



## Effective recovery of struvite from wastewater by electrochemical process

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### ABSTRACT

Depleting natural resources of phosphorus and the need to increase food production have made it imperative to look for alternative resources of nutrients, particularly phosphorus. Since natural chemical elements are conserved in this universe, most of the nutrients after their stay through various living bodies reach the water bodies as waste in significant quantities leading to environmental degradation through eutrophication. Many studies have reported the recovery of nutrients, especially nitrogen and phosphorus, in the form of struvite, chemically known as magnesium ammonium phosphate, based on chemical, electrochemical, and biological methods. Electrochemical methods appear attractive, eco-friendly, without external pH adjustment and suitable for all environments and capacities. Studies were carried out with in-house built electrochemical cells using the sewage wastewater collected after the secondary treatment from the SRMIST wastewater treatment plant for the recovery of the struvite. The investigation aimed to reduce energy consumption by improving the conductance of wastewater through the addition of sodium chloride, which is cheap and abundantly available. The results indicate a 33.5% reduction in energy consumption under optimal conditions, with the addition of NaCl to wastewater leading to a recovery of 84.5% of phosphate and 50% of ammonium species.

*Keywords:* Nutrient recovery; Sewage wastewater; Struvite; Electrochemical reactor; Energy consumption

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### 1. Introduction

Phosphorous, a primary nutrient that many NPK fertilizers contain, is generally sourced from phosphate rocks, whose reserves are depleting faster [1]. In the long run, it may lead to the inadequacy of phosphorus nutrients for the agricultural sector, resulting in reduced food production [2]. The demand for food is rising rapidly in the world. According to the UN FAO [3], the need for food will

increase by 70% in 2050 compared to current consumption levels [4]. Consequently, the need for phosphorus would increase, endangering the farming sector leading to a crisis with inadequate availability of phosphorus nutrients for the crops [5].

In nature, each chemical element is conserved even if their presence may differ in the chemical forms or in the environment in which they are available. Whatever the nutrients used as fertilizer, they return to the environment

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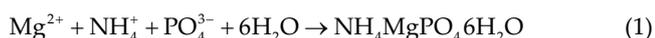
through a chain of transformations, for example, food-culture (living beings) – excreted – environment. Only a tiny portion of what is consumed is retained by the user, even temporarily for a short period. This natural cycle, valid for every element, be it phosphorus, nitrogen or carbon, offers us the opportunity to seek alternative sources of phosphorus to bridge the gap between supply and demand.

If the food is consumed or wasted, some of the nutrients end up in sewage and many other contaminants, including harmful microorganisms like e-coli, toxic and several other chemicals, and threaten human health. At the same time, sewage is an enormous source of water and nutrients such as phosphorus and nitrogen [6]. One of the primary reasons for the eutrophication of the water bodies is the discharge of sewage into water bodies [7], leading to the unwarranted growth of algae [8]. As the demand for water increases and the availability of natural phosphorus decreases, it is only natural that we must recover the valuable products [9]. Water recovery and recycling have become an integral part of any sewage treatment plant [10,11]. No serious considerations have been given for the recovery of phosphorus (P) and nitrogen (N), which have high value as fertilizers [12].

The existing conventional methods, such as chemical/biological processes, have many economic and logistic viability challenges, mainly due to seasonal fluctuations of effluent volumes and accessibility [13]. Recovering the N and P as magnesium ammonium phosphate (struvite) from sewage wastewater is an alternative fertilizer source and is considered a sustainable solution based on recent studies [14].

Struvite is a compound of magnesium ammonium phosphate prepared with a source of magnesium at a controlled pH, as indicated in Eq. (1) [15].

The chemical formula for struvite formation is written as:



The most popular method of producing struvite from wastewater is chemical precipitation by dosing magnesium salts while adjusting the pH with a base [16] or picking  $\text{CO}_2$  by aeration [17]. The most common sources of Mg used in struvite studies include salts of Mg, viz.  $\text{MgCl}_2$ ,  $\text{MgSO}_4$ , and  $\text{MgO}$  [18–20].

Studies on commercial production of struvite from waste streams indicate the use of crystallization reactor, continuous cone flow reactor, fluidized bed reactor, etc. [21] M/s Pearl technology [22] (North America, UK) has established a few commercial plants using a fluidized bed reactor christened as ‘CRYSTAL GREEN’ in Canada, England, Japan, Germany, Australia, Italy, Netherlands, etc. [23]. Even though the process is quite efficient in recovering phosphorus, it has a few challenges in crystal size distribution, high energy consumption, and cost [24].

The comparative studies in the literature [25] indicate several advantages of an electrochemical process, particularly about pH adjustment, simplicity of equipment, and eco-friendliness over the other chemical and physico-chemical processes. Besides, it provides an opportunity for energy generation in the form of hydrogen gas. The

electrochemical recovery method has various advantages over the other methods; it is environmentally compatible as it utilizes electrical energy and no other chemicals [26].

Generally, an electrolysis cell is used as a sacrificial anode in this approach, in which magnesium or magnesium alloy is used. During the dissolution of the anode,  $\text{Mg}^{2+}$  cations are released, providing struvite with the necessary amount of Mg [27]. Electrochemical precipitation of struvite, driven by excessive potential, requires more energy than should be needed by the electrochemical cell to cause the thermodynamic reaction [28]. Consequently, some energy is required to drive struvite formation over time [29].

In their first electrochemical precipitation of struvite from source-separated urine, Hug and Udert [30] used a magnesium alloy plate as an anode with an energy consumption of 1.6 W/g of struvite. Zhou and Chen reported energy consumption of 2.8 to 30 W h/g with increased applied current from 45 to 125 mA by changing the electrochemical parameters [31]. A recent study by Liu et al. investigated the factors affecting phosphorus removal of phosphorus by struvite utilizing magnesium as an anode. Other electrode materials used in related works include Pt–Ti, Ti, Al, Fe, and Al–Mg [32].

To fabricate the electrodes in this study, we used carbon and stainless steel because they have been extensively studied at the lab- and pilot-scales and are known for their electrical conductivity [33]. Also, the processing techniques to fabricate electrodes are readily available, making it possible to configure the electrode configuration [34]. Salt bitterns collected from the salt lake was used as the magnesium source in the experimentation. The electrochemical needs energy input but a power management system (PMS) and modified electrodes can bring costs down concurrently with expanding research in this area. In the current study, a small-scale electrochemical reactor was set up to demonstrate the potential recovery of struvite from domestic sewage near-neutral pH with cost-effective electrodes.

The ionic conductivity of the solution is a primary attribute in electrolysis. A solution’s ionic conductivity determines the current efficiency, applied electric voltage, and electricity consumption in electric systems, as the current flowing through a circuit varies with its conductivity under a specific electric voltage [35]. The addition of NaCl increases the electrical conductivity. NaCl can be used as a supporting electrolyte because it increases the electrical conductivity of the solution and thus reduces energy consumption. Due to the simultaneous increase in conductivity and applied current density, the continuous bubbling of hydrogen in the electrode increases porosity. This helps in the detachment of the struvite formed on the electrode and improves maximum recovery.

