



P-recovery as calcium phosphate from wastewater using an integrated selectrodialysis/crystallization process



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ABSTRACT

A promising and sustainable renewable source of phosphate was obtained through crystallization of calcium phosphate from wastewater. The aim of the present study was to evaluate the feasibility of an integrated selectrodialysis/crystallization process to recover phosphate, in which selectrodialysis was first used to pre-concentrate the phosphate before crystallization in a pellet reactor. The results of selectrodialysis show that an increase in current density, initial pH of the product, and initial feed concentration of phosphate led to an increase in phosphate concentration in the product. The desalination efficiency from wastewater was 87%, the phosphate concentration in the product can reach 16 mM with a purity of 44%, the current efficiency to transport phosphate and chloride was 26.6% for standard anion exchange membrane and 63% for monovalent selective anion exchange membrane. The optimal operational conditions for phosphate recovery with a pellet reactor were examined by changing the initial concentration, the pH and the superficial velocity. A precipitation efficiency of 82.7% of phosphate was achieved at pH 11, an inlet concentration of phosphates of 2.5 mM, a Ca/P molar ratio of 1.5 and a superficial velocity of 61 m/h. From the results, it can be concluded that the pellet reactor combined with selectrodialysis is an appropriate method to recover phosphate from wastewater as calcium phosphate, which is not only technically but also environmentally feasible, reducing water pollution, preventing eutrophication and yielding valuable and sustainable resources.

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1. Introduction

Phosphate scarcity is a global challenge in the 21st century. The use of a large amount of fertilizers in developing countries and the demand of crops for bio-fuels are some of the reasons for the increasing phosphate demand. Therefore, phosphorus recovery from wastewater has become an important issue to control eutrophication and to solve the problem of phosphate shortage in

natural resources. Some potential techniques are introduced to use the natural resources in a sustainable way such as eco-industrial park, reuse/recovery from waste streams, utilizing more waste as fertilizer (Jegatheesan et al., 2009). Moreover, a good policy is needed, not only for sustainable but also for effective use of phosphorus (Cordell et al., 2011).

The most developed technique that can be applied to recover phosphate is precipitation/crystallization as a magnesium or calcium salt. The products of this recovery process are often struvite (Korchef et al., 2011; Kumar and Pal, 2013) or calcium phosphate in different phases (hydroxyapatite (HAP, $\text{Ca}_5(\text{PO}_4)_3\text{OH}$), brushite (DCPD, $\text{CaHPO}_4 \cdot 2\text{H}_2\text{O}$), and amorphous calcium phosphate (amorphous $\text{Ca}_9(\text{PO}_4)_6 \cdot x\text{H}_2\text{O}$, ACP) under different conditions such as pH, temperature, supersaturation level, Ca/P molar ratio (Lee et al., 2009; Mekmene et al., 2009). Korchef et al., 2011 used a

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degasification technique to recover phosphorus in the form of struvite from wastewater to reduce water pollution. Moreover, Kumar and Pal, 2013 also studied the production of struvite from ammoniacal waste and then used a membrane-integrated continuous approach to get 95% struvite from the stream. Besides struvite, Lee et al., 2009 used activated oyster shell – a waste product of mariculture – as a sustainable precipitant for phosphate precipitation as hydroxyapatite. However, one of the drawbacks that make phosphate recovery difficult is the low concentration of phosphate in the wastewater. The lower the initial concentration, the lower the supersaturation index is. In the Netherlands, a crystallization reactor installed for phosphorus recovery in 1999 was stopped because of a shift to the use of phosphate-free detergents which leads to a too low concentration of phosphate in the waste stream (Giesen, 1999). The initial phosphate concentration is indeed a very important parameter in controlling the crystallization process (Karapinar et al., 2004).

Many different processes were proposed for pre-concentration of phosphate, such as adsorption (Wahab et al., 2011), ion exchange (Kumar et al., 2007) and biological treatment (Kodera et al., 2013). *Posidonia oceanica* fibers were studied as a low-cost material with a high adsorption efficiency compared to common natural materials with a maximum adsorption capacity of 7.45 mg/g (Wahab et al., 2011). The potential of phosphate recovery from RO concentrates via polymeric ligand exchange resins was also investigated. More than 85% of the adsorbed phosphate was extracted from the regeneration process and precipitated as struvite (Kumar et al., 2007). However, the thermal procedure needed for desorption of phosphate from adsorbents and chemicals used for regeneration of phosphate from ion exchange resins led to an increase of the operational cost (Wahab et al., 2011). Furthermore, to concentrate the phosphate in wastewater, Kodera et al. used poly-phosphate accumulating organisms to increase the concentration of phosphate from 5 to 100 mg P/L. However, the anaerobic condition to achieve the release of ortho-phosphate is not easy to control (Kodera et al., 2013). Recently, membranes were studied to selectively pre-concentrate the multivalent ions (phosphate, sulfate) from water in the presence of chloride. Nylon microfiltration membranes with different bilayers of PEI/PSS were used to separate phosphate from a chloride solution with high selectivity and a high rejection of phosphate up to 98% at low pressure (Disha et al., 2012). A novel configured electro dialysis, denoted as “selectrodialysis”, can be used for fractionating the sulfate ions from chloride ions by standard anion exchange membranes and monovalent selective anion exchange membranes with a high product purity of 85% and a current efficiency of over 50% (Zhang et al., 2012). Furthermore, a preliminary study was reported on applying selectrodialysis to pre-concentrate phosphate from an anaerobic effluent to feed a struvite reactor (Zhang et al., 2013).

