Sewage sludge stabilization with clay minerals and biochar

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Increasing sewage sludge production requires efficient treatment and disposal routes. Agronomic use of sewage sludge promotes nutrient recycling and thus is considered a more desirable option than landfill disposal. With the appropriate treatment, removal of available heavy metals and reduction of microbial load, stabilized sewage sludge bio-fertilizers could be ideal for agronomic use. Nonetheless, we still lack treatment approaches that consider an integrated methodology towards microbial load reduction and heavy metals load control, as well as efficient nutrient reuse. The addition of clay minerals and biochars during sewage sludge stabilization, as strong ion adsorbents and bactericides respectively (Agyarko-Mintah et al, 2017; Alshameri et al, 2018; Mohanty et al, 2014; Williams, 2019), could be a promising alternative treatment. The objective of this study was to evaluate sewage sludge stabilization with clay minerals (bentonite, vermiculite, zeolite) and biochar in comparison to lime ($Ca(OH)_2$) stabilization in the perspective of agronomic use of the treated sludge.

In a mesocosm experiment (May 2020), dewatered sewage sludge (DSS) was mixed with bentonite (B), vermiculite (V), zeolite (Z), biochar (BC) and reagent grade $Ca(OH)_2$ (RG) at 15 % (B15, V15, Z15, BC15 & RG15) and 30 % (B30, V30, BC30 & RG30) per wet weight (WW) basis, including an additional treatment of unamended DSS (ADSS) as control, in three replicates. The mixtures were placed outdoors under protection, in a completely randomized design (CRD), for a two-month equilibration. Materials and stabilized sewage sludges (treatments), as well as DSS were analyzed for microbial load and physiochemical properties. Specifically, industrial grade clay minerals (Milos island and Northern Greece) and biochar (pine residues, Crete, Greece) were prepared (<2 mm) and analyzed for: moisture, pH, cation exchange capacity, electrical conductivity (EC), water-soluble cations and heavy metals, and total essential macro- and micronutrients for plants and heavy metals, employing standard methods (Sparks, 1996). Dewatered sewage sludge (DSS; Wastewater Treatment Plant of Thessaloniki, Greece) and stabilized sewage sludge samples, were tested for total *heterotrophs* (Rutgersson et al., 2020), total *coliform*s, total *enterococci*, *Escherichia coli* (*E. coli*) (ISO 4831, 2006; ISO 7251, 2005) and *Salmonella spp.* (ISO 6579, 2002) (Table 1). Also, pH, dry matter, loss on ignition (LOI), available NO_3-N and NH_4-N after extraction with 1 M KCl, EC, water-soluble nutrients and heavy metals, and total nutrients and heavy metals were determined, employing standard methods (Sparks, 1996). Moreover, the sodium adsorption ratio (SAR) was calculated. Datasets were statistically tested with one-way ANOVA and *post-hoc* LSD test ($p \leq 0.05$).

Table 1. Certain microbial and physiochemical properties of the dewatered sewage sludge (DSS)

Property (unit)	Value (±SE)
Total *heterotrophs* (CFU g^{-1} DW)	1.5e04 ±8.1e02
Total *coliforms* (CFU g^{-1} WW)	5.7e04 ±5.1e04
E. coli (CFU g^{-1} WW)	2.7e04 ±2.3e04
Total *enterococci* (CFU g^{-1} WW)	4.5e04 ±4.2e04
Salmonella spp. (25 g^{-1})	Detectable
pH (1:10 H_2O)	7.1 ±0.1
Dry Matter (% WW)	13.8 ±0.5
LOI (% DW)	67.9 ±1.4
NO_3-N (mg kg^{-1} DW)	2265 ±1100
NH_4-N (mg kg^{-1} DW)	2927 ±432

At the end of the experiment, all microbial indicators showed reduction and *Salmonella spp.* was non-detectable in any treatment (Table 2). A microbial load reduction of at least one logarithmic unit (log_{10}) was indicated for the B15, V15, BC15 and B30 treatments, whilst BC15 achieved the highest microbial load reduction when compared to the DSS. Microbial load was non-detectable for both RG treatments, due to strongly alkaline pH (~12).

