QUARTERLY PROGRESS REPORT

April 1, 2017 to June 30, 2017

PROJECT TITLE: Electromagnetic Wave-Induced Heavy Metal Removal for Dewatered Biosolids Composting

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http://www.eng.famu.fsu.edu/~gchen/index_files/Page570.htm

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Project Summary

Heavy metal contents in dewatered biosolids prevent the nutrient-rich organic materials from being used in composting. Variable chemical extraction methods have been investigated for heavy metal extraction from biosolids and the results are not very satisfactory owing to the high costs as well as the potential environmental impact. Electromagnetic wave (EW)-induced heavy metal removal offers a sound solution for this problem.

Work Accomplished during This Reporting Period

Heavy Metal Extraction from Biosolids
Biosolids form Graceville Wastewater Treatment Plant (Graceville, FL) were collected. After collection, the biosolids were oven dried to constant mass at 105^{o}C. The dried biosolids were ground and sieved through #10-mesh sieves, and then stored in dry condition at room temperature for the following experiments.

Six heavy metals, including copper, zinc, nickel, lead, cadmium and iron were extracted through hot plate-assisted Aqua Regia digestion following the standard procedure. The extracted metal concentrations were measured by a Microwave Plasma-Atomic Emission System (MP-AES). The measured cadmium concentrations were below the 5ppb, which was the detection limit of the MP-AES system. Thus, cadmium was considered not detectable in the biosolids. The concentrations of other five metals in the biosolids are listed in Table 1.

Table 1. Metal Contents in the Biosolids

Metal Species	Cu	Zn	Ni	Pb	Cd	Fe
Content (mg/kg)	728	573	51	93	ND	11964

Three conventional chemical extraction methods were conducted for heavy metal removal, including inorganic acid, organic acid and EDTA. Triplicate experiments were conducted for each method and the average results are presented in Figure 1.

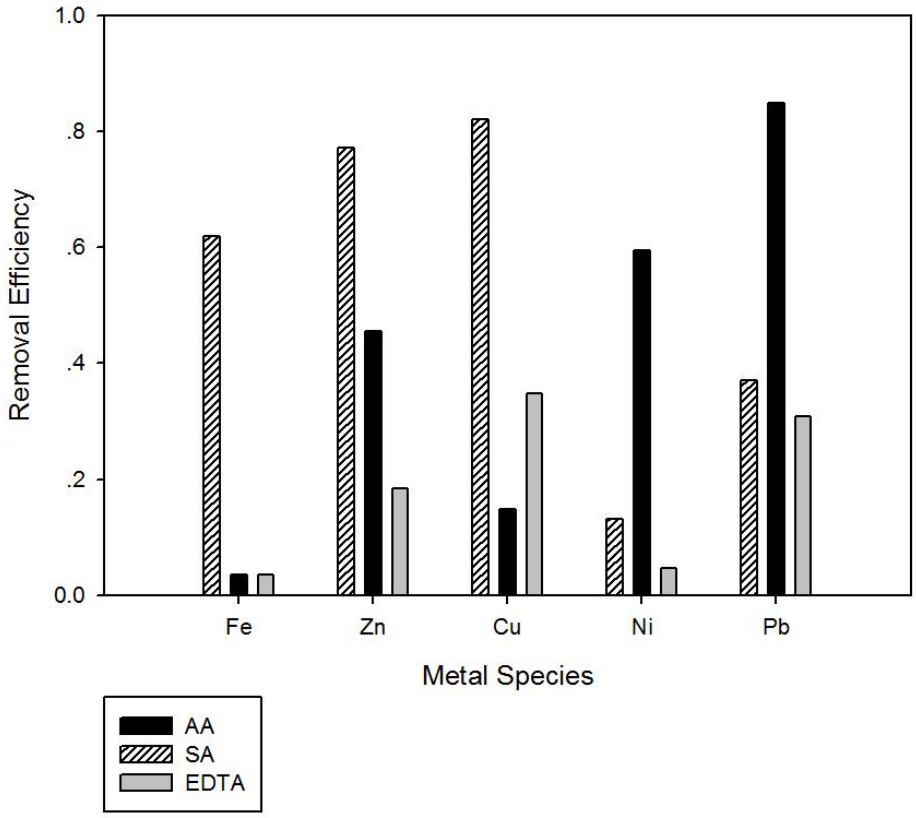

Figure 1. Heavy Metal Removal Efficiency

The removal efficiency for each method was metal specific. It seemed that sulfuric acid had the best extraction results for most of the metals including iron, zinc, and copper. Acetic acid showed the best extraction for nickel and lead.

