



Potential agronomic and environmental properties of thermophilic anaerobically digested municipal sewage sludge measured by an unsupervised and a supervised chemometric approach

Barbara Scaglia^a, Fulvia Tambone^a, Luca Corno^a, Valentina Orzi^a, Yuri Lazzarini^b, Gilberto Garuti^b, Fabrizio Adani^{a,*}

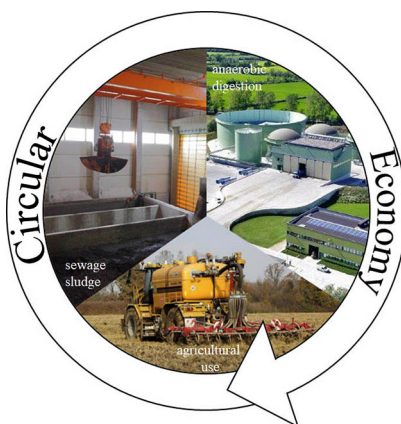
^a Gruppo Ricicla Labs – DiSAA, Università degli Studi di Milano, Via Celoria 2, Milano, Italy

^b Acqua e Sole, Centro Operativo, Via Giulio Natta, 27010 Vellezzo Bellini (PV), Italy

HIGHLIGHTS

- Anaerobic digestion of Sewage sludge (SS) produces SS-dig. to be used in agricultural use.
- The effects of SS-digestate on soil need to be evaluated before field application.
- Chemometric supervised (KNN) and unsupervised (PCA) methods were applied with this aim.
- The KNN resulted better than PCA to compare the SS-dig. effects with other biomasses.

GRAPHICAL ABSTRACT



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ABSTRACT

Anaerobic digestion (AD) is the most widely used method of sewage sludge treatment (SS) before its agricultural use. AD achieves the required “sterilisation” of pathogens and is able to cover the energy required by the process, reducing pre-treatment costs, thanks to the production of biogas.

The SS agronomic (fertilizer properties), environmental (pollutants contents) characteristics and nuisance to people (odours and pathogens) need to be evaluated together for the safe and useful deployment of SS in agriculture.

To evaluate SS properties an unsupervised (Principal Component Analysis) and a supervised (K nearest neighbours) chemometric approach was applied to rank digested SS for agronomic and environmental properties in comparison with other organic matrices for which the agronomic and/or environmental properties are well known or expected.

To do so, complete chemical, biological and “impact on people” characterization was carried out on SS ingestate (SS-ing.) and SS digestate (SS-dig.) and another 10 biomasses.

The SS-AD process enhanced the agronomic properties of sewage sludge and did not lead to a substantial concentration of pollutants because of the low degradation of organic matter.

* Corresponding author at: Gruppo Ricicla Labs – DiSAA, Università degli Studi di Milano, Via Celoria 2, Milano, Italy.

E-mail address: fabrizio.adani@unimi.it (F. Adani).

The best PCA performances were reached for amendment and fertilizer modules but the results found for the environment and nuisance to inhabitants were not satisfactory.

The KNN approach proposed to evaluate the suitability of a biomass for agricultural purposes, represents a win-win approach as it allows one to avoid time-consuming and costly full field studies.

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1. Introduction

Sewage sludge results from wastewater treatment and it is a material composed of organic matter (OM), microbial bodies and ash.

Sewage sludge world-production amounts to 105,369,000 Mg year⁻¹ (Mateo-Sagasta et al., 2015) and the EU produces about 10.13 million tons of dry solids (European Commission, 2008).

EU-rules (1999/31/CE) requested the progressive reduction of bio-degradable waste landfilling (EU, European Parliament, 1999), including SS, so that alternatives for SS disposal have to be found.

New proposals for SS disposal include SS burning and agricultural use. Incineration has very high costs and SS first needs to be dried consuming large amounts of energy (Mateo-Sagasta et al., 2015). Nevertheless, this approach is used in many countries such as for example, Netherlands, Germany and UK (Mateo-Sagasta et al., 2015).

Agricultural spreading represents an alternative to SS burning, it is well-established method for SS disposal (Nkoa, 2014; Yoshida et al., 2015; Yoshida et al., 2018) and in EU nearly 40% of SS produced is estimated to be spread on land for agricultural use (European Commission, 2008).

The Sewage Sludge Directive 86/278/EEC (Council Directive, 1986) seeks to encourage the use of sewage sludge in agriculture and to regulate its use in such a way as to prevent harmful effects on soil, vegetation, animals and man. To this end, it prohibits the use of untreated sludge on agricultural land unless it is injected or incorporated into the soil. Treated sludge is defined as having undergone "biological, chemical or heat treatment, long-term storage or any other appropriate process so as significantly to reduce its fermentability and the health hazards resulting from its use" (European Commission, 2008).

