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# Uncertainty in degradation rates for organic micro-pollutants during full-scale sewage sludge composting

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ABSTRACT

Composting can potentially remove organic pollutants in sewage sludge. When estimating pollutant removal efficiency, knowledge of estimate uncertainty is important for understanding estimate reliability. In this study the uncertainty (coefficient of variation, *CV*) in pollutant degradation rate ($K_1$) and relative concentration at 35 days of composting ($C_{35}/C_0$) was evaluated. This was done based on recently presented pollutant concentration data, measured under full-scale composting conditions using two different sampling methods for a range of organic pollutants commonly found in sewage sludge. Non-parametric statistical procedures were used to estimate *CV* values for $K_1$ and $C_{35}/C_0$ for individual pollutants. These were then used to compare the two sampling methods with respect to *CV* and to determine confidence intervals for average *CV*. Results showed that sampling method is crucial for reducing uncertainty. The results further indicated that it is possible to achieve CV values for both $K_1$ and $C_{35}/C_0$ of about 15%.



## 1. Introduction

Sewage sludge is a common urban biodegradable waste material but it is also a valuable resource containing organic matter, nitrogen and phosphorous (Chen and Bester, 2009) and is widely used in agri-, silvi-, and horticulture to improve soil structure and fertility (Sadef et al., 2015). Sludge application increases soil organic matter (Gabrielle et al., 2005), and improves soil structure and aggregate stability (Annabi et al., 2007) which control soil water holding capacity, soil aeration, and plant root development. Composted sewage sludge is increasingly used due to its ability for rebuilding soil organic matter, provide nutrients, and suppress plant diseases (Ostos et al., 2008). It is easier and more economical to store and handle due to its lower water content and odor compared to raw or digested sludge. Sludge, however, very often contains hazardous compounds such as heavy metals, organic pollutants, and pathogens. Sludge potentially contains a wide range of organic micro-pollutants at elevated concentrations (Brändli et al., 2005; Buyuksnomez et al., 2000; Hogg et al., 2002; Kupper et al., 2008; Poulsen and Bester, 2010; Sadef et al., 2013) originating from both industrial and domestic (household) sources. These are used in personal care products, detergents, plasticizers, cleaning agents, pharmaceuticals, and flame retardants, etc. (Aparicio et al., 2009; Poulsen and Bester, 2010; Sadef et al., 2013). As the number of organic chemicals used in industry and households increases continuously, so do the number found in sludge. Many organic micro-pollutants have adverse environmental and human health effects and are of potentially major concern (Sadef et al., 2013). In some regions, the food industry does not accept agricultural products (vegetables, grain, meat, milk, eggs, etc.) produced using sludge (including use for animal feed production). As a result, farmers are increasingly reluctant to use sludge (Buyuksnomez et al., 1999). This has caused increased difficulties with respect to sludge management and disposal especially because landfilling of sludge in many regions has been outlawed.

Aerobic composting facilitates removal of organic micro-pollutants in sludge (Brändli et al., 2007; Buyuksnomez et al., 1999, 2000; Chen and Bester, 2009; Poulsen and Bester, 2010; Rihani et al., 2010). Composting therefore, seems viable for reduc-

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ing sludge micro-pollutant concentrations. Microbial degradation rate is the key parameter controlling the efficiency of composting in removing organic micro-pollutants. Sludge is usually mixed with other materials such as straw and yard/park waste during composting and hence compost is often very heterogeneous, especially early in the process. Consequently, contaminant concentration measurements, especially during full-scale composting are potentially associated with significant uncertainty (Sadef et al., 2014a). Knowledge about the level of uncertainty in both concentration and degradation rate measurements is crucial when evaluating the reliability of composting as a remediation method. Recently Sadef et al. (2013) and Sadef et al. (2014a) presented a comprehensive procedure for sampling and measuring contaminant concentrations in compost to reduce measurement uncertainty. This procedure was later applied in measurements of organic contaminant degradation during sewage sludge composting (Sadef et al., 2014b, 2014c, 2015). Micro-pollutant concentration measurement uncertainty using this method was assessed based on a limited set of measurements taken at a single sampling event early in the composting process (Sadef et al., 2014a) and the method was found to significantly reduce concentration uncertainty compared to earlier methods. Due to the limited data used, however, Sadef et al. (2014a) were not able to assess the uncertainty in contaminant degradation rate and removal efficiency using their method as this requires measurements of contaminant concentrations at multiple points in time throughout the composting process. Sadef et al. (2014a) further compared the accuracy of their method to a simpler approach by Poulsen and Bester (2010). Although the data set by Poulsen and Bester (2010) contains concentration measurements at multiple times during the composting process, Sadef et al. (2014a) focused on assessment of concentration uncertainty at specific points in time (for comparison to their own data) and did not attempt to assess the uncertainty associated with contaminant degradation rate or removal efficiency. To the knowledge of the authors, there have been no other attempts in the published literature to quantify the uncertainty in organic micro-pollutant degradation rates and removal efficiency during full-scale commercial composting using comprehensive sampling methods. There is thus, a need to investigate this issue further to provide information about the general reliability of degradation rate measurements and the suitability of composting as a potential remediation method for organic chemicals.

