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Phosphate and nitrogen removal by iron produced in electrocoagulation reactor

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ABSTRACT

In this study, concentrated iron solution generated by electrocoagulation (EC) process was used for the removal of nutrients from human urine (yellow water). Yellow water was prepared synthetically with major components: nitrogen and phosphorus. As a result of the pH adjustment, nitrogen and phosphorus, which are present in urine, react with electrochemically generated iron solution and then accumulate in the sludge by precipitation. All experiments were done under different pH values for the evaluation of this novel EC nutrient removal process. The effect of the EC treatment on the removal of nutrients was followed by total nitrogen and phosphate measurements and Fourier transform infrared spectroscopy (FTIR). The effects of treatment parameters (pH, iron/phosphate, iron/nitrogen, and iron/nutrient ratios) on the performance of the EC treatment were also examined for the evaluation of applicability of this novel EC method for the treatment of yellow waters. The results show that the pH increase improved phosphate removal efficiencies at definite molar nutrient ratios. The phosphate removal efficiency was approximately 98% at pH 8 for 1:1 of iron/nutrient molar ratio and on the other hand, nitrogen removal was only 21% at pH 8 for 4:1 of iron/urea molar ratio. Total organic carbon (TOC) removal efficiency was reached to 26% of the process. FTIR adsorption spectra were discussed for the sludge obtained.

Keywords: Electrocoagulation; Iron electrode; Nutrient removal; Human urine; Yellow water

1. Introduction

Phosphorus and nitrogen are essential for all living matter, including bacteria, plants, and animals. On the other hand, with the increased usage of fertilizers for agricultural activities and wastewater discharges, the nutrient level of many lakes and rivers has increased dramatically. Excessive nutrients were loaded into water bodies, resulting in eutrophication, fast growth of algae and other plankton, and their presence caused many water quality problems [1]. Eutrophication is critic for fishes, drinking water supplies, affects the aesthetics of recreational areas, and the ability to navigate through rivers and lakes. Commonly, phosphorus is the rate-limiting nutrient for the aquatic organisms in the freshwater ecosystems [2].

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Although phosphorus is one of the essential nutrients for life, phosphate rocks are limited in the world [3]. It is estimated that phosphorus resources will serve for 100–250 years. Around 80% of phosphate produced by the world's industry today is used in fertilizers, with further 5% being used to supplement animal feeds [4]. For a more sustainable future, all available resources should be evaluated [5]. For this reasons, both the removal and recovery of nutrients, especially phosphate, from wastewater have been a growing concern.

Human urine (yellow water) is the largest contributor of nutrients to municipal wastewater. Separating and collecting human urine from source can be a promising alternative for wastewater management causing the sustainability of nutrient recovery [6]. It takes only 1% (by volume) of municipal wastewater but contains the major part of human daily excretion of nitrogen (N), phosphorous (P), and potassium (K) around 80–90%, 50–80%, and 70–90%, respectively [7–10].

Several benefits of yellow water separation and pretreatment provide higher treatment capacity and better effluent quality of existing wastewater treatment plants with lower operation costs [8,11–13].

Separated human urine can be used directly to the farmland area as a fertilizer for plants and it would be very effective as a mineral fertilizer [14]. On the other hand, direct application of the human urine to the farmland area has some problems. When collecting and transporting its large quantities from urban areas to agricultural lands, social taboos and possible health risks are associated with high amount of hormones, pharmaceuticals, etc., and high salt content in the yellow wastewater [11,14–17].

Methods for the nutrient removal from municipal wastewater are evaporation, reverse osmosis, nitrification, precipitation, ammonia stripping, electrodialysis, nanofiltration, and advanced oxidation [18–20]. The mostly used phosphorus and nitrogen removal methods are chemical precipitation and biological nutrient removal. Biological nutrient removal requires complicated operating conditions and plant structure. Chemical precipitation is more useful because of its simplicity and cost-effectiveness. But, on the other hand, the use of chemicals can increase the cost of the treatment [1].