Among biological treatments, anaerobic digestion (AD) is the most widely used method of sludge treatment (Gianico et al., 2016; Montalvo et al., 2018; Liu et al., 2018), which achieves the required "sterilisation" or pathogen kill (Smith et al., 2005; Ziemba and Peccia, 2011; Scaglia et al., 2014). Moreover, because of the production of biogas, AD is able to cover the energy required by the process, reducing pretreatment costs (Jenicek et al., 2012; Gebreyessus and Pavel, 2016).

The use of SS in agriculture poses some environmental concerns because of the presence of organic pollutants (OP) and heavy metals (HM). Anaerobic digestion could affect the presence of pollutants because the organic matter (OM) degradation during AD determined HM concentration and OP could be transformed, degraded or concentrated.

The current National and regional regulations (e.g. Italian Parliament, 1992; Lombardy Region, 2014) and recent EU Working Document (European Commission, 2000) pose SS concentration-limits for pathogens, heavy metals and OP, but unfortunately they did not consider emerging micro-pollutants such as metabolic active substances, antibiotics and pharmaceuticals that can cause adverse effects on human health (Boix et al., 2016), as well as potential SS odour emission that is an emerging issue to be considered in the agricultural SS employment because of the adverse effects on human health and nuisance to the population (Woodbury et al., 2016; Stocker et al., 2017).

Testing the appropriateness of biomasses to be used in agriculture needs chemical characterization to meet law regulation and field trials to test agronomic performance (Boudat-dechamps et al., 2017; Mousavi et al., 2017; Matos et al., 2018; Iglesias et al., 2018). Full field trials are time-consuming (years) and involve high research costs, and may not provide adequate answers because SSs have different

characteristics and the results obtained cannot be generalized. Various techniques are being applied to enable fast and inexpensive evaluation of SS fertilizers and their environmental properties. Particular attention has been paid to chemometric methods that give valuable information starting from a wide range of complex data sets and facilitate the detection of hidden relationships between variables (Efenberger-Szmechtyk et al., 2017). The most extensively methods applied are for the unsupervised methodology such as the Principal Component Analysis (PCA) and the Cluster Analysis (CA). These kind of methods synthesize the input information (i.e. analytical data) into a condensed form of output revealing samples' similarity/dissimilarity degrees and they can also indicate which analytical parameters contribute most to the differentiation among samples. PCA is very often applied to SS characterization because of the possibility to display the result in a two/three dimensional plot leading to an easy output interpretation (Tambone et al., 2010; Bhaduri et al., 2017; Ivanová et al., 2018; Caldeira et al., 2018).

However, PCA is not always an effective chemometric method, showing some limits: (i) the number of samples can influence the result, (ii) the significant PCs are sometimes >3 becoming not easily graphically interpretable, (iii) no clear separation among samples is ever achieved (iv) PCA is suitable when there are obvious differences between the samples but it can fail to separate samples having small differences and finally (vi) the samples ranking is based on qualitative criterion (Efenberger-Szmechtyk et al., 2017).

Taking into consideration these aspects, supervised pattern recognition methods such as the Linear Discriminant analysis (LDA), K-nearest neighbour (KNN) and others were recently preferred, based on their higher capability to discern among samples (Efenberger-Szmechtyk et al., 2017). The KNN (Altman, 1992) presents several advantages: (i) mathematical simplicity, (ii) is free from statistical assumptions, (iii) its effectiveness does not depend on the space distribution of the samples (Berrueta et al., 2007), iv) samples distance is a quantitative measure. The KNN is actually employed in some scientific sectors (i.e. alimental, pharmaceutical, etc.) but to our knowledge had previously been applied to the SS study only once (Efenberger-Szmechtyk et al., 2017; Kim et al., 2016).

In this work, the AD was tested at a pilot scale to pre-treat SS with a view to applying this process at full scale and to employ the digestate (SS-dig.) for agronomic uses. With this purpose the AD effect on contaminant species was been considered and an SS-dig. was completely evaluated for the fertilizers and environmental properties. While doing so, biomass quality categories (agronomic effect, inhabitant nuisance generation and contaminant soil supply phenomenon) were identified by using proper analytical data; then a comparison with other biomasses for which the same properties were well-known or well-identifiable was done. The comparison was carried out by applying an unsupervised and a supervised approach by using PCA and KNN methods, able to rank sewage sludge characteristics vs. other biomasses studied.

2. Materials and methods

2.1. Biomass origin and collection

2.1.1. Sewage sludge-AD treatment and sample collection

Untreated and digested sewage sludge (SS-ing. and SS-dig.) were sampled at a pilot-scale plant of 0.78 m³ volume. The reactor performed

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