

## Gravimetric Evolution During Sewage Sludge Biostabilization

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### ABSTRACT

Sewage sludge is a by-product in the wastewater treatment and is an inherent hazardous issue because of the pathogenic contamination of natural resources. Therefore, in this study, domestic sludge was treated with pre-montane forest soil, macronutrients, and also pasteurization to reduce the content of volatile solids and pathogens. The best biostabilization treatment using pre-montane forest soil and pasteurization obtained a volatile solids reduction of 87% according to the environmental regulations, in which a biosolid is stable in a range of 38% of volatile solids reduction. In less than 30 days in a mesophilic range, the coliform count was reduced up to 71% when using forest soil and pasteurization. Thus, a biosolid-class B was obtained using gravimetric means as a platform to promote fast quality control.

**Keywords:** pathogens, biosolids, sewage, mesophilic, anaerobic.

### INTRODUCTION

In the next 40 years, the world population will double and consequently, the capacity to treat the volume of wastewater according to the population increase will barely meet the demand; this would cause the waste to be discharged without known control to ecosystems. Municipal wastewater contains pathogenic bacteria, protozoa, viruses, and parasites, as well as oils, fats, detergents, soaps, nutrients, salts, and particles of hair, food, and paper. The treatment of municipal wastewater accounts for the worldwide production of approx. 48 million dry tons of sewage sludge, and its disposal has become an environmental problem because of the pathogenic risks (Fytili and Zabaniotou, 2008; Krüger et al., 2014; Li et al., 2007; Mu et al., 2016; Snowden-Swan et al., 2016; Wang et al., 2008a; Wei et al., 2003; Zahan et al., 2016). A typical drawback of the wastewater treatment system is the high-energy cost in the context of

energy efficiency, carbon footprint, and recycling. Sludge treatment and its disposal account for up to 60% of the total operating costs, and the elimination of harmful pathogens and a new kind of contaminant, namely, emerging pollutants constitutes a critical step (Barrios et al. 2015; De Vrieze et al., 2016; Jenicek et al., 2012; Lewis et al., 1999; Ruffino et al., 2015; Wang et al., 2008b; Weemaes and Verstraete, 1998; Ye et al., 2014). Biological sewage sludge is mainly composed of organic matter (approx. 59–88%) from scum or the solids removed in wastewater treatment (Tchobanoglous et al., 2013). Sewage comes from human excreta and is a mixture of fats, proteins, carbohydrates, lignin, amino acids, sugars, cellulose, humic material, fatty acids, non-essential trace metals and organic micropollutants, which can be decomposed and produce offensive odors (Environmental Protection Agency, 1994; Kinney et al., 2006; Rogers, 1996; Singh and Agrawal, 2008; Weemaes and Verstraete, 1998).

In 1991, the term *biosolid* was adopted to apply to all sedimentary sludge in which mesophilic or thermophilic digestion diminishes odors, organic matter, and pathogenic risk under anaerobic conditions, which also generates bioenergy (Bright and Healey, 2003; Cain, 2010; Carrère et al., 2010; Snowden-Swan et al., 2016; Zahan et al., 2016). Biosolids have been recognized as a useful soil amendment and source of nitrogen, phosphorus, organic matter, and other nutrients, which can enhance the physical properties of soil as well as plant growth (Kinney et al., 2006); moreover, biosolids contain a good deal of energy at approx. 11,400 BTU per dry pound (Mu et al., 2016). Among Canada, the US, and European countries, 53 percent of biosolids are used in agriculture directly or after composting, totaling more than 2.39 million dry tons per year yield (Kinney et al., 2006; Stasinakis, 2012; Wang et al., 2007). However, biosolids are not completely secure for land application due to offensive odors and toxic elements, e.g., heavy metals and persistent organic pollutants, found in the sewage sludge (Hale et al., 2001; Krach et al., 2008; Lewis et al., 2002; Wei et al., 2003). A total of 87 synthetic organic chemicals were found in biosolids, including chemicals like polychlorinated biphenyls (PCBs), pharmaceuticals such as triclosan (antimicrobial disinfectant), tonalide (a musk fragrance), diphenhydramine (antihistamine), carbamazepine (an antiepileptic drug), and heavy metals like As, Cd, Cr, Pb, Hg, Ni, and Se (Barrios et al., 2015; Cain, 2010; Egan, 2013; Mulla et al., 2016; Venkatesan and Halden, 2014). Biosolids also carry high densities of enteric viruses, helminth eggs, and *Salmonella spp.*, which pose risks to the human health (Barrios et al., 2015; Gerba et al., 2011; Oron et al., 2014). Given that a stabilization process should be performed with high-quality standards to prevent the human health risks, the principal contributions in the present study are:

- An anaerobic process to stabilize sewage sludge using pre-treatments under different conditions like the addition of premontane forest soil under differing nutritional conditions and pasteurization.
- A thermogravimetric technique to monitor sludge stabilization.

In the pursuit of testing the hypothesis, we found out that the interactions between the biological, thermal, and salinity treatments influence the

change of total and volatile solids, namely a gravimetric evolution of a biostabilization process.

## MATERIALS AND METHODS

### Biostabilization process

Residual sludge was collected from the secondary settler tank of a municipal wastewater treatment plant located in Quito (Ecuador). The residual sludge (A) was thickened by sedimentation (B) with a sedimentation column (2 m in length and 30 cm in diameter). Afterwards, the resulting sludge was filtered with cellulose pads (C) and then pre-treated under different conditions (D). The following pre-treatments were made before the anaerobic digestion: (T<sub>1</sub>) 0.6 kg of soil from a premontane secondary forest was added per each kg of sludge; (T<sub>2</sub>) again, 0.6 kg of soil from a premontane forest was added per each kg of sludge, and also, a 0.1% nutrient solution of 10% N, 40% P and 10% K (Merck, USA) was added twice daily for 30 days; (T<sub>3</sub>) the sludge was pasteurized in an oven (Wiseven 165, USA) at 70°C for 30 minutes; (T<sub>4</sub>) a 0.1% nutrient solution was added every morning, and a 1.0% NaCl solution (Merck, USA) was added at night for 30 days. After pre-treatment, anaerobic digestion was performed in a sealed 1000 mL Erlenmeyer flask (E). The flasks were thermally insulated and were slowly mixed twice a day for 30 days. At the end of the process, the biosolid was sundried (Figure 1).

### Analysis

The temperature and pH were measured three times a day using a soil multiparameter tester (HANNA HI 99121, USA). Total solids (ST) and volatile solids content (SV) were determined at 105°C and 550°C, respectively, using ASTM methods. Arsenic, cadmium, mercury, and lead were quantified by atomic absorption according to APHA standard methods. The biological activity of the sludge was measured using OxiTop® Biological Oxygen Demand (BOD) respirometry system (WTW, Germany) at 25°C. Helminth eggs were determined using EPA method 9132, and total coliform was counted using the violet bile red Petrifilm plates (3M, USA) with tetrazolium as the indicator at 37°C for 48 hours; the sludge samples were diluted at 1:100 with sterile peptone water because sludge is characterized by a dark color.