After the removal efficiency of the conventional chemical methods was determined, the electromagnetic wave treatment was tested. At the current stage, domestic microwave oven (GE, Model JES2051SNSS) was used as the microwave generator, which produced a frequency equal to 2.45 GHz with a local energy level of 140W to 350W. Several potential impact factors were investigated in order to identify the optimal treatment condition: the application time of microwave treatment from 5 seconds to 20 minutes, the moisture content of biosolids from 50% to 100%, and liquid to solid ratio from 1 to 20. In addition, the effect of 24-hour shaking after microwave treatment on metal extraction was further tested. In combination with the microwave

treatment, besides the three conventional chemical extraction methods described above, 0.01M sodium chloride and 0.002M ferric chloride were also included to investigate the influence of ionic strength and ion exchange on metal extraction. Under the condition of liquid to solid ratio of 1, biosolids moisture content of 100%, microwave treatment of 30 seconds with a power level of 140W, and a reagent contact time of 24 hours, sulfuric acid showed the best removal efficiency, followed by EDTA and acetic acid, with much reduced acid usage.

A series of experiments were further designed to test EDTA in combination with microwave treatment. The experiment conditions are listed in the Table 2 and the results are summarized in Figure 3.

Table 2. Microwave-Mediated EDTA Experiments

Exp. Condition	#1	#2	#3	#4	#5
S/L Ratio	1:1	1:1	1:5	1:5	1:5
MW Time	10 sec	10 sec	10 sec	10 sec	NA
Power Level	140W	140W	140W	140W	NA
Post Treatment	Shaking	Centrifuge	Centrifuge	Shaking	Shaking

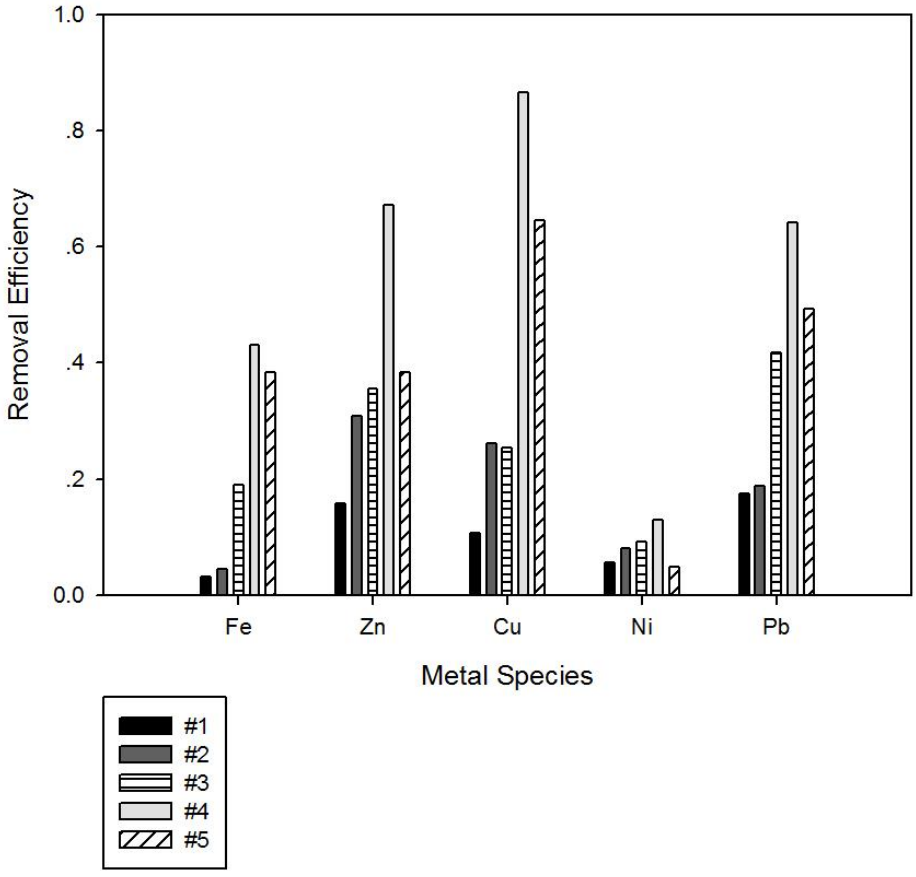

Figure 3. Microwave Assisted EDTA Heavy Metal Removal

The optimal experimental condition for the combination of EDTA with microwave treatment was identified as: liquid to solid ratio of 5 followed by a 24-hour shaking. Under this condition, obvious improvement of metal removal efficiency was observed for all measured heavy metal species.