Microbial fuel cells (MFCs) can be a promising technology for treating waste whilst producing electricity without the need to add chemicals like NaOH, which may amount to about 97% of total chemical costs [37,38]. The struvite production and power generation using microbial fuel cell with varying operating parameters were shown in Table 1. Air-cathode MFC (microbial fuel cell) studies indicated that struvite precipitated on the cathode surface with increasing concentrations of  $\text{NH}_4$  and Mg [37] and produced

Table 1  
Struvite production and power generation and from various sources of wastewater using microbial fuel cell

Source	Output	Reactor	Operating conditions	Observation	References
Raw human urine	Power generation, contaminant removal, and nutrient recovery	Single-chamber microbial fuel cells	<ul style="list-style-type: none"> <li>- Cathode: Pt-based Pt-free carbon cloth with a carbon-based microporous layer</li> <li>- Anode: Carbon brush with a titanium wire core</li> <li>- Feeding cycles: 1, 2 and 4-day cycle</li> <li>- Constant temperature: <math>30^{\circ}\text{C} \pm 2^{\circ}\text{C}</math></li> </ul>	<ul style="list-style-type: none"> <li>- COD degradation of 60%–75% in 4-days cycle</li> <li>- An increase in ammonium ions and a decrease in sulfate was observed</li> <li>- Struvite, potassium struvite and hydroxyapatite are formed</li> </ul>	[36]
Synthetic wastewater (substrate – sodium acetate; buffer – phosphate buffer and trace minerals)	Struvite	Air-cathode single-chamber microbial fuel cell	<ul style="list-style-type: none"> <li>- Cathode: Pt/C catalyst on wet-proofed porous carbon paper</li> <li>- Anode: Carbon felt discs</li> </ul>	<ul style="list-style-type: none"> <li>- Addition of only <math>\text{NH}_4^+</math>: No precipitate</li> <li>- Addition of only <math>\text{Mg}^{2+}</math>: formation of cattite</li> <li>- Addition of both <math>\text{NH}_4^+</math> and <math>\text{Mg}^{2+}</math>: formation of struvite</li> </ul>	[37]
Swine wastewater	Power generation, struvite	Air–single cathode chamber microbial fuel cells	<ul style="list-style-type: none"> <li>- Cathode: Pt/C catalyst on wet-proofed porous carbon paper</li> <li>- Anode: Carbon felt discs</li> </ul>	<ul style="list-style-type: none"> <li>- Maximum power density: <math>1\text{--}2.3\text{ W/m}^2</math></li> <li>- Maximum current density: <math>6.0\text{--}7.0\text{ A/m}^2</math>; COD removal efficiency: 76%–91%</li> <li>- Coulombic efficiency: 37%–47%</li> <li>- Phosphate recovery: 70%–82%</li> </ul>	[38]
Source – separated urine	Power generation and struvite	Single-chamber microbial fuel cell	<ul style="list-style-type: none"> <li>- Cathode: Hot-pressed activated carbon onto carbon fiber veil</li> <li>- Anode: Plain carbon fibre veil</li> <li>- Membrane: Cation exchange membrane</li> </ul>	<ul style="list-style-type: none"> <li>- Phosphate removal: 82%</li> <li>- COD removal: 20%</li> <li>- Absolute power: <math>14.32\text{ W/m}^3</math> (First stage)</li> <li>- Absolute power: <math>11.76\text{ W/m}^3</math> (Third stage)</li> </ul>	[39]
Digested sewage sludge	Power generation and struvite	Single-chamber microbial fuel cell	<ul style="list-style-type: none"> <li>- Cathode: Reticulated vitreous carbon</li> <li>- Anode: Six carbon felt</li> </ul>	<ul style="list-style-type: none"> <li>- Yield: 80% or 600 mg/L</li> <li>- Iron phosphate was reduced through electrons and protons obtained by the metabolic pathway of the bacteria</li> </ul>	[40]
Real urine	Power generation and struvite	Single-chamber microbial fuel cell	<ul style="list-style-type: none"> <li>- Cathode: Carbon</li> <li>- Anode: Carbon veil folded and wrapped around the ceramic cylinder</li> </ul>	<ul style="list-style-type: none"> <li>- The addition of sea mix increased the maximum power by 10% and achieved 94% of the phosphate recovery</li> <li>- Increase in pH, conductivity and concentration of chloride ions</li> </ul>	[41]
Synthetic wastewater	Power generation and struvite	Double-chamber microbial fuel cell	<ul style="list-style-type: none"> <li>- Cathode: Carbon fibre brush coated with a titanium bar</li> <li>- Anode: Cylinder-shaped graphite felt</li> <li>- Membrane: Cation exchange membrane</li> </ul>	<ul style="list-style-type: none"> <li>- <math>\text{NH}_4^+\text{-N}</math></li> <li>- Removal &gt; 97.58%</li> <li>- <math>\text{PO}_4^{3-}</math>: P removal: 94.9%</li> <li>- COD removal efficiency: more than 85%</li> </ul>	[42]

70%–82% of phosphorous from the influent [36,38,40]. These results show that MFC coupled with electrolyzer function can greatly benefit struvite precipitation from waste biomass and treat wastewater simultaneously.

MFCs and struvite precipitation could be integrated for energy generation and product recovery from urine, rendering waste sustainability to energy and resource recovery systems. Ben Moussa et al. [28], the electrochemically induced precipitation process was influenced by a strong pH increase near the cathode surface due to the electrochemical reduction of oxygen and/or water molecules. The purity of the struvite yield slightly differed with the influents used; electrochemical precipitation [41] yielded highly pure crystals, while swine wastewater [38] and digested sewage sludge [40] produced 90%.

In the current study, an electrochemical method for the production of struvite was adopted to assess the parameters for reducing energy consumption and evaluating the quality. Therefore, it is necessary to ensure that the wastewater stream has sufficient electrical conductivity to efficiently utilize energy, leading to the cost reduction of struvite produced. Hence, the experimental studies optimize salt (NaCl) concentration and energy consumption by utilizing the wastewater streams available in the SRMIST campus.

## 2. Materials and methods

### 2.1. Sewage wastewater and chemicals

The wastewater utilized in this study was collected from the SRMIST sewage treatment plant. The wastewater effluent from the secondary treatment was collected and stored in a 20 L storage tank for 24 h at 4°C to settle the particulate matter and minimize ammonia loss due to volatilization. Table 2 shows the physicochemical characteristics of the wastewater used in this experiment. The AR grade chemicals sodium chloride, ammonium chloride, Nessler's reagent, sulphuric acid, potassium antimony tartrate solution, ammonium molybdate solution, ascorbic acid, sodium dihydrogen phosphate used for the standardization methods for the determination of ammonium ions (Nessler's method) and the phosphate ions (ammonium molybdate method) are purchased from Merck Life Science,

Table 2  
Physiochemical characterization of SRMIST sewage wastewater

Parameters	Values
Chemical oxygen demand (COD)	560 mg/L
Biochemical oxygen demand (BOD)	272 mg/L
Total nitrogen (TN)	158.5 mg/L
Total phosphorous (TP)	36.5 mg/L
Total suspended solid (TSS)	104 mg/L
Potassium	48 mg/L
pH	6.8
Turbidity	0.45 NTU
Conductivity	13.4 mS
Salinity	7.53 ppt
Total dissolved solids (TDS)	928 mg/L

India. Purified water with a total dissolved solids content of less than 4 ppm from the RO purifier was used to prepare sodium chloride solutions of various concentrations.