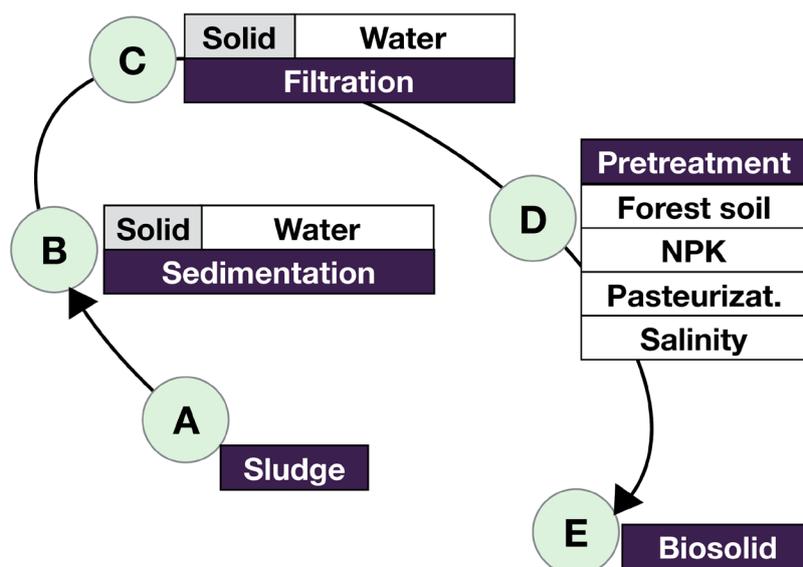


Figure 1. Biostabilization process

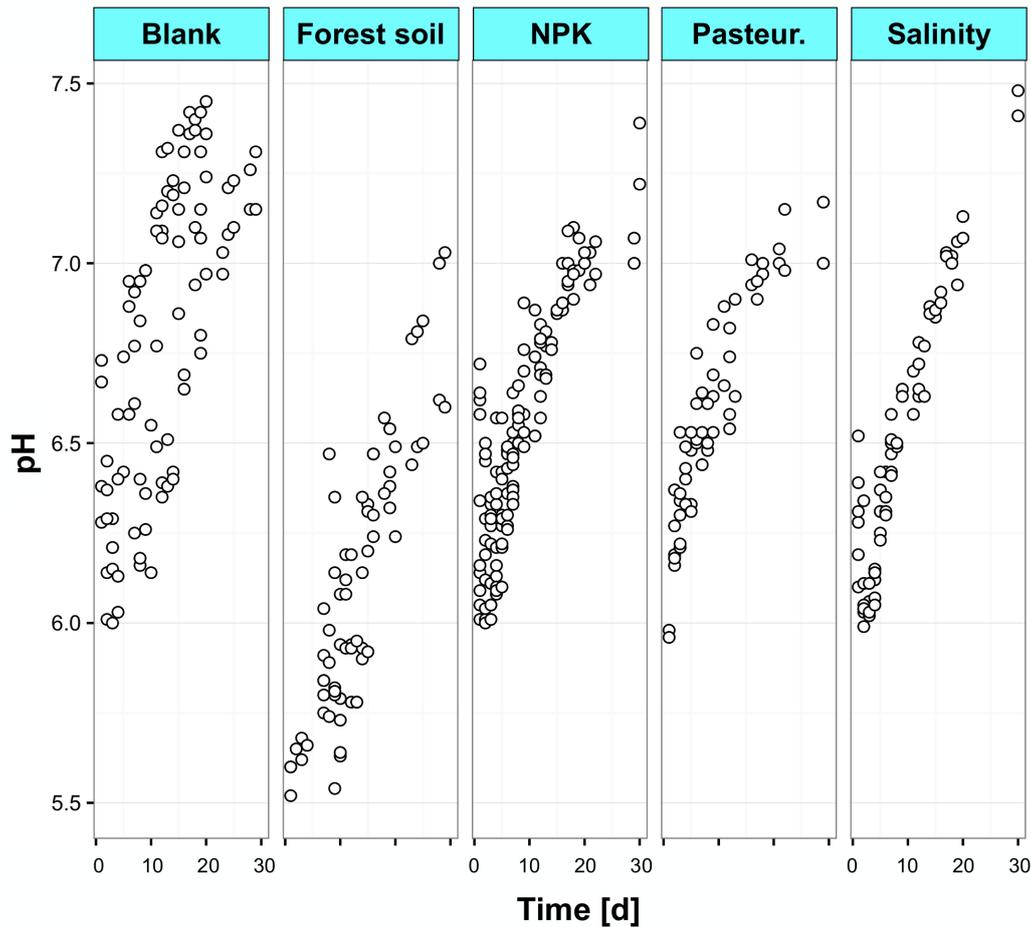
## RESULTS AND DISCUSSION

### Effect on pH and temperature

Different pre-treatment conditions using pre-montane forest soil, NPK, NaCl, and pasteurization changed pH from 6.0–6.5 to 7.0–7.5 during 30 days of biostabilization, as indicated in Figure 2. According to the results, pH changes due to the microbial decarboxylation of organic anions, heterocyclic compounds, and volatile fatty acids, suggesting that stability was reached (Wu, Ma, and Martinez, 2000; Yuan and Zhu, 2016). Normally, the first days in the anaerobic digestion are the rate-limiting step because of the hydrolysis of insoluble organic matter into a soluble form, which influences the further stabilization kinetics (Eastman and Ferguson, 1981; Parkin et al., 1986). The addition of forest soil, NPK, and NaCl boosted the release of microbial exoenzymes to cleave macromolecules like triglycerides, diglycerides, and fatty acids (Harris and McCabe, 2015). Thus, the more carbon is solubilized, the faster the stabilization process. Therefore, pre-treatments focused on hydrolysis maximize the degradation rate until mineralization of organic matter occurred. A drop in pH shows an increase of volatile fatty acids as acetic acid or propionic acid, which could be a problem in the stabilization process (Ahring et al., 1995; Amani et al., 2010; Berkday and Nas, 2007; Carrère et al., 2010; Harris and McCabe, 2015). In our case, the acid phase lasted for about 15 days, which is an indicator of hydrolysis without interruption of the

high fatty acid concentrations or non-biodegradable (refractory) components. At the end of the process (Fig. 2), the pH was 7.0–7.5 due to the microbial ammonification when ammonium compounds are nitrified to nitrate (Cofie et al., 2016).

In Figure 3, the temperature remained between 30–37°C in all treatments while the control reaction remained at approx. 20°C. One pre-treatment involved pasteurization at 70°C for 30 minutes, which is used to improve the organics solubilization to provide a carbon source for microorganisms (Weemaes and Verstraete, 1998). In this way, high temperatures expose macromolecules to achieve their breakdown and further ease the degradation by thermophilic microorganisms (Carrère et al., 2010; Harris and McCabe, 2015). Since the mesophilic digestion does not kill pathogens efficiently, the thermal hydrolysis at 70°C was utilized as another kind of disinfection technique (Oleszkiewicz and Mavinic, 2002). The thermophilic digestion is up to three-fold