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Potential valorization as fertilizers of Humic Substances extracted from landfill leachate

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Abstract
In this study, four leachates samples from 3 different landfills localized in the north of Portugal were characterized and fractionated, to understand the decomposition degree and to evaluate their potential as an agent for fertilization. Humic substances (HS) were extracted, quantified, chemical characterized and further fractionated in humic acid (HA) and fulvic acid (FA). Keeping in mind the purpose to use these fractions as fertilizers, the phytotoxicity of HS, HA and FA solutions was evaluated on cress seed germination. The HS concentration was similar for all the leachates evaluated and was higher than 780 mg/L of total organic carbon. All the leachates analysed registered higher FA concentration than HA. The chemical characterization indicated that HA had a relatively higher aromatic character than the FA obtained from same sources. These results suggest that the HS from landfill leachates were in an early stage of humification, once the degree of humification increase as the landfilling age increase. Overall, the HS extracts showed absence of phytotoxicity, with germination index greater than 80% for samples treated to achieve low electric conductivity values. This suggests that the HS from the leachate may be used to produce liquid organic fertilizers.

1. Introduction

One of the most worldwide options for Municipal Solid Waste (MSW) treatment is landfilling. Although other options arise, several experts still support this option as the most adequate for this purpose. The wastes disposal in landfill generates leachates that are a complex mixture of organic contaminants and water. During the wastes stabilisation process, the biodegradable fraction of organic compounds in leachates decomposes and refractory humic substances (HS), mainly humic acids (HA) and fulvic acids (FA) are produced. These substances have a major role as fertilizers and land correctives, which may contribute to the essential functions of global soil fertility. In particular, the role of humic acids is emphasized. For example, it addition to a soil has a positive effect on the assimilation of nutrients by plants and microorganisms. Indeed, nowadays there is a growing tendency to use humic substances as liquid organic amendment, produced from natural resources such as leonardite, peat, and lignite [1, 2], but their use can contribute to the extinction of these natural resources. However, from the resources sustainability perspective it is important evaluated others HS sources. In this study, it was intended to assess the potential valorization of humic substances extracted from landfill leachate as fertilizers.

2. Experimental

Four leachates samples from three MSW landfills with different landfilling ages (A, B, C) localized in the north of Portugal were used to carry out this study. The landfill ages were 13, 12, 16 and 17 for landfill A, B, C1 and C2, respectively. HS were extracted, quantified, chemical characterized and further fractionated in HA and FA. For the HS extraction from the leachates the methodology applied was the sorption with Amberlit XAD-8 resin [3], using sodium hydroxide and hydrochloric acid as eluents, after resin cleaning condition with sodium hydroxide, ethylic ether, acetonitrile and methanol. The HS fractionation in FA and HA was achieved by a selective precipitation process with pH control, using concentrate sulphuric acid. The total organic carbon (TOC) content of HS, HA and FA was determined by dichromate oxidation and in sulphuric acid medium [4]. The absorvances at 254 nm and at 280 nm of the samples were measured in a UV/VIS spectrometer.

The phytotoxicity evaluation was conducted performing germination tests, using *Lepidium sativum* (water cress) as tested seed. This bioindicator was chosen due to its rapid growth and high sensibility to pollutants. All the tests were done using seven replicates with fifteen seeds, in Petri dishes with sterile filter paper humidified with 4 millilitres of the testing solution. Blank tests were also done. Some of the HS solutions were dialysed or diluted in order to decrease the electric conductivity (EC) to values lower than 4 mS/cm, while others solutions were used without any further treatment. A leonardite based commercial liquid fertiliser (Humistar®) and a HA standard (Aldrich®) were included as control. The germination index (GI, %) was evaluated by equation 1.

$$GI = \frac{\overline{GS}_s * \overline{RL}_s}{\overline{GS}_c * \overline{RL}_c} * 100 \qquad (1)$$

Where $\overline{GS}$ represents de average number of germinated seeds and $\overline{RL}$ the average root length (mm) of seeds in samples – s, and the blank – c tests.

3. Results and Discussion

The HS concentration was similar for all the leachates evaluated and was higher than 780 mg/L of total organic carbon (Table I). All the leachates analysed registered higher FA concentration than HA. These results were higher than the ones reported by Fan et al that ranged between 100 and 202 mg/L for FA and 72 and 101 mg/L for HA [5]. Moreover, the concentration values found for HS were greater than the concentration found in the natural aquatic resource of HS [1]. Overall, the data indicate that the leachate may be a source of HS, because the higher carbon content of the samples.

Table I Quantification of HS, FA and HA obtained from the landfill leachates.