Table 2. Reduction of microbial indicators (log_{10} scale) for bentonite (B), vermiculite (V), zeolite (Z), biochar (BC) and air-dried sewage sludge (control, ADSS) treatments.

Microbial indicator	Treatment								
	ADSS	B15	B30	V15	V30	Z15	Z30	BC15	BC30
Total *heterotrophs*	0.5	0.3	0.4	0.6	0.6	0.0	0.6	0.0	-0.2
Total *coliforms*	0.4	0.6	0.7	0.6	0.6	0.5	0.2	1.1	0.8
E. coli	0.7	1.0	1.3	1.3	0.9	0.7	0.7	1.5	1.0
Total Enterococci	1.0	1.1	1.4	1.1	0.9	0.9	0.5	1.6	0.5

After equilibration, organic matter content was lower (~25%) than the control (~66%) in all treatments, except for BC (~84%). The total concentrations of macro- and micronutrients in all treatments were at similar levels to ADSS. However, in certain cases, total nutrients' concentrations were higher due to the chemical composition of the amendment (e.g. Z treatments had more K due to its occurrence in zeolite's structure). The ADSS had the highest available NH_4-N levels followed by the Z15, Z30 and BC15 treatments, whereas the V15, V30, RG15 and RG30 treatments had the lowest available NH_4-N. The pH increased to slightly alkaline levels (pH ~7.3) for B, V and BC, except for RG (pH ~12). There was an increase for EC in all treatments, and ADSS, RG15 and RG30 showed the greatest values (7.2, 7.8 and 6.1 dS m^{-1}, respectively). The highest SAR values were obtained for the B15 (8.4), B30 (8.3), BC15 (10.9) and BC30 (12.4) treatments and the control (7.8). Total concentrations of heavy metals were far below the legislative critical limits for agronomic use of sewage sludge. Especially, total Cd and Hg were non-detectable and the same stands for all water-soluble heavy metals.

In conclusion, the treatment of dewatered sewage sludge with bentonite, vermiculite and biochar appears to be a promising stabilization method (reduction of microbial load) while retaining nutrients and controlling heavy metal load for the perspective agronomic application of treated sewage.

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Agyarko-Mintah, E., Cowie, A., van Zwieten, L., Singh, B.P., Smillie, R., Harden, S., Fornasier, F. 2017. Biochar lowers ammonia emission and improves nitrogen retention in poultry litter composting. Waste Management 61: 129-137.

Alshameri, A., He, H., Zhu, J., Xi, Y., Zhu, R., Ma, L., Tao, Q. 2018. Adsorption of ammonium by different natural clay minerals: Characterization, kinetics and adsorption isotherms. Applied Clay Science 159: 83-93.

ISO 4831. 2006. Microbiology of food and animal feeding stuffs – horizontal method for the detection and enumeration of *coliforms* -most probable number technique. 3rd ed.

ISO 6579. 2002. Microbiology of food and animal feeding stuffs – horizontal method for the detection of *Salmonella spp*. 4th ed.

ISO 7251. 2005. Microbiology of food and animal feeding stuffs – horizontal method for the detection and enumeration of presumptive *Escherichia coli* - most probable number technique. 3rd ed.

Mohanty, S.K., Cantrell, K.B., Nelson, K.L., Boehm, A.B. 2014. Efficacy of biochar to remove *Escherichia coli* from stormwater under steady and intermittent flow. Water Research 61: 288-296.

Rutgersson, C., Ebmeyer, S., Lassen, S.B., Karkman, A., Fick, J., Kristiansson, E., Brandt, K.K., Flach, C.F., Larsson, D.G.J. 2020. Long-term application of Swedish sewage sludge on farmland does not cause clear changes in the soil bacterial resistome. Environment International 137: 105339.

Sparks, D.L. 1996. Methods of soil analysis -Part 3 - Chemical methods. SSSA Book Series 5. SSSA, ASA, Madison, WI.

Williams, L.B. 2019. Natural antibacterial clays. Clay and Clay Minerals 67: 7-24.