higher than the mesophilic one, which enhances the hydrolysis conversion rates with an impact of higher volumetric biogas production at a lower hydraulic retention time (HRT) (De Vrieze et al., 2016). However, in economic terms, the mesophilic temperatures are more stable and have lower energy costs than the thermophilic treatments (Braguglia et al., 2015). Moreover, a mesophilic process is less inhibited by ammonium and long-chain fatty acids (LCFA) (Fernández-Rodríguez et al., 2015). According to Figure 4, the process started almost at 45°C and had an acidic pH dur-



**Figure 2.** Effect of different biostabilization pre-treatments on pH during 30 days

ing the first few days, which favored the growth of fungi and actinomycetes. However, afterwards, a neutral pH and a temperature close to ambient temperature (20°C) were obtained. Together, pH and temperature progressively interacted due to high microbial activity during a solid residence time (SRT) of 30 days at 20–45°C. Thus, SRT and the mesophilic range in our biostabilization process performed according to the EPA regulations for biosolids under class B (Mustafa et al., 2014).

### Gravimetric evolution

One method to evaluate biostabilization is to measure the change in total solids (TS) and volatile solids (VS), which is herein recognized as gravimetric evolution. Over a period of 30 days, the content of TS and VS changed dramatically, and showed tendencies that demonstrated biostabilization (Fig. 4). The content of organic matter is virtually represented by VS, and its reduction is due to the microbial mineralization and the conversion of organic matter into

humic substances (Gómez et al., 2005; Otero et al., 2002). The measurement of volatile solids destruction is an indicator of the mineralization degree of organic carbon to mineral forms like CO<sub>2</sub> or CH<sub>4</sub> (Ahring et al., 1995; Bernal et al., 1998). According to VS destruction, the USEPA uses the value of 38% reduction in the threshold for considering the sludge to be stabilized (Environmental Protection Agency, 1994; Oleszkiewicz and Mavinic, 2002). In Figure 4, the VS/TS relationship is presented. Due to the low mineralization rate of the organic nitrogen found in sludge, enrichment with minerals like nitrogen, phosphorous, potassium and bacterial inoculant (forest soil) enhances the microbial activity and further organic matter degradation (Cofie et al., 2016). Our results confirm that nutrients, forest soil, thermal treatment, and even salinity are the enhancers of VS reduction (Bhattacharya et al., 1996; Ruffino et al., 2015).

