Analysis of biological stability of dried sewage sludge from anaerobic digestion mixed with inorganic by-products

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1. Introduction

According to the UN Sustainable Development Goals, namely Goal 2 related to "*end hunger, achieve food security, and improved nutrition and sustainable agriculture*", fertilizers must be used in a balanced way to maintain global crop productivity at current levels. The macronutrients for plants (N, P, and K) are mainly supplied by synthetic fertilizers. However, due to intensive agriculture, increased fertilizers prices, and tensions in the phosphorus market, reliance on synthetic fertilizers can create difficulties in the crop production (Brunelle et al., 2015; Roberts, 2009). In this scope, sewage sludge (SS) from wastewater treatment plants (WWTP) can have a relevant role in the future of the fertilizers and soil conditioners industry. Indeed, SS contains high levels of organic matter (OM) and macronutrients, with a positive effect on the physical and chemical properties of the soil. Nevertheless, this management route requires attention to the concentration of potentially toxic metals, organic pollutants, microplastics, and pathogens (Gomes et al., 2019). Additionally, the organic matter stability is one of the most relevant parameters to consider for a safe application on the soil. The respirometric assays based on the O_2 consumption seem to be a reliable option to determine the aerobic biological activity of sewage sludge. According to the new Fertilizers Regulation (EU) 2019/1009, oxygen uptake rate (OUR) is considered as "*an indicator of the extent to which biodegradable organic matter is being broken down within a specified time*", and should be determined, in some categories, to frame a new fertilizer into the market. In this context, this work aims to assess the aerobic biological activity of raw SS and after conditioning with industrial inorganic by-products or drying process. The biological stability of the materials will be determined based on the OUR parameters using an *Oxitop*® apparatus.

2. Materials and methods

SS sample was collected after anaerobic digestion and dehydration by centrifugation from a Portuguese WWTP with a capacity of 140,000 population equivalent. Two industrial inorganic by-products were considered: weathered coal fly ash (CFA) from a landfill that receives about 40 kt/year of CFA not complying the cement industry requirements; eggshell (ES) sample from an egg pasteurization industry that produces 4 kt/year. Five different mixtures were prepared with SS, CFA, and ES, aiming at developing an NPK organic fertilizer based on Portuguese legislation (*Decreto-lei* n.º 103/2015). The materials were ground and sieved through a 425 µm screen, and a proper blend was achieved with dried SS at 100 and 130 ºC (SS100 and SS130). The content of OM, N, P and K were determined following the analytical methods described in Gomes et al. (2019). For comparison purposes, it was also tested a commercial organo-mineral fertilizer (OMF).

An *Oxitop*® system was used to perform the respirometric analyses. Thus, hermetically sealed vessel (1 L of capacity) with a pressure sensor were employed. The pressure drop is measured over time and is related to oxygen consumption, as shown in Eq. (1), according to EN 16087-1:2011,

$$RA = \frac{\Delta P}{R\cdot(273{,}15+T)}\cdot\frac{V_{gas}\cdot 10000}{W\cdot DM\cdot OM} \qquad (1)$$

where RA is the respirometric activity (mmol O_2/kg OM), ΔP is the pressure drop (kPa), R is the universal gas constant (mL·kPa/K mol), T is the temperature (°C), W is the fresh sample mass (kg), DM is the dry matter content (%), OM is the organic matter content (%), V_{gas} is the gas volume (mL). The CO_2 produced is retained by NaOH as solid pellets. According to EN 16087-1:2011, the OUR is obtained by dividing the RA by 72 h (time considered for RA determination).

3. Results and discussion

Table 1 presents the formulations of the five different mixtures and their compositions. The requirements related to K content were not achieved in none of the formulations, considering the prerequisites to obtain an NPK organic fertilizer (2% K_2O). It will be necessary to explore alternative sources of K to achieve this goal, such as biomass ashes. Nevertheless, the respirometric activity was determined in these mixtures. Fig. 1 shows the OUR results obtained for fresh SS (FSS), SS dried at 100 and 130 ºC (SS100 and SS130), OMF, and the mixtures with SS at both temperatures (M1-M5). As reported by standard EN 16087-1:2011, the stability limits for considering the stable organic matter in these materials are: 50 mmol O_2/kg OM.h for digested sludges (L3); 25 mmol O_2/kg OM.h for fertilizers and soil amendments intended to professional applications (L2); and 15 mmol O_2/kg OM.h for soil amendments intended to non-professional applications (L1). Fig. 1 (a) shows that the OMF presents the highest

