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# Effect of high salt concentration on phosphorus recovery from sewage sludge and dewatering properties

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ABSTRACT

Phosphorus is an essential element in the life cycle of all organisms. Phosphorus rock is mined to produce fertilizers which are necessary to gain high crop yields. Phosphorus rock is limited, predictions of experts vary between 60 and 300 years availability of mineral deposits. A high phosphorus level in water bodies leads to eutrophication which is not only harmful to aquatic ecosystems but can also be a hazard for humans. Sewage sludge in wastewater treatment plants acts as a phosphorus sink and hence represents a promising source for phosphorus recovery.

This paper is a study on the influence of high salinity waters on the dissolved phosphorus content in the liquid phase of waste activated sludge. Next to the dissolution effectiveness as a function of the sodium chloride concentration, the influence of the salt dose on the dewaterability is investigated. It is demonstrated that it is possible to increase the phosphorus content in the liquid phase by high salinity. Furthermore the sludge structure is changed which is beneficial for dewatering processes. In fact, dosing of salt can enhance phosphorus recovery efficiently in cases in which brines or residual salt is available close by wastewater treatment facilities.

## 1. Introduction

The element phosphorus naturally occurs as phosphate which is abundant in mineral deposits and in soil. By physical and chemical weathering, mineral phosphate is transferred into soluble and plant available forms in the pore water of the soil [1,2]. Taken up by plants and transferred further in the food chain, phosphorus plays an essential role as nutrient for all living organisms [3]. Hence, fertilizers containing phosphorus are required for an intensive agriculture, and the availability of phosphorus is a limiting factor for the world's food supply [4,5]. Minable phosphorus rock is concentrated in few countries worldwide. 74% of the phosphorus rock which is still available today is located in only one region: Morocco and Western Sahara [6]. Europe entirely depends on phosphorus imports [7]. As a consequence, there is an increasing tendency towards a more sustainable phosphorus management by closing phosphorus cycles of matter. A promising secondary phosphorus source is wastewater containing the main part of phosphorus waste coming from human consumption [8–10].

In recent years intensive research and development about technologies for the recovery of phosphorus from wastewater were performed. These activities resulted in several promising process concepts for the recovery from sewage sludge or sludge liquor [11]. Sewage sludge represents an important phosphorus sink in the wastewater treatment plant in order to reduce phosphorus emissions and to prevent eutrophication of surface water bodies. Some of the recovery concepts were successfully implemented in full scale, e.g. Pearl® process, Crystalactor, AirPrex, Stuttgarter processes and Seaborne process [12–17]. The experiences with these technologies show that enhanced sludge leaching is necessary if a high percentage of the phosphorus contained in the sludge should be recovered. Extraction methods mentioned in the literature apply acidic treatment which leads to high costs for the consumption of acids. This paper presents results about an alternative approach for phosphorus extraction from sewage sludge by application of high concentration salt solutions.

Previous studies showed that sodium chloride can have an effect on microorganisms regarding uptake and release of phosphorus by biological cells and on dissolution from biomass into the liquid phase. Most microorganisms are unable to cope with a high salinity environment, they die or become dehydrated and dormant [18]. Especially phosphorus accumulating organisms (PAO) are highly sensitive towards sodium chloride [19].

High salinity has also an impact on the dewaterability of sewage

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sludge. The difference of the osmotic pressure between the free and interstitial water causes a shift of the Donnan equilibrium between flocs and water. This leads to a lower swelling pressure of the flocs [20,21]. The force keeping the "trapped" water in the sludge flocs is decreased and hence the dewaterability is increased. Another effect of high salinity is the presence of a higher percentage of monovalent ions in comparison to multivalent ions. According to the double cation bridging (DCB) theory divalent cations serve as bridges between the extracellular polymeric substances (EPS). The presence of many monovalent cations leads to a replacement of the divalent cations connecting extracellular polymeric substances to each other [22,23]. An increase in the ratio of monovalent and divalent ions (M/D) to levels above two leads to degradation of the sludge structure [22]. The sludge loses its stability since monovalent cations are not able to apply the same electrostatic forces as divalent cations [24,22,23]. As a consequence, phosphorus trapped in the interstitial water is released into the free water during dewatering and subsequently the phosphorus can be recovered from the liquid phase. This will be favorable when for example applying the AirPrex process. During this process air and $MgCl_2$ is added to the sewage sludge in a reactor. This will lead to precipitation of struvite, which can be harvested at the bottom of the reactor [12].

We suggest that this fundamental phenomenon could be a suitable option for phosphorus mobilization in locations with existing sources of brines with high salt concentrations. Streams of high salt concentration could be seawater concentrates or brines from drinking water production and reverse osmosis. Therefore, wastewater treatment plants which are located close to such desalination plants could benefit from this type of extraction.

## 2. Material and methods

### 2.1. Sewage sludge samples

Waste activated sludge was provided by the wastewater treatment plant (WWTP) Birs, Birsfelden, Switzerland, with a current capacity of 150,000 P.E. In this WWTP mechanical pretreatment with screening, grit removal and fine screening is applied. The activated sludge treatment includes a sequencing-batch-reactor (SBR) with nutrient removal [25]. Phosphorus precipitation with $Fe^{III}$ is applied. The waste activated sludge contains 6 g/L of dry matter, the total organic carbon (TOC) value in the liquid phase is 16 mg/L and the sludge volume index (SVI) is 107 mL/g. The total phosphorus concentration in the sludge is 185 mg/L.

