



# Potential of the electrodialytic process for emerging organic contaminants remediation and phosphorus separation from sewage sludge



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## ABSTRACT

The present work discusses the efficiency of the electrodialytic process (ED) applied to sewage sludge aiming phosphorus recovery and organic contaminants removal. Six emerging organic contaminants were studied: caffeine (CAF), ibuprofen (Ibu), 17 $\beta$ -oestradiol (E2), 17 $\alpha$ -ethinyloestradiol (EE2), bisphenol A (BPA), and oxybenzone (MBPh). Three different ED experimental cell designs were tested, in a 2-compartment cell set-up, using sewage sludge spiked with 8 mg/L of each compound. In total, five ED experiments were carried out during 5 days. Two control experiments without current were also carried out. At the end of the experiments, changes in the sewage sludge pH and in the microbial communities were observed and these changes affected contaminants degradation. Still, independently of the cell design used, the application of a low level direct current, improved the degradation of Ibu between 46 and 97%, as well as Caf between 20 and 47% (except in Exp-4), comparing against the control experiments. A contrary effect was observed for MBPh which degradation was less effective when the sewage sludge was subjected to ED, being the only exception the results obtained for Exp-5. This cell design, sewage sludge in the cathode compartment separated from the anode by an anion exchange membrane, also promoted higher contaminants degradation and in the final sludge all contaminants, except MBPh, were below limit of quantification and limit of detection. In terms of P, the highest recovery was achieved when the pH increased to 12.5 in Exp-5, 78  $\pm$  2% of total P was recovered in the electrolyte. Independently of the experiments, the recovered P was still mixed with the studied organic contaminants.

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## 1. Introduction

Phosphorus (P) is essential to all life being and a key ingredient in fertilizers to sustain high crop yields. It has no substitute in food production and in a world of 9 billion people by 2050, securing sufficient P will be critical for future food security [1]. The world's remaining high-grade phosphate rock reserves are concentrated in only a few countries, with Morocco and Western Sahara controlling approximately 75% of the world's share [2]. This makes phosphate-importing countries like European Union (EU) vulnerable to geopolitical dynamics in producing countries [3].

The use of sludge is considered a viable option to maintain/enhance the recycling of elements with socio-economic value like P, other nutrients and organic matter, being advocated as a means

of avoiding the environmental and economic costs of disposal [4]. It has been estimated that in EU-27 by 2020, 43% of the treated sludge will be used in agriculture [5]. However, direct use of treated sewage sludge in agriculture may be difficult due to public perception, odours or difficulties of transport and storage and, in other cases it is not feasible due to the presence of contaminants like organics, heavy metals and pathogens.

Degradation and attenuation during wastewater and sludge treatment remove significant amounts of organic contaminants. But many of these compounds are transferred to sewage sludge and may be present in residual concentrations in the dry solids, depending on their initial amounts, their lipophilicity and the extent of destruction during treatment [6]. The organic contaminants that can be found in low concentrations in the environment often suffer biomagnification and bioaccumulation into biological organisms and cause irreversible damages in biological systems through direct or indirect toxic effects such as endocrine disruption and tumour promotion [7].

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If P recovery from sewage sludge is the final target the process generally aims at: (i) separating contaminants from the valuable P and, if possible, (ii) converting P either into a plant-available form for reuse as fertilizer or into a raw material for the P-industry. If P recovery is successful, it offers the immediate advantage of avoiding the environmental impacts associated with primary production from phosphate rock, complemented with the reuse of an essential nutrient that is being wasted. For these reasons, the present technologies that promote P reuse within the wastewater treatment scheme are particularly attractive, if they achieve a product which is free from contaminants and of high quality as fertiliser.

The electrokinetic process is a method that relies on the application of a low level direct current (DC) for the removal of contaminants from soils and other porous matrices [8]. Electrokinetic treatment relies on several interacting mechanisms but the dominant and most important electron transfer reactions that occur at electrodes during the process is the electrolysis of water, which will cause a pH increase and decrease in the cathode and anode compartments, respectively.

Few studies tested the applicability of the electrokinetic process (EK), using passive membranes, and the electro-dialytic process (ED), using ion exchange membranes, for heavy metal [9–13] and surfactant [14] removal from sewage sludge. More recently, the applicability of the EK for the remediation of emerging organic contaminants from soil was carried out in a stationary laboratory cell [15]. The results showed that EK is a viable method for the remediation of organic contaminants, both through mobilization by electroosmotic flow and electrodegradation. Still, to the best of our knowledge, no works have been published regarding P recovery and simultaneous organic contaminants remediation from sewage sludge using ED.

In order to improve the process, adjustments can be made to the earlier established three compartment ED cell [16] depending on the final objective. For example, a stirrer can be introduced in the central cell compartment [17] or the number of cell compartments can be reduced into two [18], introducing the matrix directly into the anode compartment.