The objective of this study was therefore, to quantify the uncertainty in organic micro-pollutant degradation rates and removal efficiency using the sampling method of Sadef et al. (2014a) during active full-scale commercial windrow composting. The evaluation was based on two previously published data sets for organic pollutant concentrations as a function of time during full-scale commercial composting of sewage sludge (Poulsen and Bester, 2010; Sadef et al., 2015). These data were chosen as they represent the largest and most comprehensive data sets currently available involving key groups of organic micro-pollutants commonly found in sewage sludge (i.e., household chemicals, personal care products and industrial additives, Poulsen and Bester, 2010; Sadef et al., 2013). The two data sets further represent two different measurement approaches; the comprehensive approach of Sadef et al. (2013) and a simpler and more traditional approach used by Poulsen and Bester (2010). Quantification of the uncertainty in organic micro-pollutant degradation rates and degradation efficiency was carried out for both methods.

## 2. Theory

Biological degradation of organic micro-pollutants in sewage sludge during composting has been documented to follow first order degradation kinetics (Poulsen and Bester, 2010; Sadef et al., 2014b, 2014c, 2015) as:

$$\frac{C}{C_0} = e^{-K_1 t} \tag{1}$$

where $C_0$ and $C$ are the initial and actual (at time $t$) micro-pollutant concentrations, respectively, $t$ is time and $K_1$ is the first order degradation coefficient. Eq. (1) can be rewritten in linear form to:

$$\ln(C) = \ln(C_0) - K_1 t \tag{2}$$

The value of $K_1$ is estimated as the slope of the best-fit straight line to a set of $\ln(C)$ versus $t$ measurements. A commonly used measure of the uncertainty (or variability) in a given parameter, that is independent of the parameter value, is the coefficient of variation (CV) defined as:

$$CV = \frac{s}{m} \tag{3}$$

where $s$ is the standard deviation and $m$ is the mean of the parameter. For small data sets estimates of $s$ and $m$ (and thus, $CV$) in Eq. (3) can be very uncertain, especially for very variable data. In such cases, $CV$ estimates can be improved by generating a larger data set with the same probability distribution as the measured data and then estimating $CV$ from this larger data set. As the statistical properties (including $m$ and $s$) of the true distribution for the measured data are usually not known exactly, generation of the larger data set is usually done by a non-parametric method such as bootstrapping (Efron and Tibshirani, 1993). To estimate $CV$ for $K_1$ for a selected organic compound, bootstrapping is applied as follows: From the $n$ measured $\ln(C) - t$ values for the compound, one value is randomly selected and recorded. This is repeated $n$ times (the same $\ln(C) - t$ value may be selected more than once) yielding a new (artificial) $\ln(C) - t$ data set also containing $n$ values. This procedure is applied $N$ times (where $N$ is a large number) yielding $N$ artificial $\ln(C) - t$ data sets each containing $n$ data points. For each of these $N$ data sets, a value of $K_1$ is then fitted by linear regression using Eq. (2), yielding $N$ $K_1$ values from which $CV$ for $K_1$ is estimated using Eq. (3).

In case the $CV$ values are not normally distributed, a 95% confidence interval for the mean $CV$ across all organic compounds ($m_{CV}$) can also be estimated using bootstrapping. In this case the procedure is as follows: Initially $N$ artificial $CV$ data sets each containing $n$ $CV$ data points are generated from the individual $CV$ values for each organic compound (estimated using the above approach). For each of these $N$ data sets, the average $CV$, and standard deviation in $CV$ are calculated. These are then used to calculate a statistic given as:

$$T^* = \frac{m_{CV^*} - m_{CV}}{\frac{s_{CV^*}}{\sqrt{N}}} \tag{4}$$

where $T^*$ is the statistic, $m_{CV*}$ and $s_{CV*}$ are the mean and standard deviation in CV for the artificial data sets, respectively and $m_{cv}$ is the mean $CV$ for the original data. This will yield $N$ $T^*$ values which are then ranked from smallest to largest. The 95% confidence interval for $m_{CV}$ is then given as:

$$Lower\ limit = m_{CV} + T^*_{0.025N} \frac{s_{CV}}{\sqrt{N}} \tag{5a}$$

$$Upper\ limit = m_{CV} + T^*_{0.975N} \frac{s_{CV}}{\sqrt{N}} \tag{5b}$$

where $s_{CV}$ is the standard deviation for the original $n$ $CV$ data and the subscript on $T^*$ refer to the rank. Values of $CV$ were determined for the degradation rate ($K_1$) and for the relative reduction in micro-pollutant concentration after 35 days (5 weeks) of composting ($C_{35}/C_0$, calculated based on $K_1$) using the above procedure. This