Attractive way to recover and treat wastewaters like yellow water having high nitrogen and phosphorus content is using struvite precipitation. It was reported to be especially effective for phosphorous recovery with about 90% of human urine [5,21]. Struvite recovery worked with synthetic urine, and phosphorus removal reported the average of 62% [22]. To enhance the NH_4-N removal, high amounts of external P and Mg source should be added. However, the high cost of Mg salts and P consumption for the effective removal of ammonia would become the main obstacle to the wide application of the struvite precipitation process [3].

In recent years, electrochemical methods have been successfully used to treat wastewater, and these methods could be an alternative method for the nutrient removal from human urine. These methods have some advantages over chemical treatment methods, e.g. less coagulant ions are required, no addition of chemicals is needed, relatively low area demand, low investment cost, and small volume of sludge produced, comparing with those in classical chemical process [23–25]. On the other hand, the electrochemical process efficiency depends on several factors such as electric voltage, ionic concentration, electrode material, and reaction time and temperature, and optimizing such factors plays an important role to the success of the process [5].

The aim of this study is to determine the efficiency of nitrogen and phosphorus removal from synthetic human urine by electrochemically produced iron. This method is different from classical electrocoagulation (EC) processes. Phosphorus and nitrogen removal performances were determined for this novel EC nutrient removal process under different pH's and different molar ratios of produced iron to phosphorus, nitrogen, and nutrients.

2. Materials and methods

2.1. Chemicals and measurements

The analysis of phosphate was carried out by using yellow vanadomolybdophosphoric acid method by spectrophotometer (Hach-Lange DR 5000) according to the Standard Methods for Examination of Water and Wastewater [26]. Total nitrogen (TN) was measured by Total Organic Carbon (TOC) apparatus with Total Nitrogen Measuring Unit (Shimadzu TOC-V CPN-TNM-1). Iron concentrations generated in the reactor were determined by Inductively Coupled Plasma (ICP; Perkin-Elmer ICP).

2.2. Preparation of urine wastewater

In this study, human urine sample has been prepared synthetically by dissolving KH_2PO_4 and $CO(NH_2)_2$ (urea) in distilled water. KH_2PO_4 and $CO(NH_2)_2$, amount of synthetic urine is listed in Table 1 [27].

Table 1
Composition of synthetic human urine

Salt	g/L	mM
$CaCl_2 \cdot 2H_2O$	0.65	4.4
$MgCl_2 \cdot 6H_2O$	0.65	3.2
NaCl	4.6	78.7
Na_2SO_4	2.3	16.2
Na_3citrate–$2H_2O$	0.65	2.6
$Na_2(COO)_2$	0.02	0.15
KH_2PO_4[a]	4.2	30.9
KCl	1.6	21.5
NH_4Cl	1	18.7
$CO(NH_2)_2$[a]	25	417
$C_4H_7N_3O$	1.1	9.7

[a] Used for synthetic urine.

2.3. Electrocoagulation treatment reactor system

The reactor was constructed for iron production. The laboratory-scale reactor (4 L) was used during experimental studies (Fig. 1).

Two groups of alternating Fe electrodes being cathodes and anodes (by seven of each type) were arranged vertically. Distance between an anode and a neighboring cathode is 3 mm. The electrode system is monopolar. Before starting the experiment, iron electrodes were immersed in a solution of strong acid and cleaned.

3. Results and discussion

The mechanisms of reactions that happen in the electrochemical units are studied extensively. When direct current passes through the electrodes, metal ions dissolve and react with OH^- ions in water. Metal hydroxyls that can dissolve in water partially are being formed under appropriate pH level. This stage ends with the formation of colloidal particles. Hydroxyls form the nucleus of the colloidal particle, and the adsorption layer of cations and anions is being formed around the nucleus. So, the nucleus and the adsorption layer form the positively charged colloidal granule. Diffusion layer then occurs around the granule, and the particle becomes neutral. Some chemical reactions, which occur on electrodes and in the bulk wastewater, are shown below.