### 2.2. Electro-chemical cell components and configuration

A single-chamber electrolytic cell made of polyethylene terephthalate (PET) with a working volume of 2 L was used for the experimental studies. The electrolysis reactor's schematic representation and the electrochemical setup photograph with all accessories used in this study for struvite recovery are shown in Figs. 1 and 2, respectively. The stored and filtered effluent from the secondary treatment of the SRMIST sewage treatment plant was used as the study's feedstock. The carbon rod of 16 mm diameter and 150 mm length was used as the anode, and the stainless steel mesh (150 cm<sup>2</sup> cross-sectional area; woven wire cloth fabric made of stainless steel; 100 mm × 150 mm mesh; 0.048 mm wire diameter) was used as the cathode for the production of struvite.

The three electrodes, anode, cathode, and the reference electrode (Ag/AgCl), were installed into the experimental electrolytic cell and connected externally to the pH meter, gas collector, and aliquot dispenser, allowing samples to be taken periodically without disturbing the process. The 60W DC power unit was used to supply power to the electrochemical cell with a function to set operations to desired power levels and voltages. Experiments were conducted at three power settings: 5 W (5 V, 1 Å), 12 W (10 V, 1.2 Å), and 22.5 W (15 V, 1.5 Å). According to the selected experimental conditions, sodium chloride was added to the electrochemical cell in the appropriate amounts.

The phosphate percentage recovery was calculated using the following formula:

$$\text{Percentage recovery (\%)} = \frac{C_i - C_o}{C_i} \times 100 \quad (2)$$

where  $C_i$  is the initial concentration;  $C_o$  is the final concentration.

The parameters (Mg:P ratio, ammonium concentration, pH) optimized by batch experiments and previously reported [43] were used for the present study. As the amount of Mg present in wastewater was less than the stoichiometric levels, MgO was added to the feedstock to facilitate the formation of struvite. Experimental runs were carried out by changing the concentration of NaCl in steps.

The NaCl concentration was varied gradually (0.02%, 0.04%, 0.06%, 0.08% and 0.1%) together with the increase in the power supply (5 W, 12 W and 22.5 W). A series of experiments were performed for each NaCl concentration, varying the power and estimating the percentage of phosphate recovery for each trial. No external reagents were added to maintain the pH of the solution, as was achieved during the electrochemical process in which hydroxide ions are generated with hydrogen gas [44].

The progress of the reaction was analyzed by taking the aliquot sample and acidified instantly with a dilute solution of hydrochloric acid (0.1 M) to stop the reaction in the samples, and the remaining phosphate concentration was determined. The final experiment was performed with the

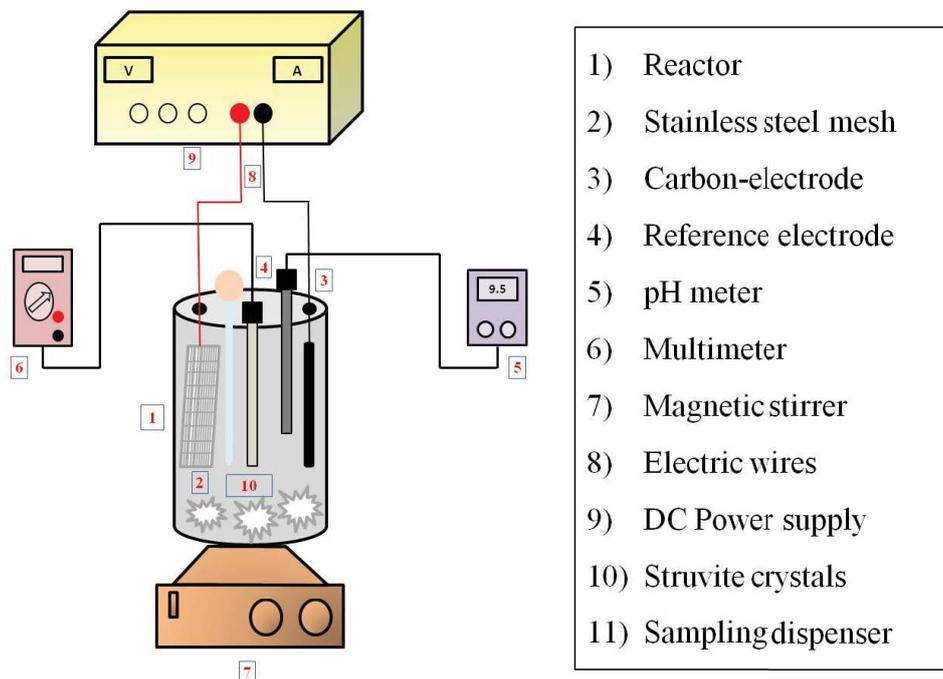


Fig. 1. Schematic representation of the electrolysis reactor.



Fig. 2. Photograph of the electrochemical setup.

optimized power supply and NaCl concentration, and the recovered struvite was analyzed for purity and elemental analysis using X-ray diffraction (XRD) and scanning electron microscopy (SEM)-energy-dispersive X-ray spectroscopy (EDX).

The potential difference of the experimental trials was estimated after measuring the anode and cathode potentials with the reference electrode (Ag/AgCl) with a digital multimeter and recorded at regular time intervals (10 min).

### 2.3. Analytical techniques

SEM and X-ray crystallography (XRD) are the methods used to analyze struvite precipitate [45]. The precipitated struvite crystals are analyzed with the SEM to check the topography and morphology of the crystal formed. The XRD is used to analyze the structure of the struvite crystals deposited on the cathode [46].

## 3. Results and discussion

### 3.1. Description of the process

Water is split into hydrogen and hydroxide ions during the electrochemical reaction. Hydrogen ( $H_2$ ) gas is released after reduction at the cathode, resulting in the deposition of struvite at the cathode. The main advantage of this method is that it does not need additional chemicals for pH adjustments making it economical and feasible [47,48]. It is environmentally compatible as it uses electrons, a fundamental and clean reagent [49]. The additional magnesium requirement to meet struvite stoichiometry was provided in the form of MgO.

### 3.2. Effect of NaCl concentration on phosphate recovery

An experimental test was conducted with the filtered wastewater with an applied voltage of 10 V and a nominal

current of 1.2 A corresponding to a power input of 12 W. The percent recovery vs. time indicates more than 68% after 1 h as shown in Fig. 3. However, the product quality was found to be poor with many impurities. Although the pH value measured was beneficial to the precipitation, other contaminants were also found.

Since the wastewater contains very few dissolved salts, its natural conductance is insufficient to enable the electrochemical reaction to occur efficiently. Thus, a suitable electrolyte is added to improve the conductivity of the solution and increase the process's efficiency. NaCl is one of the best-reported electrolytes that has been used over other chemicals like  $\text{NaNO}_3$ ,  $\text{Na}_2\text{SO}_4$  and  $\text{CaCl}_2$  as it enhances the reaction by increasing the electrical conductivity of the solution and thereby reducing energy consumption [50,51]. Various studies indicate that the oxide film formed on the surface of the electrode can be removed with chloride ions [52].