In this work, the ED process was applied to the sewage sludge aiming a simultaneous P-separation and organic contaminants remediation. In the case of the contaminants remediation it is expected that bio and/or electrodegradation mechanisms may occur. Six contaminants, classified as emerging, that have been detected in the influent, effluents and sewage sludge samples all over the world [19–21] were selected to the study: two estrogenic steroid hormones, one industrial reagent, one pharmaceutical active compound, one neural stimulant and one sunscreen compound. The target compounds were 17 $\beta$ -oestradiol (E2), 17 $\alpha$ -ethinyloestradiol (EE2), bisphenol A (BPA), ibuprofen (Ibu), caffeine (Caf) and oxybenzone (MBPh). Their chemical properties can be found in Table SM1. An evaluation of the biodegradation potential and overall changes in the microbiological community were also assessed in the ED experiments.

Supplementary material related to this article found, in the online version, at <http://dx.doi.org/10.1016/j.electacta.2015.03.167>.

## 2. Experimental

### 2.1. Chemicals and standards

Caffeine ( $\geq 90\%$ ), BPA ( $\geq 99\%$ ), E2 ( $\geq 97\%$ ), EE2 ( $\geq 98\%$ ) were purchased from Aldrich (Steinheim, Germany) and Ibu from Fluka (U.S.A.). All used solvents were from Sigma–Aldrich (Steinheim, Germany), Panreac (Barcelona, Spain) and Merck (Darmstadt, Germany). Acetonitrile (ACN), methanol (MeOH) and acetone were gradient grade. The water used for analyte extractions and their analytical determinations was deionized and purified with a

Milli-Q plus system from Millipore (Bedford, MA, USA). Formic acid (LC–MS grade), acetic acid (LC–MS grade) and sodium hydroxide (NaOH) were purchased from Sigma–Aldrich. Sodium nitrate (NaNO<sub>3</sub>) was reagent grade from Panreac. Individual stock solutions for calibration purposes were prepared at 400 mg/L in MeOH and stored at  $-18^\circ\text{C}$ . Working solutions were prepared by the adequate mixture and dilution of the stock solutions.

### 2.2. Sewage sludge sampling procedure

The sewage sludge samples were collected at a wastewater treatment plant from *Simarsul* located in Quinta do Conde, Sesimbra, Portugal ( $38^\circ 34' 13''$  N,  $9^\circ 2' 7''$  W). The plant has the capacity to treat in the project horizon 19,300 m<sup>3</sup>/day of urban wastewater, corresponding to about 94,000 equivalent inhabitants. The level of treatment installed is tertiary. The secondary tank is an aerobic reactor, of suspended biomass, where appropriate conditions are secured to promote the development of a population of microorganisms which ensure biological purification of wastewater conditions. The effluent of the reactor is then routed to the secondary settling tank for phase separation, where the samples were collected. The sampling of the sewage sludge was carried out in January 2014, following the recommendations of the norm NF EN ISO 5667-15 (October 2009) on the conservation and treatment of sludge and sediment samples. Sample characterization was carried out at the wastewater treatment laboratory. The sample presented a pH of 7.01, a conductivity of 710  $\mu\text{S}/\text{cm}$ , and a total P concentration of 123 mg P/L. Total solids in suspension and total volatile solids were 4367 and 3783 mg/L, respectively, being the total organic carbon 2011 mg C/L. Five day biochemical and chemical oxygen demand were 1300 and 4700 mg O<sub>2</sub>/L, respectively. The sewage sludge presented a total nitrogen concentration of 395 mg N/L, nitrate was  $< 2$  mg NO<sub>3</sub>/L and ammonia concentration was 3.82 mg NH<sub>4</sub>/L. An initial screening of the studied contaminants was carried out in the sewage sludge used in the experiments and none of the compounds was detected in the samples. The samples were packed in wide-mouth bottles of PTFE and frozen until use.

### 2.3. Extraction procedures for organic contaminants determination

#### 2.3.1. Aqueous samples

The extraction of the analytes present in the electrolytes and effluent (aqueous phase of the sewage sludge) solutions was performed by solid phase extraction (SPE), using Oasis HLB (200 mg, 6 mL) from Waters (Saint-Quentin En Yvelines Cedex, France). The SPE cartridges were conditioned by washing with  $3 \times 6$  mL of methanol, followed by re-equilibrium with  $3 \times 6$  mL of Milli-Q water. For organic compounds enrichment, the samples were acidified to pH 2, using nitric acid, before extraction. The aqueous samples, 200 mL, were passed through the cartridge at a flow-rate of approx. 10 mL/min by applying a moderate vacuum. After that, the cartridges were dried for approx. 2 min by vacuum. The retained analytes were eluted sequentially with  $2 \times 3$  mL of methanol. All the extracts were collected as one and concentrated under a gentle stream of nitrogen till 1 mL. Samples were transferred to a vial and kept at  $-18^\circ\text{C}$  until analysis.

#### 2.3.2. Solid samples

Sludge (solid phase of the sewage sludge) extraction was performed using QuEChERS extraction (adapted from [22]). Extract tubes were obtained from Waters (Dublin, Ireland). The acetate buffer contained 1.5 g NaOAc and 6 g MgSO<sub>4</sub>. The dispersive phase contained 150 mg PSA (primary and secondary amine) and 900 mg MgSO<sub>4</sub>. A 2 g aliquot of homogenized sludge was weighed in a 50 mL polypropylene centrifuge tube containing NaOAc and

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