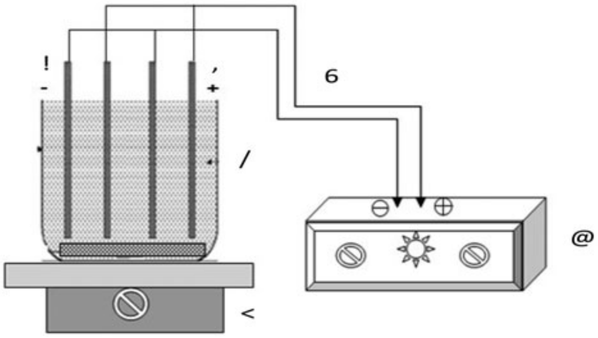


Fig. 1. A schematic diagram of the electrochemical reactor (1: cathode, 2: anode, 3: electrochemical cell, 4: electrochemical reactor containing parallel iron electrodes, 5: magnetic stirrer, 6: direct current source).

Reactions on the anode:

$$Fe^0 - 2e^- \rightarrow Fe^{2+}$$

$$Fe^{2+} + OH^- \rightarrow FeOH^+$$

$$FeOH^+ + OH^- \rightarrow Fe(OH)_2$$

$$Fe(OH)_2 \text{ (solution)} \rightarrow FeOH^+ + OH^-$$

$$FeOH^+ \rightarrow Fe^{2+} + OH^-$$

$$2H_2O \rightarrow 4H^+ + O_2 + 4e^-$$

In cathode:

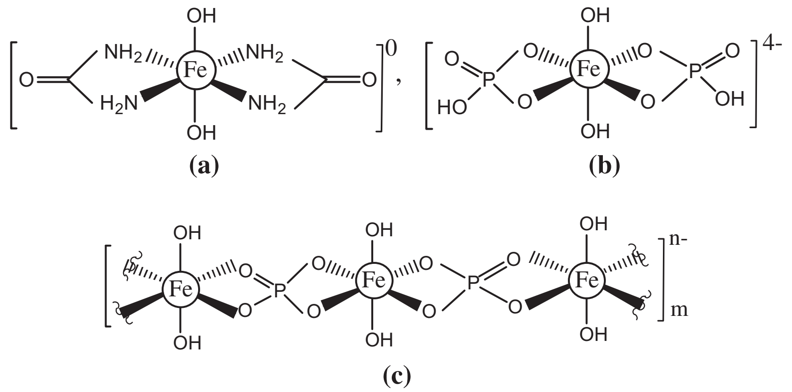
$$2H_2O + 2e^- \rightarrow H_2 + 2OH^-$$

The metal hydroxyls that are formed in the EC process have a high adsorption capacity. Coagulated particles attract and absorb micro-colloidal particles and ions from the wastewater. In the presence of nitrogen-containing urea molecule and phosphate ion (HPO_4^{2-}), octahedral complexes can be formed (see Fig. 2(a and b)).

For the first molecule, a neutral complex is being formed at the account of coordinate bond between Fe^{2+} ion and amino groups. Otherwise, covalent bonds are being formed by phosphate ion with Fe^{2+}. The formation of polymeric chains for the account of new coordinate possibilities of –P(OH)=O groups is not excluded as well (see Fig. 2(c)).

3.1. Effect of pH on the iron generation

After setting up the DC volts and DC current, the reactor was run and the experiment terminated after 60 min, while sampling every 10 min. Iron concentration generated in the reactor was measured by ICP. The time needed to obtain a high iron concentration is determined as a reaction time. This study differs from other traditional electrochemical approaches: first of



(a) **(b)**

(c)

Fig. 2. Prospective complexes of iron with urea and phosphate ion.

all, the iron solution is generated by electrochemical cell, and only then it reacts with synthetic wastewater outside the reactor.