Accordingly, NaCl was added to the wastewater solution in small increments to maintain the concentration levels from 0.02% to 0.1% in the experimental volume for each run. The samples were taken regularly at scheduled intervals (30 min), and the pattern of phosphate precipitation was determined at different NaCl concentrations. The phosphate recovery was found to increase with the NaCl concentration (Fig. 4A–C). At 0.02% and 0.04%, the phosphate

recovery rate was comparatively lower than the other concentrations. However, the percent phosphate recovery increases significantly with 0.06%, 0.08%, and 0.1% of the NaCl concentration, but no significant phosphate recovery changes were observed at these concentrations.

The findings suggest that NaCl concentration in the wastewater improves the energy efficiency and the recovery of the phosphate as struvite. Therefore, it can be

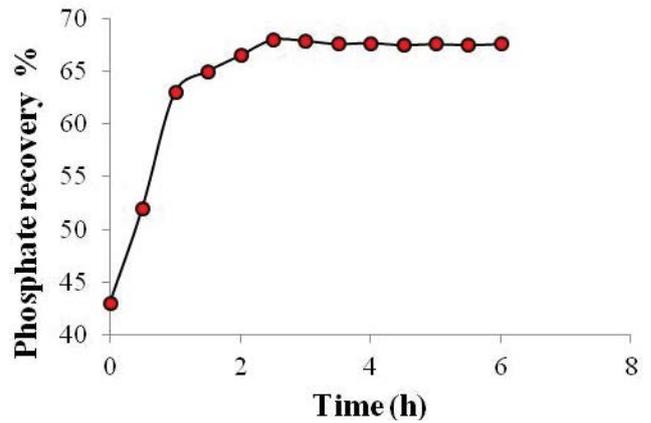


Fig. 3. Impact on phosphate recovery without NaCl addition.

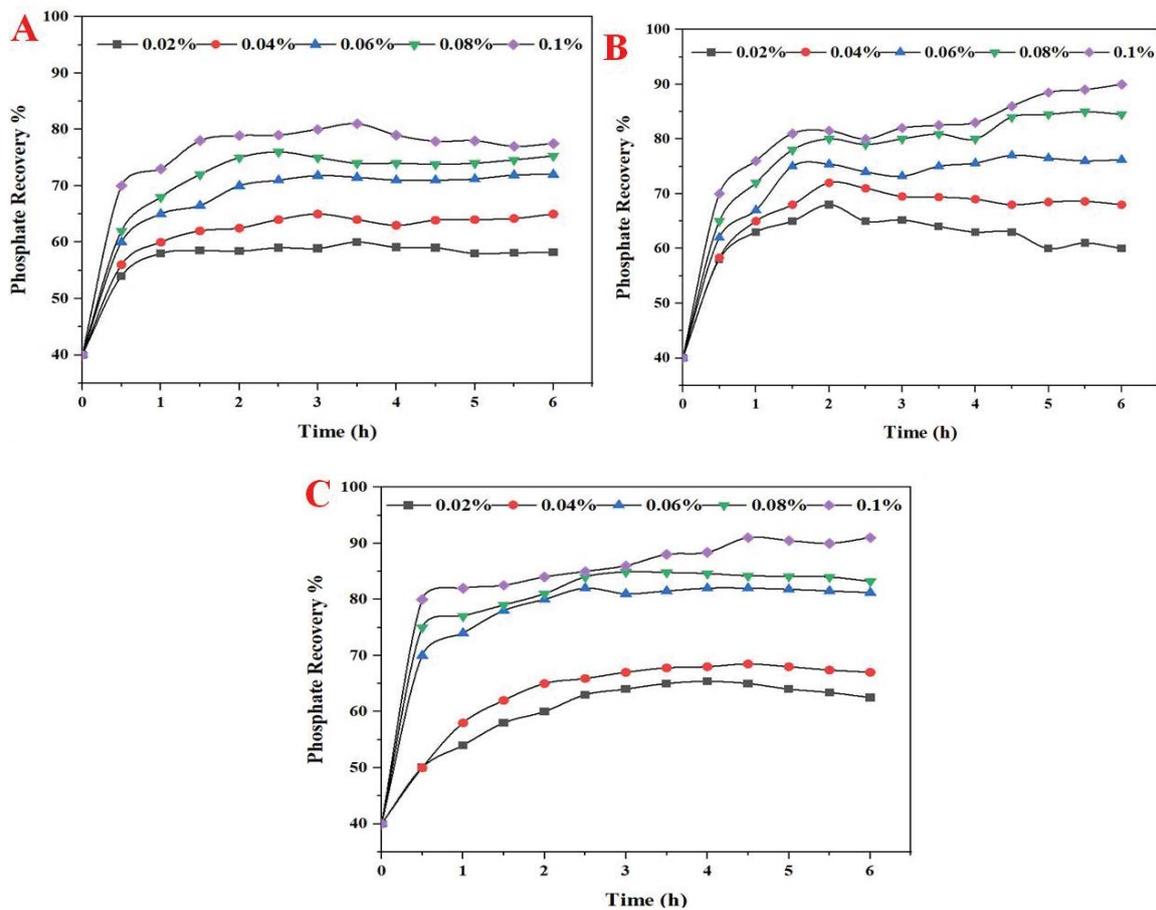


Fig. 4. Effect of power and NaCl concentration on phosphate recovery (A-5 W, B-12 W, C-22.5 W).

concluded from this study that 0.1% NaCl concentration would be optimal for the maximum recovery of  $\text{PO}_4^{3-}\text{-P}$ . Studies have shown that struvite solubility varies with ionic solution strengths [53], and adding NaCl to wastewater can help improve electrical efficiency [54].

Sodium chloride enhances phosphorus recovery regardless of the operating power load, and the percent recovery increases as sodium chloride concentration increases. For lower quantities of sodium chloride, maximal recovery takes around 1 h. The highest phosphate recovery is achieved when the voltage is set to 10 V (12 W). Phosphate recovery at 10 V (12 W) and 15 V (22.5 W) is similar, suggesting more recovery can be achieved with less power.

### 3.3. Effect of NaCl concentration on ammonium recovery

An increase in ammonium concentration of the solution was observed with increasing NaCl concentration (0.02%–0.1%). The samples were withdrawn at regular intervals (30 min) from the reactor to determine the ammonium recovery %. The effect of power and NaCl concentration over time on ammonium recovery is shown in Fig. 5A–C. The experimental results found that the ammonium concentration did not change significantly at 0.02% NaCl concentration. The ammonium recovery rate was slightly higher

at 0.04% and 0.06% NaCl concentrations when the power consumption factor was not considered. There was a gradual drop in the recovery at 0.08% NaCl and a sharp drop at 0.1% NaCl concentration.