This study aims at the fractionation and concentration of phosphate from synthetic water using selectrodialysis to increase the concentration as a pretreatment prior to crystallization in a pellet reactor. This allows to optimize the operational conditions of a pellet reactor to obtain the highest efficiency of phosphate recovery.

2. Materials and methods

2.1. Solutions

Experimental solutions were prepared from the following chemicals: KH_2PO_4 (UPC, Belgium), $\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$ (Chem-Lab, Belgium), and NaCl (BDH Prolabo, Belgium) dissolved in de-ionized water. The pH was adjusted to an initial value by using a 6 M NaOH solution (Fisher Scientific, UK).

2.2. Selectrodialysis

2.2.1. Experimental setup

A lab-scale, three-compartment ion exchange membrane electro dialysis system (selectrodialysis) was used to perform the experiments. The ED stack (ED-64-004) was provided by PCell GmbH, Germany. The configuration of selectrodialysis was described elsewhere (Zhang et al., 2012). Three pieces of anion exchange membranes (PC-SA), three pieces of monovalent selective anion exchange membrane (PC-MVA) and four pieces of cation exchange membranes (PC-SK) were used in the stack, each with an effective surface area of 64 cm^2 , dividing the stack into four types of compartments: electrode rinsing, feed, product, and brine compartment (Fig. 1). Due to the applied electrical field, the anions (Cl^- and $\text{H}_x\text{PO}_4^{x-}$) move across PC-SA while the cations (Na^+ and K^+) move across PC-SK. Since PC-MVA membranes were set between PC-SA and PC-SK, the multivalent ions $\text{H}_x\text{PO}_4^{x-}$ are kept in the product compartment (Fig. 1). The equipment consisted of four separated circuits for the feed, the product, the brine and the electrode rinsing solutions, each 3 L in volume, except for the feed compartment that contained 10 L and were recirculated by a centrifugal pump. A flow rate of 20 L/h was used for the feed, product, brine streams and of 100 L/h for the electrode rinsing stream. The membrane stack was connected to a DC power source (Delta Elektronika Power Supply ES 030–5). The electrode rinsing solution was a 0.1 M Na_2SO_4 solution for all experiments.

The characteristics of the three kinds of membranes manufactured by PCell GmbH, Heusweiler, Germany are given in Table 1.

Only KH_2PO_4 and NaCl were present in the feed stream in the experiments. When increasing the initial concentration of phosphate from 3 to 5 and further to 7 mM, the initial concentration of chloride in the feed solution decreased accordingly from 17 to 15 and finally to 13 mM. For the product and brine stream, 20 mM NaCl was used as the initial solution to control the conductivity. Different currents (I) (0.2, 0.3 and 0.4 A) corresponding to a current density of 31.3, 46.3 and $62.5 \text{ A} \cdot \text{m}^{-2}$, applied pH in the product compartment (8, 10, 12), and initial phosphate feed concentrations (3, 5, 7 mM) were used in the experiments to determine the effect of current density, pH and concentration on the selectrodialysis performance.

The current and also the applied pH in the product stream which was adjusted by adding NaOH 6 M were kept constant during 6 h continuous operation in all of the experiments. In some cases where the voltage increased to the maximum value of 12 V due to

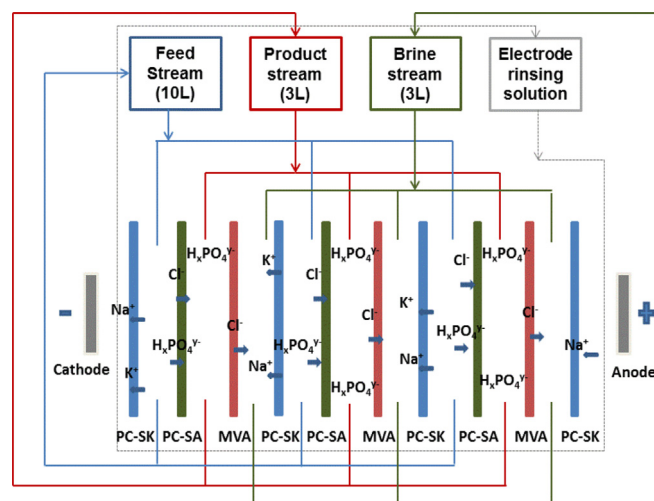


Fig. 1. Cell configuration of selectrodialysis with monovalent selective anion exchange membrane.

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