The use of soluble anodes causes the pH increase in the solution, and the most considerable pH changes were observed in the reactor with iron anodes. As seen in Fig. 3, pH value in the reactor during the water electrolysis with dissolving iron anodes increases up to 9.5–10.0. Along with this, the final pH value of the solution depends not only on the concentration of the metal ions emitted but also on the initial pH of water as well.

During the initial periods of electrolysis (initial pH 4, 5), the pH increases sharply until a maximum value. After that, the pH stabilizes to a pseudo stationary value. Thus, the final pH value of the acidic solutions (initial pH 2, 3) under the electrolysis does not exceed 8.3–9.0.

It should be noted that the best yield of iron ions is observed for pH 2. By this, a low pH value of the solute is being observed for a long enough time, and only after 45 min of processing pH value becomes higher than 3. The corresponding iron quantity that passes into the solute exceeds 5 g ion/L.

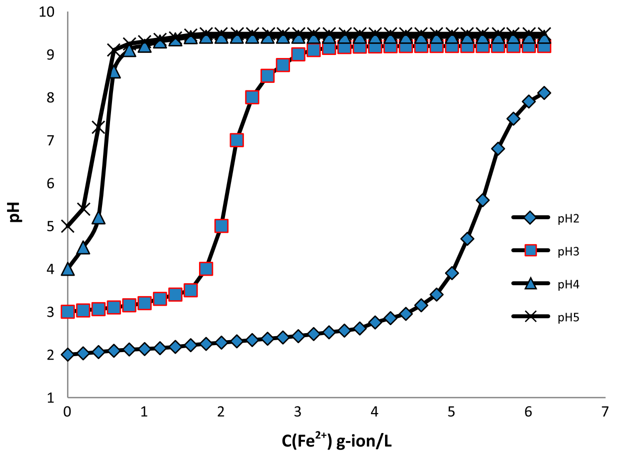


Fig. 3. The electrolite's pH vs. electrogenerated Fe^{2+} ions concentration and initial pH of solution (processing time—60 min; C_{NaCl}—2.5 g/L; current density— 30 mA/cm^2).

After measuring the iron concentration, different Fe/P, Fe/N, and Fe/Nt molar ratios were calculated. N is total nitrogen, P is phosphorus, and Nt indicates a combination of N and P.

3.2. *Effect of iron/phosphorus*

The effect of phosphate concentration on the phosphate removal by EC was studied on iron plate electrodes. To determine the most appropriate concentration of iron, different molar ratios of Fe/P were prepared, and phosphorus in the liquid phase was determined after precipitation. Fe/P molar ratios were taken as 0.3, 0.6, 1.2, 2.5, and 5. Fe and P concentrations were determined from calculations. Initial pH of wastewater is an effective parameter for the nutrient removal by EC. Initial pH was about 2, and it increased to the pH 5 and pH 8 with the pH adjustment. Phosphorus removal efficiency is shown in Fig. 4.

According to the experimental data, the maximum removal (about 99%). was observed at the Fe/P molar

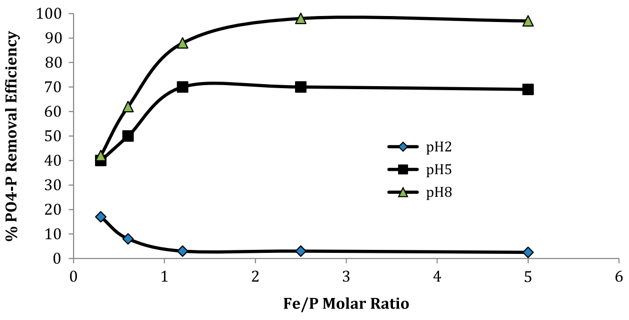


Fig. 4. Phosphorus removal efficiency with different Fe/P molar ratio (0.3–5) and pH 2–5–8.