In Figure 5, the gravimetric evolution of VS/TS is shown in every treatment. The control treatment (blank) did not overcome the stability range

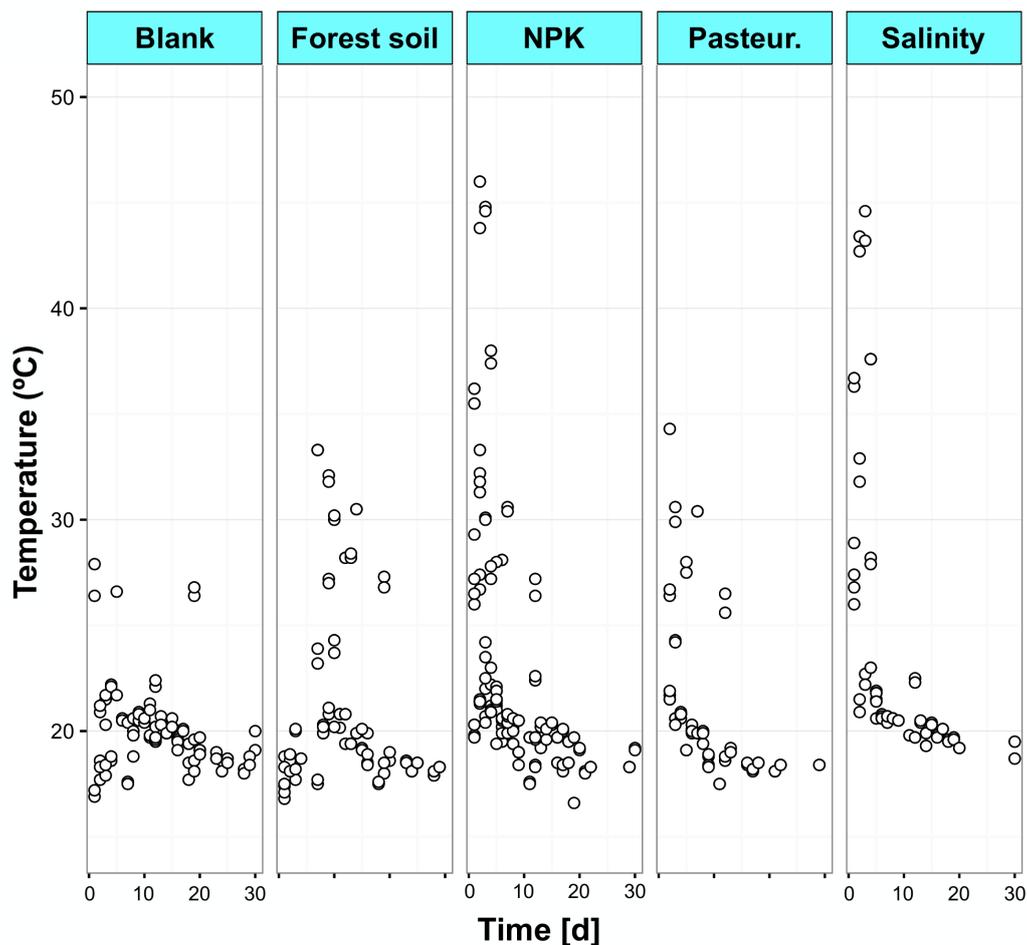


Figure 3. Effect of different biostabilization pre-treatments on temperature during 30 days

above 11.4% VS reduction. The forest soil, as a pre-digestion treatment, reduced 55.7% VS/TS because the more diverse the microbes, the faster digestion occurs. The pre-treatment that used forest soil and NPK nutrients, altogether reduced by 87.3% the VS/TS content. An optimal pre-treatment was also using pasteurization, in which an 87.2% VS/TS reduction was obtained. The pre-treatment with NaCl destroyed the VS content just by 36.7%; in this case, NaCl inhibited the microbial growth.