Work of Next Steps

After heavy metal removal, biosolids will be composted with sawdust, wood chips, yard clippings, or crop residues. Physical and chemical properties as well as nutrient and heavy metal contents of the compost (including controls) will be determined according to the standard testing methods. Specifically, bulk density will be determined from the volume and mass of the sample that has been dried at 105^{o}C. Water content will be determined by gravimetric method based on the weight loss of wet sample at 105^{o}C. pH and soluble salts will be determined in a 1:1 suspension and organic matter content will be measured by the loss on ignition method with sample ashing at 550^{o}C. Total Kjeldahl Nitrogen will be measured with the Kjeldahl method based on the wet oxidation of organic matter and conversion of organic nitrogen to the ammonium form. Nitrate and extractable ammonium will be measured by equilibrium extraction for nitrate and ammonium with 2N KCl. Total phosphorus will be determined by acid digestion of the sample using concentrated HNO_3 and $HClO_4$. Extractable phosphorus will be measured by the Olsen method, which is based on alkaline extraction using 0.5N $NaHCO_3$. Exchangeable potassium will be measured by extracting the sample with 1N CH_3COONH_4 at pH7.0. After extraction, nitrogen and phosphorous concentrations will be quantitatively determined by UV–VIS spectrophotometry, while those of potassium will be determined by MP-AES. Extractable heavy metals will be measured by extraction using DTPA solution (0.005M DTPA, 0.1M TEA, and 0.01M CaCl2 at pH 7.3) and MP-AES.

Information Dissemination Activities:

Metrics:

1. List graduate or postdoctoral researchers funded by this Hinkley Center project

Last name, first name	Rank	Department	Professor	Institution
Runwei Li	Ph.D.	Civil and Environmental Engineering	Gang Chen	Florida State University
Hongying Yuan	Postdoctoral Researcher	Civil and Environmental Engineering	Gang Chen	Florida State University

2. List undergraduate researchers working on this Hinkley Center project

Last name, first name	Department	Professor	Institution
Kadeem Rowe	Civil and Environmental Engineering	Gang Chen	Florida State University

3. List research publications resulting from this Hinkley Center project
   Li, R., Tang, Y., Tawfiq, K. and Chen, G., 2017, Electromagnetic Wave-Induced Heavy Metal Removal for Dewatered Biosolids Composting, Environmental Technology, in preparation.

4. List research presentations resulting from this Hinkley Center project
   Li, R., Tang, Y., Tawfiq, K. and Chen, G. "Electromagnetic Wave-Induced Heavy Metal Removal for Dewatered Biosolids Composting". Florida Branch Meeting of American Society for Microbiology, Clearwater Beach, October 13-15, 2017.

5. List who has referenced or cited your publications from this project?
   Current research is in process. Our prior research on composted sewage sludge that was published in 2007 has been cited 121 times:

   Cheng, H., W. Xu, J. Liu, Q. Zhao, Y. He and G. Chen, 2007, Application of composed sewage sludge (CSS) as a soil amendment for turfgrass growth. Ecological. Eng., 29, 96-104.

   Cited by 121 (http://www.sciencedirect.com/science/article/pii/S0925857406001753) (https://scholar.google.com/scholar?cites=8788015943956282734&as_sdt=40005&sciodt=0,10&hl=en)

6. How have the research results from this Hinkley Center project been leveraged to secure additional research funding?

"Electromagnetic Waves-Induced Heavy Metal Removal for Biosolids" by Gang Chen and Youneng Tang will be submitted to Environmental Research and Education Foundation in response to Environmental Research and Education Foundation Request for Proposals: High Need Topics in Solid Waste Research.

7. What new collaborations were initiated based on this Hinkley Center project?
   We have initiated collaboration with John Hallas from Talquin Electric Cooperative, Inc. and Hafiz Ahmad from Florida State University at Panama City Campus from this research. In addition, we have been contacted by Jeffrey Cunningham from University of South Florida and requested for collaboration through an EPA-funded research center (http://usf-reclaim.org/). We are now working with Dr. Sarina J. Ergas on nutrient management in solid waste.

8. How have the results from this Hinkley Center funded project been used (not will be used) by the FDEP or other stakeholders? (1 paragraph maximum).
   We keep close contact with managers of Leon County Landfill, Springhill Regional Landfill (Jackson County) and Perdido Landfill (Escambia County). In addition, we work closely with Thomas P. Smith Water Reclamation Facility located in Tallahassee, FL. We discuss the technical achievement of this project with the managers and request for suggestions to further our research. We also share the results with FDEP through TAG members of Joe Dertien and Owete Owete. We also discuss the results with Talquin Electric Cooperative, Inc., which operates seven wastewater treatment plants.

Tag Members: Joe Dertien, Owete Owete, John Hallas, Chen Lin, Hafiz Ahmad, and Matthew Hendrix

TAG meetings: Information of this project is available through http://www.eng.famu.fsu.edu/~gchen/index_files/Page570.htm. The first TAG meeting was held on May 4 at FAMU-FSU College of Engineering. The second TAG meeting will be held in September, 2017. The meeting minutes and presentation are available at the project website.