Landfill Leachate	HS	FA	HA
	[mg TOC/L]		
A	1211	845	162
B	839	677	28
C1	780	288	30
C2	1311	966	631

Once, the FA concentrations values are higher than the HA, is possible to state that the landfills are not in the maturation phase [6]. These results suggest that the HS from landfill leachates were in an early stage of humification, once the degree of humification increase as the landfilling age increase.

The absorbance at 254 nm gives the total dissolved organic matter (DOM) in the solution and the values at 280 nm show the organic matter aromaticity degree (Ar_D) [7]. This characterization was made for leachates from landfill C and the results obtained are described in Table II.

Table II Total dissolved organic matter (DOM) and the organic matter aromaticity degree (Ar_D) values for leachate of landfill C

Landfill leachate		HS	FA	HA
C1	DOM [mg/L]	11733	10289	nd
	Ar_D [mg/L]	7454	6126	nd
C2	DOM [mg/L]	4064	3570	2614
	Ar_D [mg/L]	3204	1786	2570

nd – not determined

Overall, the DOM and the Ar_D content are in the same order in the HA, behaviour not seen in HS and FA, where a slight difference is verified. It was found that the aromatic organic matter represents only 50% of the total dissolved organic matter for the FA. In opposition, the organic matter aromaticity of the

HA reached 98% of the total dissolved organic matter. This fact indicat that the HA fraction is constituted by organic matter with high aromatic degree, which is in accordance with the literature [8].

The chemical characterization indicated that HA had a relatively higher aromatic character than the FA obtained from same sources. This aromatic character of HA may influence the behaviour of some aquatic pollutants. These substances may increase the solubility of hydrophobic compounds by complexation or partition and modify the bioavailability and biotoxicity of certain compounds. This information is an important knowledge, allowing to understand the interactions of HS with pollutants in terrestrial and aquatic environments. It is also important to optimize the leachate treatment processes with respect to landfill age, because these substances are refractory and may difficult the leachate treatment.

The HS, mainly the HA have several applications, such as fertilizers and land correctives, and may be applied in soils however its toxicity must be previously evaluated. The HS, FA and HA were submitted to different conditions: landfill leachate A was dialysed; landfill leachate C1 was used without any further treatment and landfill leachate C2 was diluted. Dialysis and the dilutions were made in order to decrease the electric conductivity (EC) to values lower than 4 mS/cm [9]. Overall, the HS extracts treated to achieve low electric conductivity, showed absence of phytotoxicity, with germination index greater than 80% (Table III). These GI values were lower than the value reached for HS commercial liquid fertilizer Humistar® (154%).

Table III Germination Index (GI) for landfill leachate A and C

Landfill leachate		**HS**	**FA**	**HA**
A	**GI (%)**	113	nd	nd
	EC (mS/cm)	0.2	nd	nd
C1	**GI (%)**	0	nd	nd
	pH	12.6	nd	nd
	EC (mS/cm)	10.0	nd	nd
C2	**GI (%)**	115	0	68.0
	pH	12.1	2.0	11.0
	EC (mS/cm)	3.56	3.80	3.06
Commercial liquid fertilizer (Humistar®)	**GI (%)**	154	-	-
	pH	10.3	-	-
	EC (mS/cm)	0.4	-	-
HA Standard (Aldrich®)	**GI (%)**	-	-	123
	pH	-	-	11.9
	EC (mS/cm)	-	-	1.93

nd – not determined

The HA sample from C2 leachate achieved a GI value of 68%, that is lower than the GI value obtained for the HA standard Aldrich® (123%), but nevertheless indicating that the sample is not phytotoxic. This suggests that the HS extract from the leachate may be used to produce liquid organic fertilizers an important way to valuing this type of waste. Moreover, the conductivity is an important chemical parameter in the toxicity once the samples without treatment showed absence of germination. The pH demonstrated also a negative effect in the seed germination. Although the dilution made of 1:5 for HS, 1:200 for FA extract, and 1:2.5 for HA, to decrease the EC value, the pH character did not change, which may indicate buffering effect of the humic material. With these results it can be stated that dialysis can be replace by the dilution, in order to reduce the phytotoxicity character of HS and HA, making the process less time consuming and expensive.

4. Conclusions

In this work, it has extracted HS and fractionated in humic and fulvic acids from three landfills with different landfilling ages, and conducted their characterization. It has identified that the leachate HS, HA and FA aromatic components increased with increasing landfilling age. These results suggest that the degree of humification increased as the landfilling age increased. HS content in the leachates were higher than the typical values found in the natural aquatic humic sources. It was found that HA are constituted by organic matter with a higher aromatic degree than FA. In summary, the results suggest that HS of landfill leachate may be used as liquid organic fertilizers, substituting those produced from natural resources.

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