Extraction of Humic and Fulvic acids from sewage sludge at different pH

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Introduction

Humic and fulvic acids (HFA) are biostimulants used to enhance plant growth and soil fertility that are usually recovered from non-renewable sources such as lignite or peat. In a circular economy context, sewage sludge could be a promising alternative to recover HFA due to their wide availability, continuous production, and stable physic-chemical characteristics.

Alkaline treatment of sludge has been showed as a promising way to recover HFA since a 0,1 M NaOH solution (pH 13) could dissolve more than half of the sludge organic matter (Li et al. 2009, Li et al. 2014), enabling their separation from the sludge solid phase and facilitating their recovery. The HFA extraction at pH 13 will be used as reference to compare the results obtained in this study.

The aim of this study is to assess the extraction of HFA from dehydrated sewage sludge at different pHs to find a balance between the use of reagents and the HFA production with the prospect of a future scaling up at industrial scale.

Materials & method

Dehydrated sludge samples were provided by a WWTP located in Vilanova i la Geltrú (Spain) and was characterised in terms of humidity and total organic carbon (TOC). For HFA extraction, a litre of extractant (at pH 10 – 13 with a step of 0.5) was mixed with 7g of dehydrated sludge and was stirred for 6 hours at room temperature. Afterwards, the mixtures were centrifugated for 45 minutes at 4100 rpm at 4ºC to separate the supernatant from the solid fraction. All experiments were conducted for duplicated to ensure the reliability of the results.

After extraction, HFA content in the separated supernatant was analysed according to the ISO 12782-5:2012 while the remaining solid fraction was dried at 105ºC for 24 hours for TOC.

The pH adjustments were performed using NaOH 5M and H2SO4 96% (Scharlab, Spain), TOC of liquid streams were analysed using a Shimadzsu TOC-L CSH/CSN, and TOC of solids was analysed using the LECO RC612L equipment

Results & Discussion

After alkaline extraction, results are presented as mg of HFA per kg of dry sludge -considering 28.52% of dry matte in dehydrated sludge-. The values obtained at different extraction pH are showed in Figure 1:

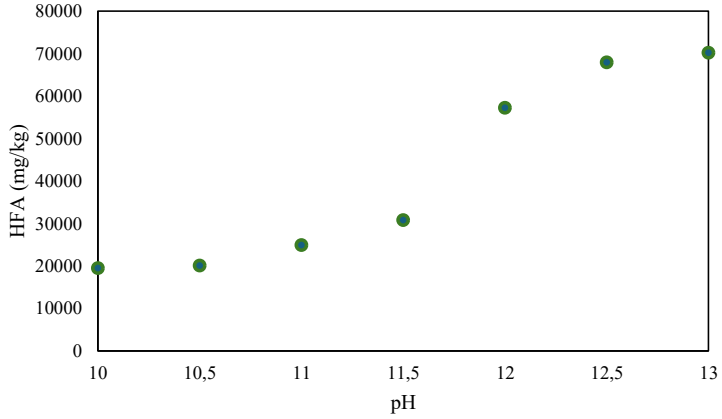

Figure 1: HFA extraction (mg/kg) at different pH.

The total amount of HFA is used to determine the efficiency of the extraction. Starting at 70234 mg/kg at pH 13 -state of the art conditions-, there is a clear tendency to the decrease of extracted HFA when pH is decreased, which agrees with what is stated in the bibliography. As shown in Figure 1, the extraction of HFA at different pH values follows a S-shape where the most significant slope modification occurs at pH between 11 and 12.5.

Bellow pH 11, the extraction efficiency does not undergo any further extreme change, achieving stable values (with less than 1% difference). Even though the solubility low solubility of HFA at lower pH, part of HFA are easily released at pH 10 and 10.5, as at these points a similar concentration is obtained, corresponding mainly to humic acids.

Moreover, at pH over 12.5, no significant improvement on HFA extraction is observed, showing the exhaustion of sewage sludge samples, thus a nearly total extraction of HFA.

From these results, it can be observed that the effect of extractant liquid pH on HFA extraction follows a S-shaped relation. Focusing on the balance between HFA extraction and reagents use, the best pH for future scale up is pH 12. This pH provides a high extraction, being less than 20% below the maximum obtained at pH 13. Using pH values below that point would be too detrimental on the extraction of HFA. Furthermore, decreasing just by 1 point the pH value on an industrial scale, an enormous quantity of reagent would be saved, allowing a high extraction while reducing costs and minimizing environmental impact.

Conclusions

In this study, the effect of pH on HFA extraction from sewage sludge has been evaluated. It has been observed a decrease in the extraction efficiency of HFA at lower pHs, finding its most significant reduction occurring between pH 11 and 12.5. The HFA extraction at pH 12 is selected as the best operational condition for a future scale up, achieving less than 20% of HFA recovery rate reduction compared with the values obtained at pH 13 (used as reference). These working conditions allow a balance between the efficiency of HFA extraction and the use of reagents, enabling a reduced environmental impact related to the extraction process.

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