Studies have shown that most of the  $\text{NH}_4\text{-N}$  are converted into nitrogen gas in the presence of chloride [55]. The chloride in electrolytic solutions plays a catalytic role in degrading  $\text{NH}_4\text{-N}$ , but it does not react directly [56]. Furthermore, experiments have shown that a suitable quantity of chloride can effectively remove  $\text{NH}_4\text{-N}$  [57]. The study also showed that as the initial chloride concentration increased, the depletion of  $\text{NH}_4\text{-N}$  increased. The reduction of  $\text{NH}_4\text{-N}$  in this research could be attributable to the electrolytic conversion of dissolved  $\text{NH}_4\text{-N}$  to nitrogen gas [58,59]. The experimental results showed that the maximum percentage of ammonium ions could be recovered in the first hour. Further experiments result in ammonia loss, especially when the sodium chloride concentrations are lower.

### 3.4. Effect of the electrical voltage on phosphate recovery

In the previous study, it was found that the optimal NaCl concentration was 0.1%, and this concentration is further tested with three different power supplies (5 W, 12 W, and 22.5 W) to find the most optimum power supply necessary

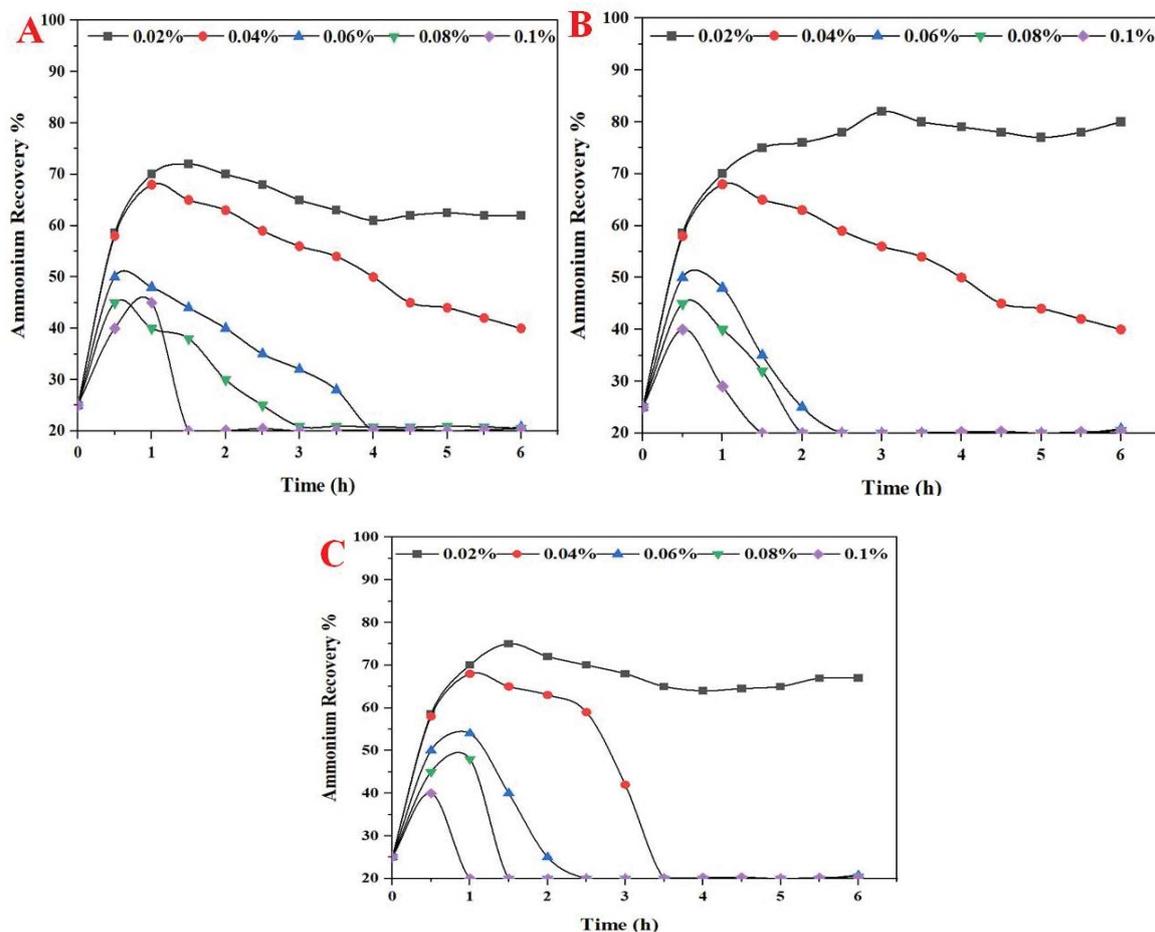


Fig. 5. Effect of power and NaCl concentration on ammonium recovery (A-5 W, B-12 W, C-22.5 W).

for the recovery of phosphate and ammonium (Figs. 4 and 5). The experimental results were observed that the phosphate precipitation at 5 W was relatively lower than 12 W and 22.5 W (Fig. 6). However, the phosphate concentration increased with the increase in the power supply, but there was no drastic difference in phosphate recovery between 12 W and 22.5 W. Considering the energy consumption, the most optimum electrical voltage for phosphate precipitation is 12 W rather than 22.5 W.

### 3.5. Effect of the electrical voltage on ammonium recovery

The experiment was carried out for 6 h, and the samples were withdrawn at regular intervals (30 min) to determine the ammonium concentration in the solution. The applied energy (5 W, 12 W, and 22.5 W) influences the concentration of ammonium in the reactor (Fig. 7). Maximum recovery of ammonia was observed after the 30 mins of the reaction for all the applied power. The current density is one of

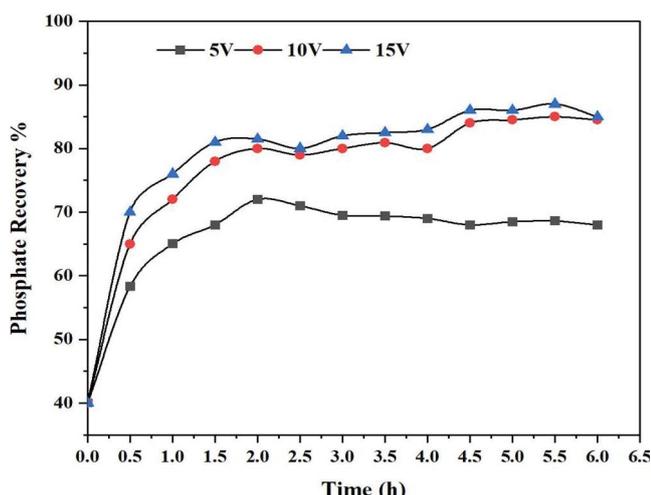


Fig. 6. Effect of voltage on phosphate recovery at 0.1% NaCl.

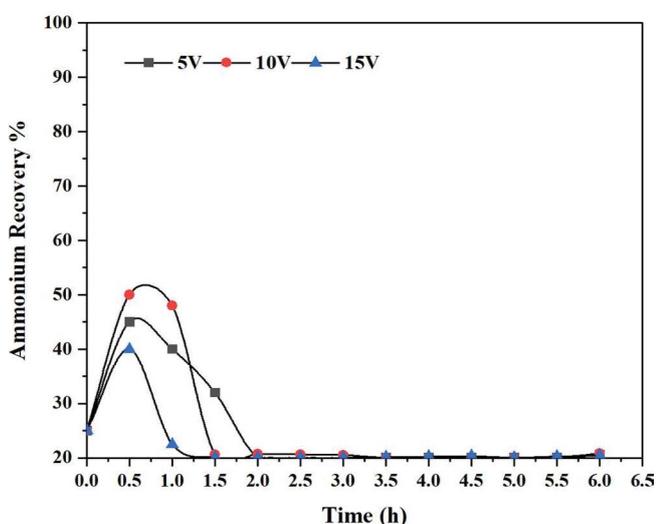


Fig. 7. Effect of voltage on ammonium recovery at 0.1% NaCl.

the most critical factors in the electrochemical process, and the power supply increases with the current density. It was found that the maximum ammonium recovery occurred with the minimum energy consumption, and the optimal power for the complete recovery of ammonium was 12 W.

