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Comparison of wet digestion and dry ashing methods for total elemental analysis of biochar

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Introduction

Quantification of biochar elemental content can inform effects on soil health and fertility [1] as well as losses during pyrolysis. Analysis of total elemental content can be accomplished with ICP-AES, but requires complete dissolution into a liquid matrix [2,3] and decomposition of organic matter that may affect analysis [4]. Numerous methods have been proposed to prepare plant samples for elemental analysis [5]. Dry ashing is simpler and safer than wet digestion methods, however, the high temperatures employed may introduce error due to volatilization [3] and yet remain insufficient to decompose pyrolytic organic materials [6,7]. Wet digestion methods operate at lower temperatures but employ potentially dangerous inorganic acids [8]. Biochar recalcitrance may resist decomposition by strong oxidizers [9,10]. The purpose of this paper is to identify safe, reliable, and accessible biochar preparation methods for total elemental analysis by ICP-AES.

Materials and Methods

Three biochars were chosen with contrasting properties: (1) corn stover pyrolized at 300°C (Corn300), (2) oak wood at 600°C (Oak600), and (3) poultry manure with sawdust at 600°C (Poultry600). Published wet digestion (PWD) and published dry ashing (PDA) methods for plant tissue, modified wet digestion (MWD) and modified dry ashing (MDA) methods to accommodate biochar recalcitrance, and a perchloric and nitric acid wet digestion (PNW) were used to decompose samples for ICP-AES analysis.

Results and Discussions

MDA was either the most precise method, or demonstrated relative standard deviation (%RSD) within 3.7% of the most precise method, for recovery of K, S, Ca, Mg, Mn, and Zn from Corn300 and Oak600.

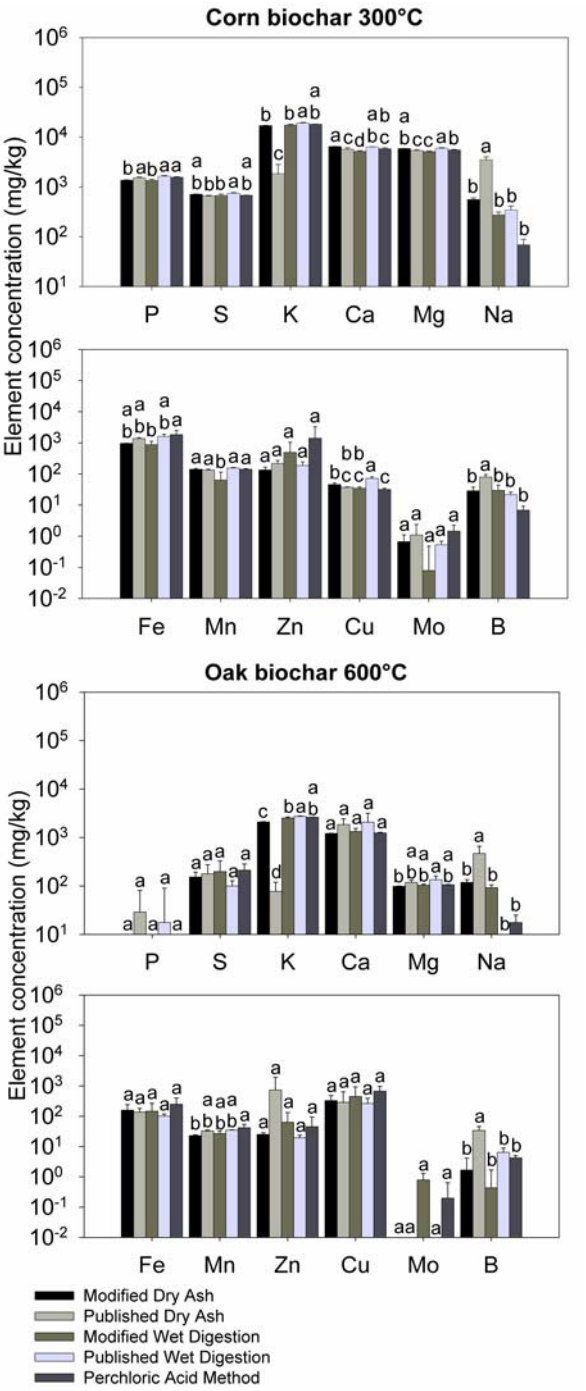

Figure 1. Total nutrient contents of corn stover biochar produced at 300°C and oak wood biochar at 600°C obtained by different digestion methods.

Additionally, MDA was the most precise method for P and Fe from Corn300. Recovery of P, K, S, Ca, Mg, Fe, Mn, and Zn from Poultry600 was 10 to 100 times lower with PWD and PDA than when using all other methods. MDA returned lower levels of Ca than PNW, otherwise there were no differences in recovery of P, K, S, Mg, Fe, Mn, and Zn between PNW, MWD, or MWA from Poultry600. PDA returned significantly more Na than any method for Corn300 and Oak600, suggesting contamination from borosilicate glassware [11,12].