According to the results shown in Figure 5, a first order kinetic was obtained in two pre-treatments (Bernal et al., 1998). Forest soil enhanced a first order kinetic ( $k = 0.02930$ ,  $R^2 = 0.9312$ ,  $p < 0.05$ ), as well as NPK ( $k = 0.064$ ,  $R^2 = 0.9706$ ,  $p < 0.05$ ). On the other hand, a second order kinetic was observed in the pre-treatments using pasteurization ( $k = 0.2390$ ,  $R^2 = 0.9833$ ,  $p < 0.05$ ) and salinity ( $k = 0.0262$ ,  $R^2 = 0.9486$ ,  $p < 0.05$ ). Thus, faster kinetics reduces the size of the reactor and decreases hydraulic retention time (HRT) (Carrère et al., 2010).

According to the literature, the mesophilic digestion requires over a 20-day retention time, and it is not as efficient as the thermophilic process (Rulkens, 2008; Song et al., 2004). However, in the present work, the change of VS/TS in pre-treatments using forest soil, NPK, and pasteurization was up to 87%, which shows that mesophilic digestion can be optimal as the thermophilic counterpart.

### Heavy metals

Table 1 shows the reduction in the heavy metal content through the biostabilization process in comparison with the EPA permissible limits. Ac-

Table 1. Chemical characterization before and after the biostabilization process

Parameter	Sludge (mg/kg)	Biosolid (mg/kg)	EPA (mg/kg)
As	0.170	0.010	41
Cd	0.110	0.025	85
Hg	0.002	0.001	57
Pb	2.080	0.400	840