ratio equal to 2.5. As to the samples containing phosphate at pH 8, phosphate removal efficiency increases with the increasing iron concentration. Maximum molar ratio was Fe/P = 5, and PO_4-P% removal for this ratio was 2% at pH 2, 34% at pH 5, and 97% at pH 8. The lowest molar Fe/P ratio was 0.3, and corresponding maximum PO_4-P% removal was about 40% for pH 5 and pH 8. From the obtained results of the experiments, efficiencies of phosphate removal at pH 8 for Fe/P molar ratios 0.3, 0.6, 1.2, 2.5, and 5 were found as 42, 62, 88, 99, and 97%, respectively. Thisleton et al. [28] worked on orthophosphate precipitation by adding iron (II) salt. Maximum removal was obtained of 85% at a molar ratio of 3.4:1 and decreased to 72% at 2:1, 60% at 1.5:1, and 52% at 1:1 at pH 8.0 and very low removals were at pH 6.5.

3.3. Effect of iron/urea molar ratio

Only the nitrogen-containing compounds were prepared by using urea ($CO(NH_2)_2$) and deionized water. After adding iron solution that had been obtained from the electrochemical process, the pH value was measured. It was about 2. pH value was increased to 5 and 8 by adding sodium hydroxide solution, and TN% (total nitrogen) removal efficiencies were investigated at different Fe/N molar ratios as shown in Fig. 5. pH 2, 5, and 8 were used in this study, and the maximum removal efficiency was 21% under Fe/N molar ratio equal to 4 and pH 8, as shown in Fig. 5. From the results of our experiments, efficiencies of nitrogen removal at pH 8 were calculated as 7, 17, 18.5, 19.5, and 21% for Fe/N molar ratios taken as 0.05, 0.5, 1, 2, and 4, respectively. Nitrogen removal could not be obtained at pH 2, and it was very low at pH 5.

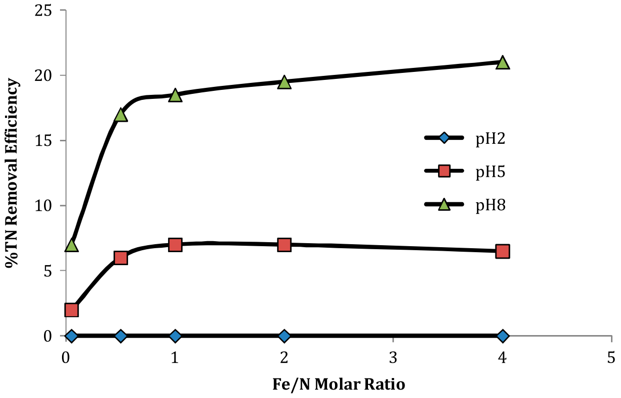


Fig. 5. Nitrogen removal efficiency with different Fe/N molar ratios (0.05–4) and pH 2–5–8.

3.4. Effect of iron/nutrient molar ratio

Both of the two nutrients (phosphate and urea) were used and called this mixture as Nt. Fe/Nt molar ratios were 0.06, 0.08, 0.15, 0.3, 0.6, 1, and 2. Iron solution generated by EC was added to the synthetic human urine prepared with phosphate and urea. P% and TN% removal at pH 2, 5, and 8 are shown in Figs. 6 and 7.

As seen in Fig. 5, efficiencies of phosphate removal are maximum 98% (Fe/Nt molar ratio is 1) and minimum 60% (Fe/Nt molar ratio is 0.06) at pH 8. For the Fe/Nt molar ratio equal to 0.3 at the same pH, 95% removal efficiency was observed. This is a satisfactory result for economic consideration. Even for very low pH (pH 2) maximum of 75% and minimum of 17% phosphate removal efficiencies were obtained for Fe/Nt molar ratio 1 and for Fe/Nt molar ratio 0.06, correspondingly. As known, pH of fresh human urine is about 5.5–6.5 and this study approached 95% and phosphate removal efficiency at pH 5 (Fe/Nt molar ratio 1). There is no need to increase the pH on human urine contrary to struvite process. Phosphate removal

