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Optimizing chemical conditioning for odour removal of undigested sewage sludge in drying processes

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ABSTRACT

Emission of odours during the thermal drying in sludge handling processes is one of the main sources of odour problems in wastewater treatment plants. The objective of this work was to assess the use of the response surface methodology as a technique to optimize the chemical conditioning process of undigested sewage sludges, in order to improve the dewaterability, and to reduce the odour emissions during the thermal drying of the sludge. Synergistic effects between inorganic conditioners (iron chloride and calcium oxide) were observed in terms of sulphur emissions and odour reduction. The developed quadratic models indicated that optimizing the conditioners dosage is possible to increase a 70% the dewaterability, reducing a 50% and 54% the emission of odour and volatile sulphur compounds respectively. The optimization of the conditioning process was validated experimentally.

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1. Introduction

Biological activated sludge is one of the most common processes in wastewater treatment plants (WWTPs), which produces large amounts of sludge wastes. One of the main characteristic of these wastes is the high water content (up to 95% w/w). For this reason, and in order to reduce the transport and disposal costs, dewatering and drying processes are used to reduce the sludge volume ([Feng et al., 2009](#page--1-0)). Prior to these units, the chemical conditioning of the sludge is necessary to improve the efficiency of the dewatering equipment ([Zhai et al., 2012; Bertanza et al.,](#page--1-0) [2014](#page--1-0)). The effectiveness of the ultrasonication of sludge or the application of advanced oxidation processes as conditioning process has been demonstrated by several authors ([Dewil et al.,](#page--1-0) [2005; Khanal et al., 2007; Pham et al., 2010; Ruiz-Hernando](#page--1-0) [et al., 2013](#page--1-0)). However, while these technologies can minimize the excess of sludge, important retroffiting complexities and/or high operational cost can be required ([Vega et al., 2014](#page--1-0)). Traditionally, inorganic chemical conditioners, such as iron chloride and calcium oxide, and organic polyelectrolytes, such as polyacrylamides, have been used. Polyelectrolytes are used to form binding structures while the inorganic conditioners neutralise the charges and adjust the pH of the sludge solids ([Mowla et al., 2013;](#page--1-0) [Zhai et al., 2012](#page--1-0)).

The effectiveness of these chemical conditioners in sludge dewaterability processes has been widely studied ([Tony et al., 2008;](#page--1-0) [Qi et al., 2011; Niu et al., 2013](#page--1-0)); however, their synergistic effects on the emission of odours have been overlooked. This fact is relevant considering that most of the new wastewater solids processing facilities use direct drying technologies leading to the need of manage the large amount of the gas used to dry sewage sludge. The emissions from sewage sludge processing have been characterised by several authors who have been identified the volatile sulphur compounds as one of the main responsible for odorous emissions ([Dincer and Muezzinoglu, 2008; Zarra et al., 2008; Godayol et al.,](#page--1-0) [2013; Lebrero et al., 2013](#page--1-0)). At the other end of the spectrum, the effect of the sludge chemical conditioning on the emission of odour compounds has been previously investigated; however their synergistic effects on dewaterability have also been overlooked. It has been reported how this process affects the odour emission during the sludge conditioning ([Liu et al., 2012\)](#page--1-0) and the field application of the processed sludges (biosolids) ([Chang et al., 2005; Adams et al.,](#page--1-0) [2008; Krach et al., 2008; Laor et al., 2011](#page--1-0)). However, there is a lack of information about how the chemical conditioning affects to the emission of odour compounds during the thermal drying of the

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So clearly, the research to date on chemical conditioning has tended to focus on either improving the sludge dewaterability, or reducing the emission of odorous compounds. The combination of these two objectives has not been previously studied. For this reason, the main goal of this work was to assess the combined effects of chemical conditioners on undigested sewage sludge on dewaterability and odour emissions, paying particular attention on sulphur compounds emissions, during the drying process. The design of the experiments, the model prediction and the determination of the optimum conditions to achieve the optimum dewatering results, and less odour emissions in the conditioning process, were carried out by means of the response surface methodology (RSM) [\(Montgomery, 1996](#page--1-0)).

2. Materials and methods

2.1. Materials

The full-scale WWTP of Girona (Catalonia, Spain), which has a capacity of 206,000 population equivalents (p.e), was selected for the sludge sampling campaign. This WWTP uses the activated sludge process which operates under anoxic and aerobic conditions for biological nutrient removal (nitrification and denitrification). Moreover, the chemical phosphate precipitation is carried out. The sludge used in this study was a mixture collected from the primary and secondary thickener tanks of a WWTP (Girona, Spain). The main characteristics of this sewage sludge are presented in Table 1.

The conditioners used in this study were a commercial cationic polyelectrolyte aqueous solution of 1% (w/w) (Snf Floerger Ibérica, Spain), an iron chloride aqueous solution (FeCl₃) of 30% (w/w) and calcium oxide (CaO) (Scharlau, Spain).