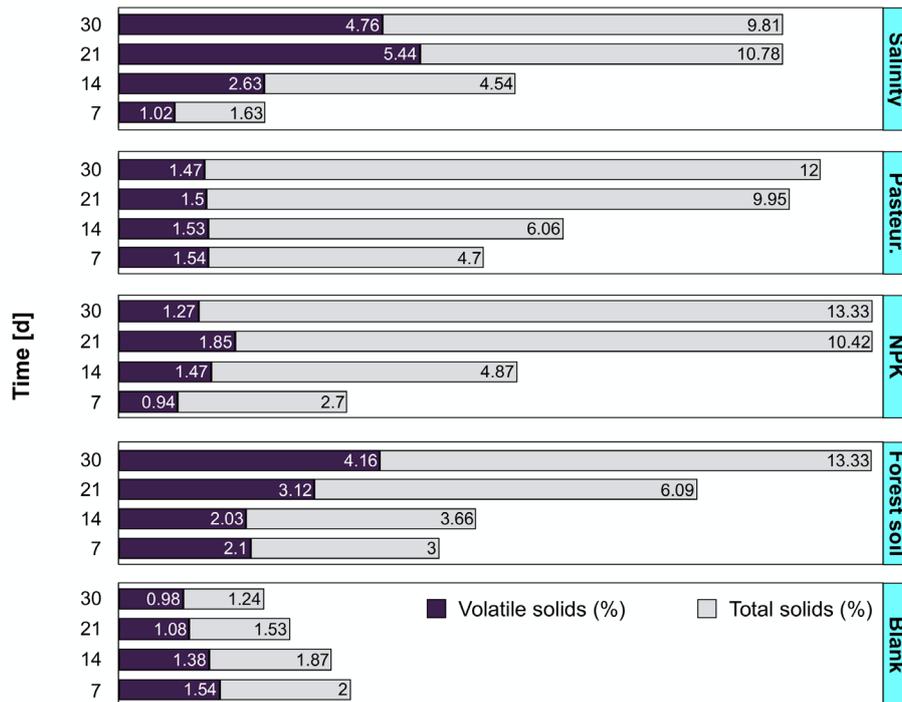


Figure 4. Change in TS and VS (%) during 30 days

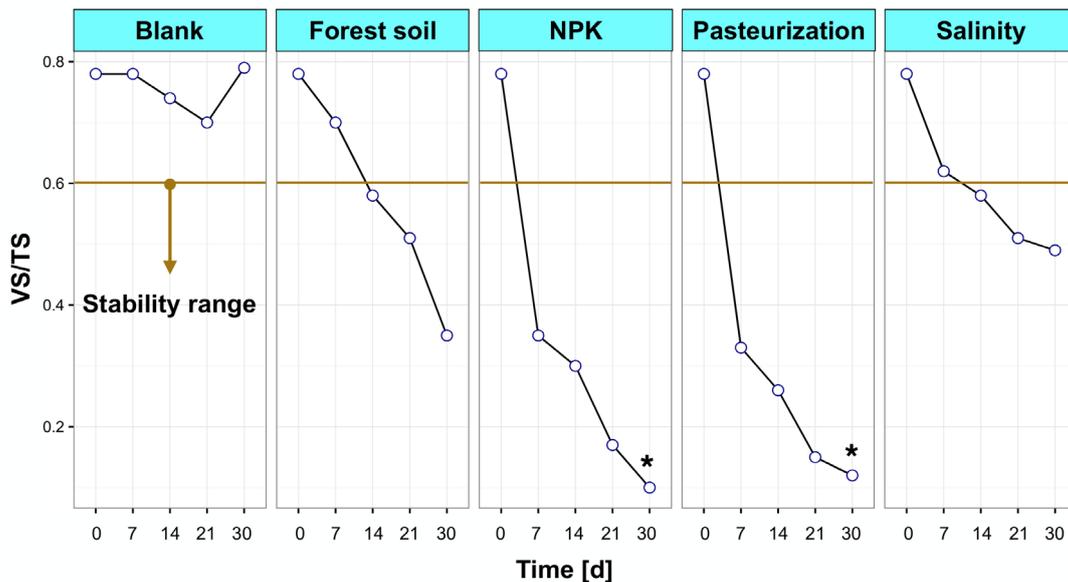


Figure 5. Gravimetric evolution (VS/TS) at different pre-treatments during 30 days

According to the results, the order of metal content at the end of biostabilization was Pb > Cd > As > Hg. The concentrations of Pb, Cd, As, and Hg decreased by 94.1%, 77.3%, 50%, and 80.8%, respectively. The reduction of the total concentrations of heavy metals in biosolids is significant due to dilution during the mixing process with forest soil; therefore, the concentrations are under the EPA regulation limits. However, the bioavailability and toxicity of heavy metals in the sludge

depend on their chemical forms and pH, which in consequence will precipitate with carbonate minerals, complexes and organic ligands at basic pH (Dong et al., 2013).

#### Pathogen removal

We measured the remaining digestion of samples in mg/L over nine days after the 30-day of biostabilization process to test any microbial