OUR (79.1 mmol O_2/kg OM.h), following by the fresh SS sample (52.1 mmol O_2/kg OM.h). Thus, even though the OMF is commercialized, it does not comply with the limits of stability. On the other hand, as the drying temperature increases, the respirometric activity decreases. When SS was dried at 130 ºC, the OUR is 26.3 mmol O_2/kg OM.h, which is near to the limit of stability for fertilizers.

Table 1. Mixtures preparation and their theoretical compositions.

	Mixtures				
	M1	M2	M3	M4	M5
SS	0.70	0.70	0.80	0.70	0.70
CFA	0.10	0.20	0.10	0.30	0.00
ES	0.20	0.10	0.10	0.00	0.30
Theoretical compositions					
OM (%)	45.45	45.02	51.39	44.59	45.88
Organic N (%)	2.73	2.73	3.12	2.73	2.73
Total P_2O_5 (%)	2.76	2.75	3.11	2.74	2.77
K_2O (%)	0.41	0.64	0.43	0.87	0.18
N + P_2O_5 + K_2O (%)	5.90	6.12	6.66	6.35	5.68

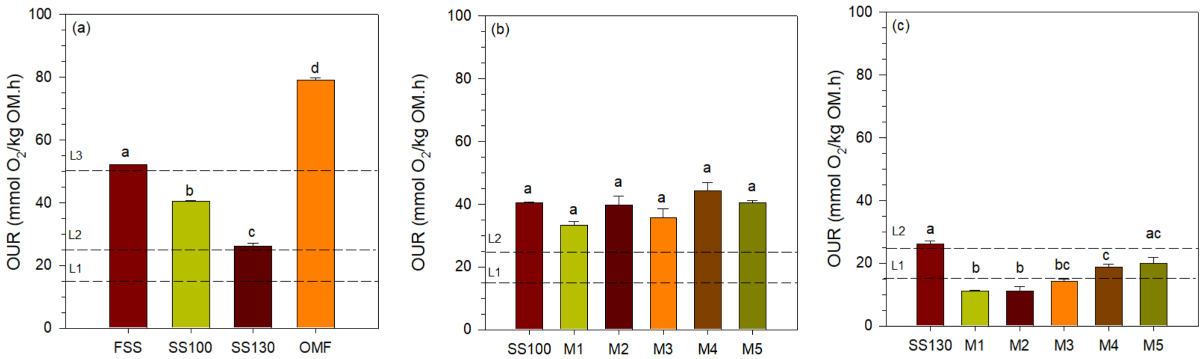

Figure 1. OUR values for (a) fresh SS, dried SS at 100 and 130 ºC, and OMF; (b) mixtures with SS dried at 100 ºC; and (c) mixtures with SS dried at 130 ºC. [L1 – limit for professional application of soil amendments; L2 – limit for fertilizer; L3 – limit for digested SS; Results marked with equal letters are statistically similar ($p<0.05$)].

According to Fig. 1 (b), when 100 ºC was used as drying temperature, the results are statistically similar (in the range of 33.4 to 44.3 mmol O_2/kg OM.h) and below the stability limit for digested sludges (L3). On the contrary, Fig 1. (c) points out that the OUR drops to values between 11.1 and 20.1 mmol O_2/kg OM.h, which indicates that the temperature can be used as a stabilization method. Also, mixtures M1, M2, and M3 present values below the limit established for soil improvers for professional applications (L1- the most restrictive limit). Whereas M5 and M4 are still above this limit but below the limit value for fertilizers (L2), which was not possible to achieve with only SS dried at 130 ºC.

Conclusions

The results indicated that neither the SS dried at 100 ºC nor mixtures thereof allowed to achieve the stability limit for fertilizers and soil improvers for both professional and non-professional applications. However, the further tests with SS dried at 130 ºC showed a decrease in the OUR values below these limits, which indicates that the high temperatures and inorganic wastes can positively act as a stabilization process.

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