According to studies, the higher electric voltage increased the rate of Cl electron loss in anodes, resulting in a faster  $\text{NH}_4\text{-N}$  oxidation rate [60]. The optimum applied power supply was determined as 12 W, taking efficiency and energy consumption into account because current density increased with applied electric voltage, resulting in higher energy consumption [61,62].

### 3.6. Analysis of X-ray diffraction methodology and scanning electron microscopy

Fig. 8 illustrates the XRD pattern of struvite samples obtained for pure struvite and recovered struvite. The orthorhombic space group analysis of the sample ( $a = 6.9950$ ,  $b = 6.1420$ , and  $c = 11, 2180$ ) agrees well with the results. The crystalline phases and d-values obtained from the XRD were compared with the JCPDS data. The figure shows an XRD pattern that fits the JCPDS files, suggesting that the sample was struvite crystalline [63,64].

The morphology of recovered struvite was observed with SEM, as shown in Fig. 9A. During precipitation and crystallization, a heterogeneous form of crystals developed, consisting of bars, bricks, and rectangular shapes. Energy-dispersive X-ray analysis is used to identify the sample's elemental composition. Fig. 9B shows the EDX spectrum of the struvite crystal. The chemical composition of the formed struvite crystals revealed the presence of Mg, P, N, and O, with an Mg/P ratio of 1.32, which is consistent with the indicated chemical composition [65].

## 4. Conclusions

The system described here is a primary type of fuel cell used to study the principle of struvite recovery. The following conclusions can be drawn from the results obtained:

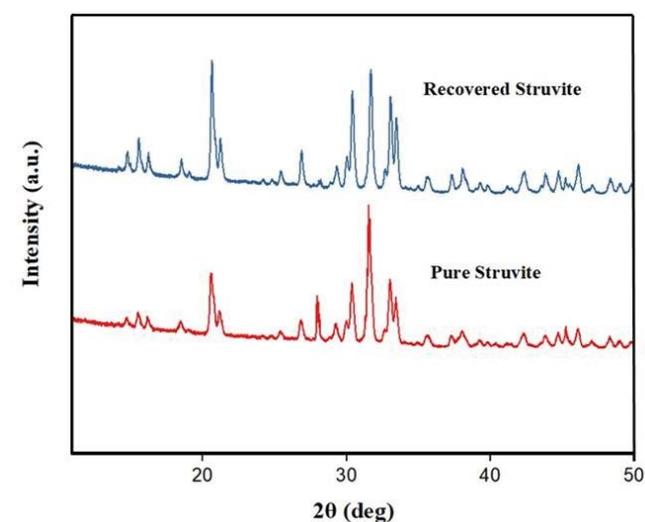


Fig. 8. XRD analysis of recovered struvite.

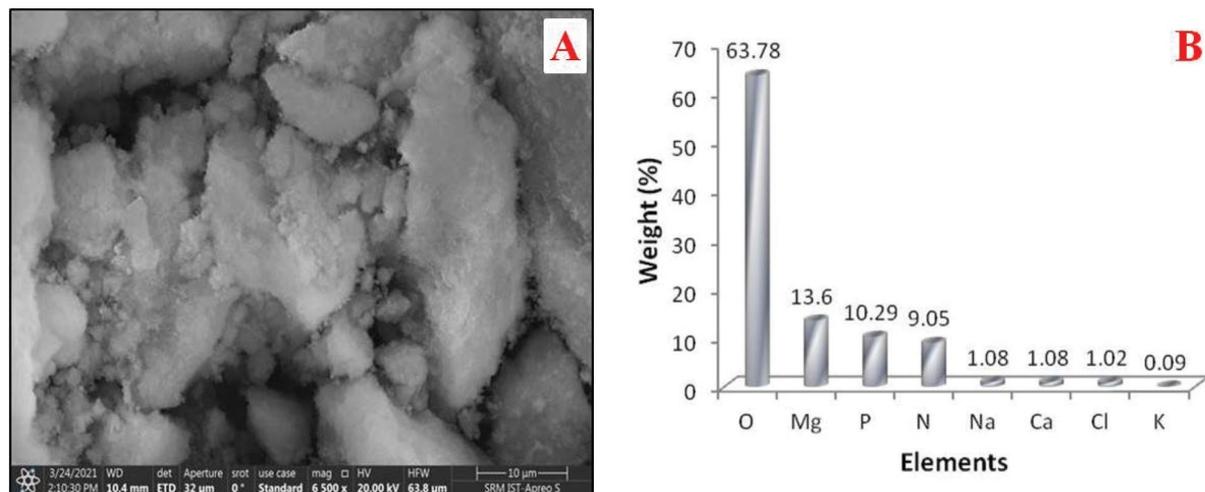


Fig. 9. Scanning electron microscope (SEM) analysis/EDX.

- The energy required to achieve a significant amount of struvite precipitation was found to be relatively high, making NaCl dissolution a critical parameter.
- The struvite was crystallized at different voltages with different proportions of NaCl concentrations. The 0.1% NaCl was found to be significant, and it was further used for struvite crystallization on the three different power supplies (5 W, 12 W and 22.5 W), and 12 W was found to be optimal.
- Purity, mineralogy, and morphology were assessed using XRD and SEM. EDS confirmed the presence of Mg, P, O, Cl, and N.
- Electrochemical recovery of struvite possesses various advantages over other methods as this method is environmentally compatible, easy to use, versatile, amenable to automation, cost-effective, energy-efficient, and safe. It overcomes the many limitations incorporated by other methods.