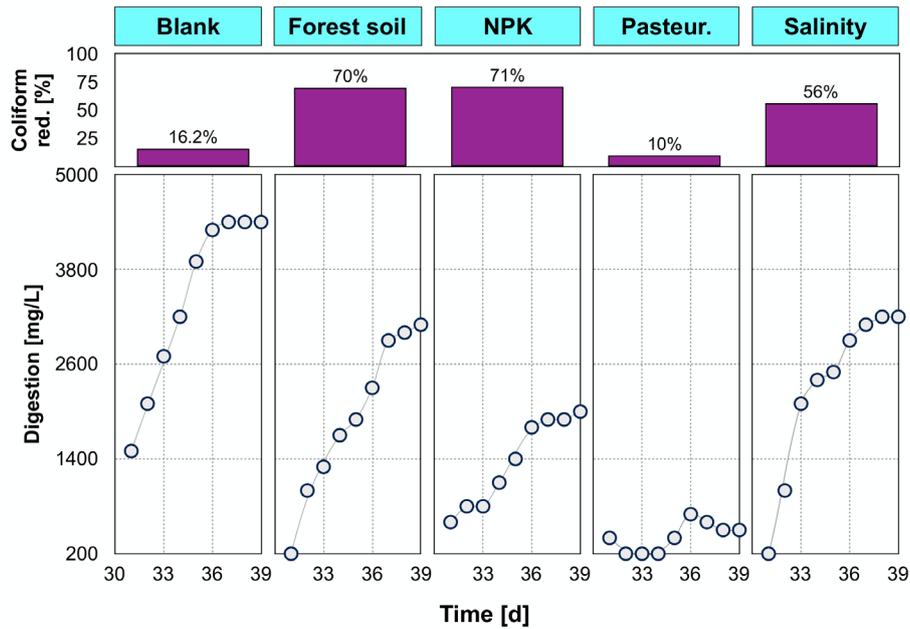


Figure 6. Digestion of sludge samples after the biostabilization process

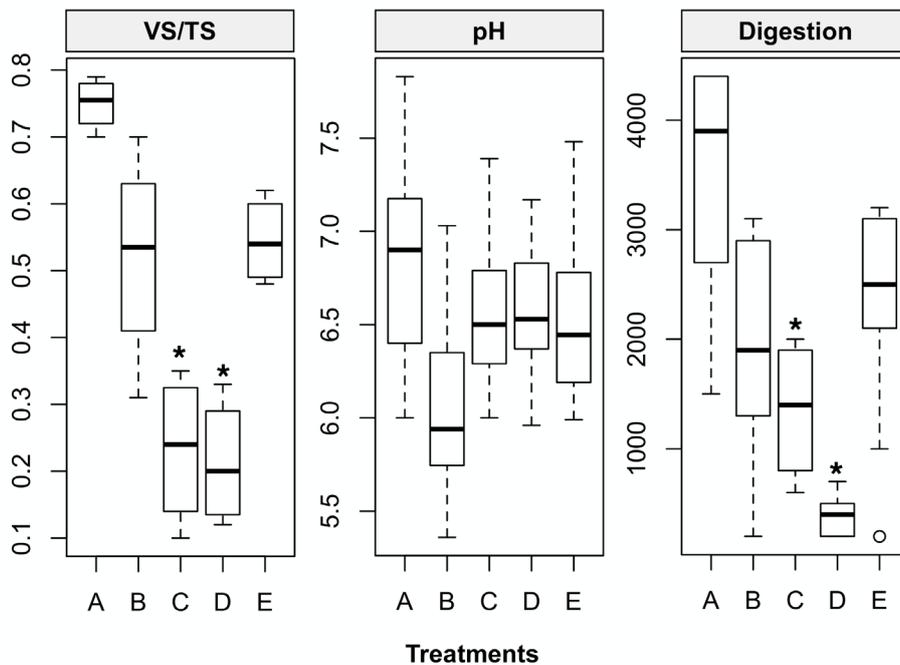


Figure 7. Results of the biostabilization process; (A) blank sample, (B) forest soil, (C) NPK, (D) pasteurization and (E) salinity treatment

activity (Figure 6). After the VS destruction, the CO<sub>2</sub> production rate should decrease, along with the pathogen removal (Wu et al., 2000). Both digestion and coliform reduction were examined to test which treatment was the most efficient way to stabilize sludge through pathogen reduction. The colony-forming units (CFU) per mL were 31700, 26550, 9650, 9150, 28500, and 13700 for blank, forest soil, NPK, pasteurization, and salin-

ity treatments, respectively. According to the results, the order of coliform reduction at the end of biostabilization was NPK > forest soil > salinity > blank > pasteurization, and the removal of coliforms was reduced by 71%, 70%, 56%, 16.2%, and 10%, respectively. In the present case, the best pre-treatment utilized NPK and forest soil.

According to the results, our biosolid is classified as class B because the US EPA recom-

mends a VS reduction by 38% and a mean coliform density of less than 2 million CFU per gram of biosolids (Environmental Protection Agency, 1994). Although a biosolid might be recognized as freshly processed class B, sewage sludges may pose a significant risk of infection due to the pathogen regrowth, and this is why such biosolids should be managed with great care to public access (Gattie and Lewis, 2003). In order to adhere to the human health standards, different treatment methods should demonstrate the ability to neutralize the pathogen viability.

Figure 7 presents the biostabilization results in box plots. The pre-treatments using forest soil as bio-accelerator, NPK and pasteurization stabilized sewage sludge up to 87% in VS/TS reduction. All treatments, except the forest soil one, got a pH between 6.5 and 7. No significant benefit was obtained by the NaCl treatment.

## CONCLUSIONS

The legal constraints to using sewage sludge in agriculture and further land applications motivate the search for cheaper stabilization processes and therefore avoiding incineration as the final option for sewage disposal. In the present study, the use of the proper combination of nutrition and temperature enhanced the performance of sludge biostabilization. However, to improve the digestion efficiency, a pasteurization process should be conducted at a higher temperature in a shorter time. According to the results obtained in this work, the further research outlook is focused on the high reduction of pathogen count.

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