2.2. Experimental set-up

Chemical conditioning was conducted at room temperature (20 \pm 2 °C). The chemical dosage was expressed as gram of chemical per kilogram of dry solids (g kg DS^{-1}). The ranges of conditioner dosages used in this study were based on preliminary experiments and previous literature ([Turovskiy and Mathai, 2006](#page--1-0)): polyelectrolyte 0–10 g kg DS $^{-1}$, iron chloride 0–60 g kg DS $^{-1}$ and calcium oxide 0–100 g kg DS⁻¹.

300 mL of sludge were homogenised using a jar test at 150 rpm during 5 min. Then, the iron chloride and calcium oxide doses were added separately ([Higgins, 2010](#page--1-0)). Once the iron chloride and calcium oxide were added, the sample was mixed for 30 s at 200 rpm. Finally, the polyelectrolyte was slowly added and mixed for 30 s at 200 rpm and then for 90 s at 50 rpm in order to promote the flocculation process.

The dewatering process was carried out using 50 mL of the previous conditioned sludge, which were placed into plastics cells and centrifuged (R380, Hettich Instrument) at 3000 \times g during 30 min. The supernatant was drawn and 30 g of the sludge pellet, which contained $15-18\%$ of DS, were extruded and placed in a glass bottle (1 L) which was capped with a rubber septum. Then, the sludge pellet sample was heated at 85 °C during 1 h in order to

reproduce the temperature conditions of a real sludge thermal drying process at low temperature. The gas phase obtained in the heating process was analysed, combining analytical and olfactometric methods. A schematic diagram of the process is shown in [Fig. 1.](#page--1-0)

2.3. Analytical procedures

The CST is a most commonly used method to measure the filterability and the easiness of removing moisture from sludge as well as determine the required amount of chemical conditioner to achieve the optimal conditions of dewaterability [\(Besra et al., 2005;](#page--1-0) [Sawalha and Scholz, 2007\)](#page--1-0). The CST test measures the filtration rate as the time required for the water to move through of paper located between the two electrodes (Triston electronics Ltd, Type 304 B). A large CST values indicates a high specific resistance to filtration and therefore less dewaterability of sewage sludge ([Sawalha and Scholz,](#page--1-0) [2007\)](#page--1-0).

The extractions of extracellular polymeric substances (EPS) were carried out using the thermal methodology established by [Forster \(1971\)](#page--1-0). According to this method, the total content of EPS was separated into two fractions: the soluble microbial products (SMP) and the bound EPS (b-EPS). First, 100 mL of the conditioned sludge samples were centrifuged at $12,000 \times g$ during 20 min at 4 °C (RC 5B PLUS, SORVALL). The obtained supernatant, which contains the SMP, was subsequently analysed. In order to extract the b-EPS, the remaining pellet was re-suspended using a NaCl 0.9% solution. The obtained mixture was digested in closed tubes at 100 \degree C during 1 h and centrifuged at 6000 rpm during 30 min ([Chang and Lee, 1998\)](#page--1-0). The obtained supernatant, which contains the b-EPS, was analysed. The protein content of both fractions was quantified using a colorimetric method, with the Total Protein Kit (TP03000, Sigma-Aldrich), according to the method proposed by [Lowry et al. \(1951\)](#page--1-0) and modified by [Peterson \(1977\)](#page--1-0).

The concentration of VSCs in gas phase was determined by a headspace-GC method using a gas tight syringe. The GC (CP-3800, Varian) was equipped with a PFP detector and a GS-GasPro column (J&W Scientific). The following VSCs were detected in this study: Hydrogen sulphide (H_2S) , carbon disulphide (CS_2) , methyl mercaptan (MTM), dimethyl sulphide (DMS) and dimentyl disulphide (DMDS). The detection limits for all detected sulphur compounds are 0.5 mg m^{-3}.

The odour unit concentration (OU m^{-3}) was determined by olfactometric analysis which was conducted by a dynamic olfactometer (BioNose 0208, Odournet, Barcelona) according to the guidelines presented in the [European Standard EN 13725 \(2003\)](#page--1-0). A gas sample (0.5 L) was extracted from the bottles used for the thermal drying with a Jumbo Syringe (SGE, Spain) and it was transferred to a nalophane bag in order to determine the odour concentration. The dynamic olfactometer gives as a result different odour ranges: Range 1, 208–450 OU m^{-3} ; Range 2, 450–1077 OU m⁻³; Range 3, 1077–2695 OU m⁻³; Range 4, 2695–9692 OU m⁻³ and Range 5, >9692 OU m⁻³.

2.4. Experimental design

RSM is a set of mathematical and statistical techniques that are suitable for the modelling and analysis of problems in which a response of interest is influenced by several variables ([Montgomery, 1996](#page--1-0)). The central composite design (CCD) was used to evaluate the combined effects of three independent variables (polyelectrolyte, iron chloride and calcium oxide doses) on the dewaterability of the conditioned sludge and on the odour emission during the thermal drying. [Table 2](#page--1-0) listed the range and levels of the studied independent variables.

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