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#### References

- [1] R.H.E.M. Koppelaar, H.P. Weikard, Assessing phosphate rock depletion and phosphorus recycling options, *Global Environ. Change*, 23 (2013) 1454–1466.
- [2] P. Walan, S. Davidsson, S. Johansson, M. Höök, Resources, conservation, and recycling phosphate rock production and depletion: regional disaggregated modeling and global implications, *Resour. Conserv. Recycl.*, 93 (2014) 178–187.
- [3] I.Y.R. Odegard, E. Van der Voet, The future of food—scenarios and the effect on natural resource use in agriculture in 2050, *Ecol. Econ.*, 97 (2014) 51–59.
- [4] V. Linehan, S. Thorpe, N. Andrews, Y. Kim, F. Beaini, *Food Demand to 2050: Opportunities for Australian Agriculture*, Algebraic Description of Agrifood Model, ABARES, Canberra, Australia, 2012.
- [5] R.B. Chowdhury, G.A. Moore, A.J. Weatherley, M. Arora, Key sustainability challenges for the global phosphorus resource, their implications for global food security, and options for mitigation, *J. Cleaner Prod.*, 140 (2017) 945–963.
- [6] A.A. Al-Gheethi, A.N. Efaq, J.D. Bala, I. Norli, M.O. Abdel-Monem, M.A. Kadir, Removal of pathogenic bacteria from sewage-treated effluent and biosolids for agricultural purposes, *Appl. Water Sci.*, 8 (2018) 1–25.
- [7] P.J. Withers, C. Neal, H.P. Jarvie, D.G. Doody, Agriculture and eutrophication: where do we go from here?, *Sustainability*, 6 (2014) 5853–5875.
- [8] H. Kroiss, H. Rechberger, L. Egle, Phosphorus in Water Quality and Waste Management, S. Kumar, Ed., *Integrated Waste Management*, IntechOpen Book Series, London, UK, 2011, pp. 181–214.
- [9] R. Bhatia, D. Jain, Water quality assessment of lake water: a review, *Sustainable Water Resour. Manage.*, 2 (2016) 161–173.
- [10] R.O. Carey, K.W. Migliaccio, Contribution of wastewater treatment plant effluents to nutrient dynamics in aquatic systems: a review, *Environ. Manage.*, 44 (2009) 205–217.
- [11] E. Kavitha, E. Poonguzhali, D. Nanditha, A. Kapoor, G. Arthanareeswaran, S. Prabhakar, Current status and future prospects of membrane separation processes for value recovery from wastewater, *Chemosphere*, 291 (2021) 132690, doi: 10.1016/j.chemosphere.2021.132690.
- [12] C.J. Dawson, J. Hilton, Fertiliser availability in a resource-limited world: production and recycling of nitrogen and phosphorus, *Food Policy*, 36 (2011) S14–S22.
- [13] J.T. Bunce, E. Ndam, I.D. Ofiteru, A. Moore, D.W. Graham, D.W. Graham, A review of phosphorus removal technologies and their applicability to small-scale domestic wastewater treatment systems, 6 (2018) 1–15.
- [14] S. Sengupta, T. Nawaz, J. Beaudry, Nitrogen and phosphorus recovery from wastewater, *Curr. Pollut. Rep.*, 1 (2015) 155–166.
- [15] A. Siciliano, C. Limonti, G.M. Curcio, Advances in struvite precipitation technologies for nutrients removal and recovery from aqueous waste and wastewater, *Sustainability*, 12 (2020) 7538, doi: 10.3390/su12187538.
- [16] L. Zeng, X. Li, Nutrient removal from anaerobically digested cattle manure by struvite precipitation, *J. Environ. Eng. Sci.*, 5 (2006) 285–294.
- [17] I. Merino-Jimenez, V. Celorrio, D.J. Fermin, J. Greenman, I. Ieropoulos, Enhanced MFC power production and struvite recovery by the addition of sea salts to urine, *Water Res.*, 109 (2017) 46–53.
- [18] A. Hug, K.M. Udert, Struvite precipitation from urine with electrochemical magnesium dosage, *Water Res.*, 47 (2012) 289–299.
- [19] S. Kiruthika, R. Jeyalakshmi, M.P. Rajesh, Optimization studies on the production of struvite from human urine–waste into value, *Desal. Water Treat.*, 155 (2019) 134–144.