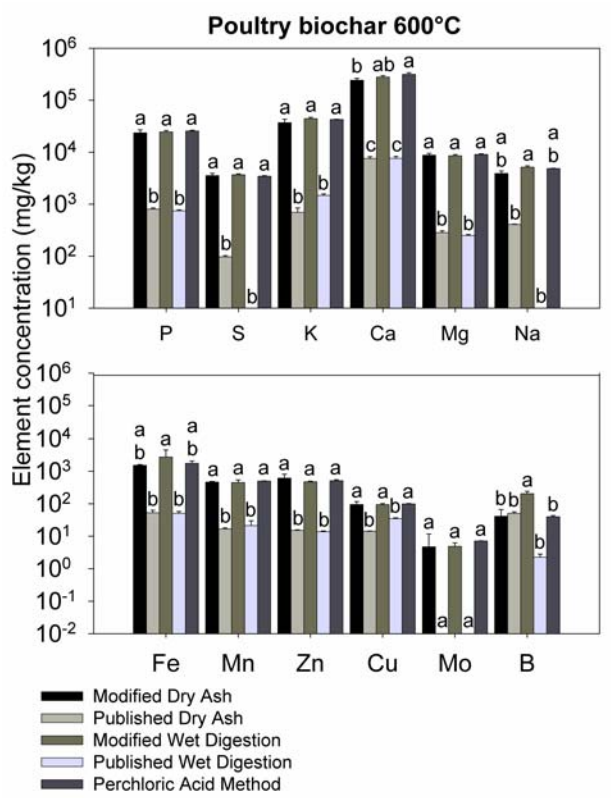

Figure 2. Total nutrient contents of poultry manure biochar produced at 600°C obtained by different digestion methods.

Conclusions

Trace elemental analysis would benefit from closed vessel methods that eliminate volatilization losses [13] or vitreous silica or platinum labware to reduce contamination [14].

MDA is a comparatively safe and effective method to prepare biochar for ICP-AES analysis of plant macro and micronutrients utilizing accessible labware and simple equipment.

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[1] Chan, K. Y.; Xu Z. H. 2009. In: Biochar for Environmental Management, J. Lehmann and S. Joseph (Eds.), Earthscan, Sterling, Va, pp. 67-84.
[2] Huang, C. L.; Schulte E. E. 1985. Commun. Soil Sci. Plant Anal., 16, 943-958.
[3] Ali, M. W.; Zoltai S. C.; Radford F. G. 1988. Can. J. Soil Sci., 68, 443-447.
[4] Wasilewska, M.; Goessler, W.; Zischka, M.; Maichin, B.; Knapp, G. 2002. J. Anal. At. Spectrom., 17, 1121-1125.
[5] Jones, J. B. 2001. In: Laboratory Guide for Conducting Soil Tests and Plant Analysis, CRC Press LLC, Boca Raton, Fl, pp. 203-207.
[6] Hsieh, Y. P. 2007. J. AOAC Int., 90, 54-59.
[7] Tartarelli, R.; Giorgini M.; Ghetti P.; and Belli R. 1987. Fuel, 66, 1737-1738.
[8] Oliva, S. R.; Raitio H.; Mingorance M. D. 2003. Commun. Soil Sci. Plant Anal., 34, 2913-2923.
[9] Masiello, C. A. 2004. Mar. Chem., 92, 201-213.
[10] Hammes, K.; Schmidt, M. W. I.; Smernik, R. J.; Currie, L. A.; Ball, W. P.; Nguyen, T. H.; Louchouarn, P.; Houel, S.; Gustafsson, Ö.; Elmquist, M.; Cornelissen, G.; Skjemstad, J. O.; Masiello, C. A.; Song, J.; Peng, P.; Mitra, S.; Dunn, J. C.; Hatcher, P. G.; Hockaday, W. C.; Smith, D. M.; Hartkopf-Fröder, C.; Böhmer, A.; Lüer, B.; Huebert, B. J.; Amelung, W.; Brodowski, S.; Huang, L.; Zhang, W.; Gschwend, P. M.; Flores-Cervantes, D. X.; Largeau, C.; Rouzaud, J. N.; Rumpel, C.; Guggenberger, G.; Kaiser, K.; Rodionov, A.; Gonzalez-Vila, F. J.; Gonzalez-Perez, J. A.; de la Rosa, J. M.; Manning, D. A. C.; López-Capél, E.; Ding, L. 2007. Global Biogeochem. Cycles, 21, GB3016.
[11] Mizuike, A.; Iino, A. 1981. Anal. Chim. Acta, 124, 427-430.
[12] Morris, A. P.; Dosdale, T.; Knott, P.; Short, N. R. 1980. J. Non-cryst. Solids, 38, 539-543.
[13] Sturgeon, R. E. 2000. Commun. Soil Sci. Plant Anal., 31, 1479-1512.
[14] Zief, M. 1980. ASTM Spec. Tech. Publ., 708, 66-77.