### 2.2. Experimental procedure

The samples were transported to the FHNW laboratories in Basel and the experiments were started immediately. During each trial 100 g of sludge was used per sample. The sludge was poured into plastic beakers which were used for centrifugation, later in the experiment. Afterwards, sodium chloride (Carl Roth, Karlsruhe, Germany) was added in solid form and stirring (280 rpm) is started using a magnetic stir bar. A duplex to four-fold repetition was done of the described routine. A scheme of the experimental-set up is shown in Fig. 1.

Two different parameters were varied during the trials. Sodium chloride was added to the sludge in different concentrations from 0 g/L to 25 g/L. Kinetics were observed within 24 h. Additionally, various stirring times ranging from 0 to 24 h were evaluated for 12% sodium chloride concentration.

After each trial the samples were centrifuged using a Beckman Coulter Avanti J-25I centrifuge (Brea, CA, USA) and the compatible JA-14 Rotor also from Beckman Coulter (Brea, CA, USA). The centrifugation was performed at an acceleration of 12,000 × *g* for a duration of 15 min. Afterwards the liquid and solid phases were separated.

### 2.3. Analytics

#### 2.3.1. Liquid samples

The liquid samples were filtered through a hydrophilic membrane with a pore size of 0.45 µm. For this purpose disposable filters Titan3, TF44517-NN (SMI-LabHut Ltd., Churcham, UK) were used. The samples were diluted with 1% $HNO_3$ (Sigma-Aldrich, St.-Louis, MO, USA).

After the dilution, phosphorus and calcium concentration was analyzed using inductively coupled plasma mass spectrometry (ICP-MS). Phosphorus ($^{31}P$) and calcium ($^{44}Ca$) were measured using an Agilent 7500cx ICP-MS (Agilent Technologies AG, Basel, Switzerland) with an argon plasma at 1550 W. A dwell time of 0.3 s was applied per isotope. The gas flows were adjusted to 15 L/min plasma gas, 0.79 L/min carrier gas, 0.35 L/min makeup gas and 5.0 mL/min helium pressurizing gas for the octopole. The quantification of the elements was achieved using phosphorus and calcium standards (Sigma-Aldrich, St.-Louis, MO, USA) diluted in 1% $HNO_3$ containing the background NaCl concentration.

#### 2.3.2. Solid samples

The solid samples were dried for 24 h at a temperature of 105 °C. The drying procedure took place in a vacuum oven (Salvis, Rotkreuz, Switzerland). The weight of the samples was measured before and after drying using the precise weighing scale XS 205 DualRange (Mettler-Toledo GmbH (Giessen, Germany)) to identify the dry weight of the sludge.

After drying, the samples were dissolved using aqua regia consisting of 1 mL of $HNO_3$ and 3 mL of HCl, purchased from Sigma-Aldrich (St.-Louis, MO, USA), and microwave extraction (Milestone S.r.L., Sorisole, Italy). Next the dissolved sludge samples were filtered and diluted in 0.5 molar $HNO_3$.

The filtered samples were analyzed by inductively coupled plasma atomic emission spectroscopy (ICP-OES) using a Spectroflame modula (Spectro Analytical Instruments GmbH, Kleve, Germany). Phosphorus was measured using a wavelength of 178 nm. The argon plasma was operated at 1200 W, 14 L/min plasma gas and 0.5 L/min auxiliary gas flow. The samples were diluted with 1% nitric acid and the quantification was achieved using phosphorus standards (Sigma-Aldrich, St.-Louis, MO, USA).

## 3. Results and discussion

### 3.1. Kinetics

As a first step to investigate the influence of high salt concentration on phosphorus dissolution an experiment was performed with constant sodium chloride concentration (12 wt.-%). The phosphorus concentration in the supernatant was measured as a function of time (Fig. 2). 12% sodium chloride was chosen as concentration for this experiment based on preliminary experiments showing a significant effect at this dose.

In the first 10 h of stirring the phosphorus concentration in the supernatant increased nearly linearly until 32.5 mg/L. Between 10 and 15 h the concentration continued to rise linearly up to 37.5 mg/L but with a lower slope. After 15 h no significant further increase of the phosphorus concentration could be observed. The maximum phosphorus concentration reached in one sample was 58.3 mg/L phosphorus in the supernatant after 22 h of stirring. The maximum value after 24 h of stirring was 57.5 mg/L phosphorus. The mean values were found to be in a range from 37.5 mg/L to 46.8 mg/L phosphorus. Van Veen et al. [26] observed the influence of an osmotic shock on *Acinetobacter johnsonii* cells two minutes after the shock was applied. They showed that the ability of phosphorus uptake was immediately lower after an osmotic shock. Neu and Chou [27] analyzed *Escherichia coli* cells in a time range from 0 to 30 min. The release of proteins and nucleotides occurred in the first 5 min after an osmotic shock, thereafter an equilibrium was reached. Sobeck and Higgings [22] looked at the influence