- [20] S. Kiruthika, S. Samdavid, R. Jeyalakshmi, M.P. Rajesh, Investigation of hydrodynamics of inverse fluidized bed reactor (IFBR) for struvite ( $\text{NH}_4\text{MgPO}_4 \cdot 6\text{H}_2\text{O}$ ) recovery from urban wastewater, *Chem. Pap.*, 76 (2021) 361–372.
- [21] C.M. Mehta, D.J. Batstone, Nucleation and growth kinetics of struvite crystallization, *Water Res.*, 47 (2013) 2890–2900.
- [22] K.S. Le Corre, E. Valsami-Jones, P. Hobbs, B. Jefferson, S.A. Parsons, Struvite crystallisation and recovery using a stainless steel structure as a seed material, *Water Res.*, 41 (2007) 2449–2456.
- [23] K. Suzuki, Y. Tanaka, K. Kuroda, D. Hanajima, Y. Fukumoto, T. Yasuda, M. Waki, Removal and recovery of phosphorous from swine wastewater by demonstration crystallization reactor and struvite accumulation device, *Bioresour. Technol.*, 98 (2007) 1573–1578.
- [24] M.I.H. Bhuiyan, D.S. Mavinic, F.A. Koch, Thermal decomposition of struvite and its phase transition, *Chemosphere*, 70 (2008) 1347–1356.
- [25] C.A. Martínez-Huitle, S. Ferro, Electrochemical oxidation of organic pollutants for the wastewater treatment: direct and indirect processes, *Chem. Soc. Rev.*, 35 (2006) 1324–1340.
- [26] S.P.S. Badwal, S.S. Giddey, C. Munnings, A.I. Bhatt, A.F. Hollenkamp, Emerging electrochemical energy conversion and storage technologies, *Front. Chem.*, 2 (2014) 79, doi: 10.3389/fchem.2014.00079.
- [27] D.J. Kruk, M. Elektorowicz, J.A. Oleszkiewicz, Struvite precipitation and phosphorus removal using magnesium sacrificial anode, *Chemosphere*, 101 (2014) 28–33.
- [28] S. Ben Moussa, G. Maurin, C. Gabrielli, M.B. Amor, Electrochemical precipitation of struvite, *Electrochem. Solid-State Lett.*, 9 (2006) C97–C101.
- [29] P. Ledezma, P. Kuntke, C.J.N. Buisman, J. Keller, S. Freguia, Source-separated urine opens golden opportunities for microbial electrochemical technologies, *Trends Biotechnol.*, 33 (2015) 214–220.
- [30] A. Hug, K.M. Udert, Struvite precipitation from urine with electrochemical magnesium dosage, *Water Res.*, 47 (2013) 289–299.
- [31] X. Zhou, Y. Chen, An integrated process for struvite electrochemical precipitation and ammonia oxidation of sludge alkaline hydrolysis supernatant, *Environ. Sci. Pollut. Res.*, 26 (2019) 2435–2444.
- [32] A. Dura, C.B. Breslin, Electrocoagulation using aluminium anodes activated with Mg, In and Zn alloying elements, *J. Hazard. Mater.*, 366 (2019) 39–45.
- [33] B.E. Logan, M.J. Wallack, K.Y. Kim, W. He, Y. Feng, P.E. Saikaly, Assessment of microbial fuel cell configurations and power densities, *Environ. Sci. Technol. Lett.*, 2 (2015) 206–214.
- [34] S.F. Ketep, A. Bergel, A. Calmet, B. Erable, Stainless steel foam increases the current produced by microbial bioanodes in bioelectrochemical systems, *Energy Environ. Sci.*, 7 (2014) 1633–1637.
- [35] W.L. Chou, C.T. Wang, K.Y. Huang, Effect of operating parameters on indium(III) ion removal by iron electrocoagulation and evaluation of specific energy consumption, *J. Hazard. Mater.*, 167 (2009) 467–474.
- [36] C. Santoro, I. Ieropoulos, J. Greenman, P. Cristiani, T. Vadas, A. Mackay, B. Li, Power generation and contaminant removal in single chamber microbial fuel cells (SCMFCs) treating human urine, *Int. J. Hydrogen Energy*, 38 (2015) 11543–11551.
- [37] K. Hirooka, O. Ichihashi, Phosphorus recovery from artificial wastewater by microbial fuel cell and its effect on power generation, *Bioresour. Technol.*, 137 (2013) 368–375.
- [38] O. Ichihashi, K. Hirooka, Removal and recovery of phosphorus as struvite from swine wastewater using microbial fuel cell, *Bioresour. Technol.*, 114 (2012) 303–307.
- [39] J. You, J. Greenman, C. Melhuish, I. Ieropoulos, Electricity generation and struvite recovery from human urine using microbial fuel cells, *J. Chem. Technol. Biotechnol.*, 91 (2016) 647–654.
- [40] F. Fischer, C. Bastian, M. Happe, E. Mabillard, N. Schmidt, Microbial fuel cell enables phosphate recovery from digested sewage sludge as struvite, *Bioresour. Technol.*, 102 (2011) 5824–5830.
- [41] I. Merino-Jimenez, V. Celorrio, D.J. Fermin, J. Greenman, I. Ieropoulos, Enhanced MFC power production and struvite recovery by the addition of sea salts to urine, *Water Res.*, 109 (2017) 46–53.
- [42] Y. Ye, H.H. Ngo, W. Guo, Y. Liu, S.W. Chang, D.D. Nguyen, J. Ren, Y. Liu, X. Zhang, Feasibility study on a double chamber microbial fuel cell for nutrient recovery from municipal wastewater, *Chem. Eng. J.*, 358 (2019) 236–242.
- [43] S. Kiruthika, R. Jeyalakshmi, M.P. Rajesh, Struvite recovery from human urine in inverse fluidized bed reactor and evaluation of its fertilizing potential on the growth of *Arachis hypogaea*, *J. Environ. Chem. Eng.*, 9 (2021) 104965, doi: 10.1016/j.jece.2020.104965.
- [44] Q. Li, S. Wang, L. Wang, L. Zhang, X. Wan, Z. Sun, The recovery of phosphorus from acidic ultra-high phosphorous wastewater by the struvite crystallization, *Water*, 12 (2020) 946, doi: 10.3390/w12040946.
- [45] N. Kumar, P. Singh, S. Kumar, Physical, X-ray diffraction and scanning electron microscopic studies of uroliths, *Indian J. Biochem. Biophys.*, 43 (2006) 226–232.
- [46] M.A.P. Manzoor, M. Mujeerurrahman, S. Ram, P.D. Rekha, Investigation on growth and morphology of in vitro generated struvite crystals, *Biocatal. Agric. Biotechnol.*, 17 (2019) 566–570.
- [47] A. Adnan, D.S. Mavinic, F.A. Koch, Pilot-scale study of phosphorus recovery through struvite crystallization examining the process feasibility, *J. Environ. Eng. Sci.*, 2 (2003) 315–324.
- [48] C.C. Wang, X.D. Hao, G.S. Guo, M.C.M. Van Loosdrecht, Formation of pure struvite at neutral pH by electrochemical deposition, *Chem. Eng. J.*, 159 (2015) 280–283.
- [49] R.D. Cusick, M.L. Ullery, B.A. Dempsey, B.E. Logan, Electrochemical struvite precipitation from digestate with a fluidized bed cathode microbial electrolysis cell, *Water Res.*, 54 (2014) 297–306.
- [50] C.T. Wang, W.L. Chou, Y.M. Kuo, Removal of COD from laundry wastewater by electrocoagulation/electroflotation, *J. Hazard. Mater.*, 164 (2009) 81–86.
- [51] L. Li, Y. Liu, Ammonia removal in electrochemical oxidation: mechanism and pseudo-kinetics, *J. Hazard. Mater.*, 161 (2009) 1010–1016.
- [52] O. Abdelwahab, N.K. Amin, E.Z. El-ashtoukhy, Electrochemical removal of phenol from oil refinery wastewater, *J. Hazard. Mater.*, 163 (2009) 711–716.
- [53] M.I.H. Bhuiyan, D.S. Mavinic, R.D. Beckie, A solubility and thermodynamic study of struvite, *Environ. Technol.*, 28 (2007) 1015–1026.
- [54] S.I. Lee, S.Y. Weon, C.W. Lee, B. Koopman, Removal of nitrogen and phosphate from wastewater by addition of bittern, *Chemosphere*, 51 (2003) 265–271.
- [55] U. Ghimire, M. Jang, S.P. Jung, D. Park, S.J. Park, H. Yu, S.E. Oh, Electrochemical removal of ammonium nitrogen and COD of domestic wastewater using platinum coated titanium as an anode electrode, *Energies*, 12 (2019) 883, doi: 10.3390/en12050883.
- [56] A. Rezagama, M. Hibbaan, M.A. Budihardjo, The process of removing nitrogen by using tubular plastic media, 2508 (2017) 4915–4922.
- [57] A.R. Purwono, M. Hibbaan, M.A. Budihardjo, Ammonia-nitrogen ( $\text{NH}_3\text{-N}$ ) and ammonium-nitrogen ( $\text{NH}_4^+\text{-N}$ ) equilibrium on the process of removing nitrogen by using tubular plastic media, *J. Mater. Environ. Sci.*, 8 (2017) 4915–4922.
- [58] Y. Vanlangendonck, D. Corbisier, A. Van Lierde, Influence of operating conditions on the ammonia electro-oxidation rate in wastewaters from power plants (ELONITA™ technique), *Water Res.*, 39 (2005) 3028–3034.
- [59] M. Darwish, A. Aris, M.H. Puteh, M.Z. Abideen, M.N. Othman, Ammonium-nitrogen recovery from wastewater by struvite crystallization technology, *Sep. Purif. Rev.*, 45 (2016) 261–274.
- [60] P. Seruga, M. Krzywonos, J. Pyżanowska, A. Urbanowska, H. Pawlak-Kruczek, L. Niedźwiecki, Removal of ammonia from the municipal waste treatment effluents using natural minerals, *Molecules*, 24 (2019) 3633, doi: 10.3390/molecules24203633

- [61] Y. Liu, L. Li, R. Goel, Kinetic study of electrolytic ammonia removal using Ti/IrO<sub>2</sub> as anode under different experimental conditions, *J. Hazard. Mater.*, 167 (2009) 959–965.
- [62] F. Bouamra, N. Drouiche, D. Si, H. Lounici, Treatment of water loaded with orthophosphate by electrocoagulation, *Procedia Eng.*, 33 (2012) 155–162.
- [63] Joint Committee for Powder Diffraction Standards Reference Card Number 20–0685.
- [64] V.B.Suryawanshi, R.T.Chaudhari, Synthesis and characterization of struvite-k crystals by agar gel, *J. Crystallization Process Technol.*, 4 (2014) 212, doi: 10.4236/jcpt.2014.44026.
- [65] X. Sun, J. Ouyang, F. Wang, Y. Xie, Formation mechanism of magnesium ammonium phosphate stones: a component analysis of urinary nanocrystallites, *J. Nanomater.*, 2015 (2015) 498932, doi: 10.1155/2015/498932.