar:water ratio that is appropriate to biochar's adsorbing ability. More applicable water ratio is necessary. (i.e, is 1:5 appropriate biochar:water ratio for accurate analysis, or should it be higher? If so, what level?)

#### SUMMARY OF ISSUE

- Consider moving EC to Level 1
- Directions are needed within methodology to require standardized particle size for all analyses (grinding & sieving)
- Methodology should also prescribe a biochar:water ratio (weight:weight) to address the adsorbing ability of biochar

#### 3.2 pH

- a. The currently selected methodology (Ahmedna et al) may produce low ionic-strength solutions and therefore unstable instrument readings. An addition of KCl would alleviate this, but must be specified in the methodology.
- b. Methodology should specify a requirement for controlled evaporation when heating samples (suggested in methodology). May otherwise cause uncontrolled evaporation, affecting the replicability of results.
- c. pH needs to stabilize following treatment prior to sampling: provide a specification for length of time between treatment and pH analysis (e.g. 3 hrs).
- d. pH can vary with particle size. How will this be addressed? Standard sieving and grinding to a specific particle size should be made one of methodology requirements.

#### SUMMARY OF ISSUE

- Methodology needs to be improved or augmented to provide standardized (or prescribed) requirements for KCl, controlled evaporation while heating, and stabilization time after process prior to pH analysis.
- Directions are needed within methodology to require standardized particle size for all analyses (grinding & sieving)

#### 3.3 Surface area and sorption

- a. Perhaps N<sub>2</sub> or CO<sub>2</sub> sorption surface area, rather than iodine for better performance prediction.
- b. Perhaps consider the EGME method used in soil science (Carter et al. 1986. "Specific surface methods of soil analysis. Chpt. 16 Agronomy No 9 pt 1 2nd ed. American Society of Agronomy, Madison WI



## SUMMARY OF ISSUE

~~Is Iodine sorption the most applicable analysis? Would others be better suited? (if citations/methodologies exist)~~

### 3.4 Available N and Available P

- a. Mineral N determination by KCL extraction and available P by formic acid extraction is not as good as weak acid extraction for both
- b. The relationship between formic acid extractable P and plant-available P is currently not published or freely available.

## SUMMARY OF ISSUE

- Is KCl and formic acid extraction the best methodology for this analysis?
- A documented methodology is needed (In Press is not acceptable)

## DISCUSSION:

Electrical conductivity and pH:

EC is an interesting property, but not needed in Level 1 since we have ash declaration. Can use same analysis procedure as for pH. Accuracy of pH measurements for biochar can be improved with slight alteration of analysis methods. Need to specify dilution rates, particle size and time in methods appendix.

Surface area and sorption:

These properties are important to many functions of biochar. Iodine is a poor choice because it reacts chemically with double bonds, dangling bonds and easily oxidized functional groups on the surface (i.e., chemisorption), which are abundant. Either N<sub>2</sub> or CO<sub>2</sub> would be better. Recommend using the universal N<sub>2</sub> BET method, despite problems with volatiles in some biochar samples.

## Issues Group 4: Determining Feedstock Changes

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### Issues Summary of Comments Received During Oct 15 – Nov 15 Comment Period

Commenters have asked for a more explicit definition for "material change in feedstock". For instance, if the feedstock is "wood chip" but the composition changes over time from pine to spruce or oak, we know this will have some effect on the product, but is it enough to require a new round of tests? What level of change in a feedstock produces enough difference in the biochar characteristics to require reporting?

Here are a few hypotheticals to consider:

- Change from oat straw to wheat straw?
- Change from 30% pine and 70% fir to 30% fir and 70% pine?
- Change from poultry litter with 10% wood chip bedding to poultry litter with 25% wood chip bedding?
- Change from chicken manure to turkey manure?





~~We really do not want to have to provide a list of discrete feedstocks. It will not be complete no matter how comprehensive it is. We would prefer to give general guidelines that are meaningful but not overly burdensome.~~

It has been suggested that for unprocessed feedstocks that species be the minimum level of differentiation. But is it really necessary to distinguish between different kinds of straw? If not species, what about genus or family? All pines, spruces, firs and cedars are in the family Pinaceae. Are there enough differences between them to produce significantly different biochars?

On the other hand, the family Gramineae (the grasses) includes wheat, corn, sugarcane and bamboo. That seems too broad. But there is the sub-family Pooideae, that includes wheat, barley, oats, brome-grass (*Bromus*), reed-grasses (*Calamagrostis*) and many lawn and pasture grasses, and that seems like a reasonable taxonomic group.

### **DISCUSSION:**

Need to find existing feedstock classification system to create broad, simple categories of feedstocks that will produce biochar with closely similar properties – categories like hardwood and softwood. Could use FAO crop type database, but a preferable database is Phyllis database of biomass feedstocks.