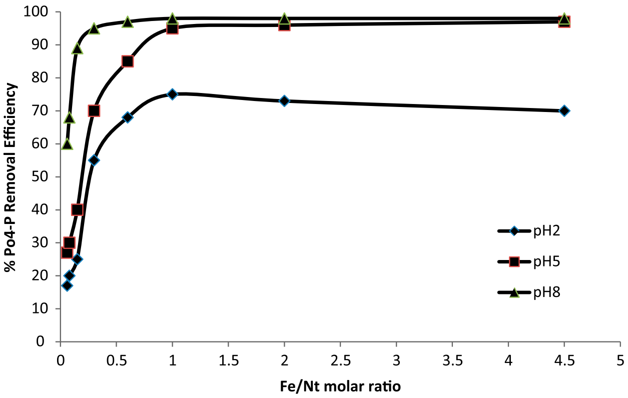


Fig. 6. P% removal efficiency with different Fe/Nt molar ratio (0.06–2) at pH 2–5–8.

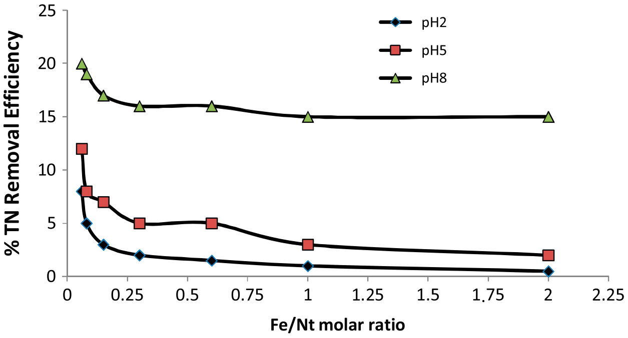


Fig. 7. TN% removal efficiency with different Fe/Nt molar ratio (0.06–2) at pH 2–5–8.

efficiencies were obtained as 95% for Fe/Nt molar ratio 1 and 27% for Fe/Nt molar ratio 0.06. Thistleton et al. [28] could get satisfactory removal results for orthophosphate at the pH values lower than 6.5.

When nitrogen and phosphate are put together to the same solution, nitrogen removal efficiency showed decreasing trend against to increasing iron/nutrient ratios as seen Fig. 7. This result is contrary to pure nitrogen solution as shown in Fig. 5. In Fig. 7, the nitrogen removal efficiency decreases with the increase in iron/nutrient molar ratios. The maximum removal was determined up to 20% at pH 8 and 0.06 of Fe/Nt molar ratio. At the same ratio and pH 5, the removal value was 12%.

3.5. FTIR analysis

By using Fourier transform infrared spectroscopy (FTIR) method, we studied some characteristics of the sediment obtained. Being the amorphous sludge of iron hydroxide, the sediment has a polymeric structure that looks like a number of mutually bound octahedral complexes [29,30] (Fig. 8).

FTIR spectrum of the FeOOH with characteristic bands ν(OH) at 3,371 cm^{-1}, δ(HOH) at 1,630 cm^{-1}, δ(OH) at 1,365 cm^{-1}, and γ(OH) at 698 is presented in Fig. 9 (curve 1). Weak peaks in the interval of 1,200–1,231 cm^{-1} are related to vibrations of OH_2 coordinated with Fe (Fe-OH_2).

In Fig. 9, separate FTIR spectra for urea (curve 2) and KH_2PO_4 (curve 3) are shown, so as a spectrum of their joint with iron hydroxide sediment (curve 3). As seen from curve 1, the area with frequencies 3,300–3,400 cm^{-1} belongs to valence N–H stretching. The band at 1,673 cm^{-1} is attributed to C=O in urea, and the band at 1,459 cm^{-1} is attributed to the C–N stretching.

Wide and smooth band that is observed at 2,671 cm^{-1} corresponds to O–H bond belonging to the

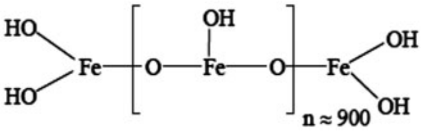


Fig. 8. Polymeric structure of iron hydroxide.

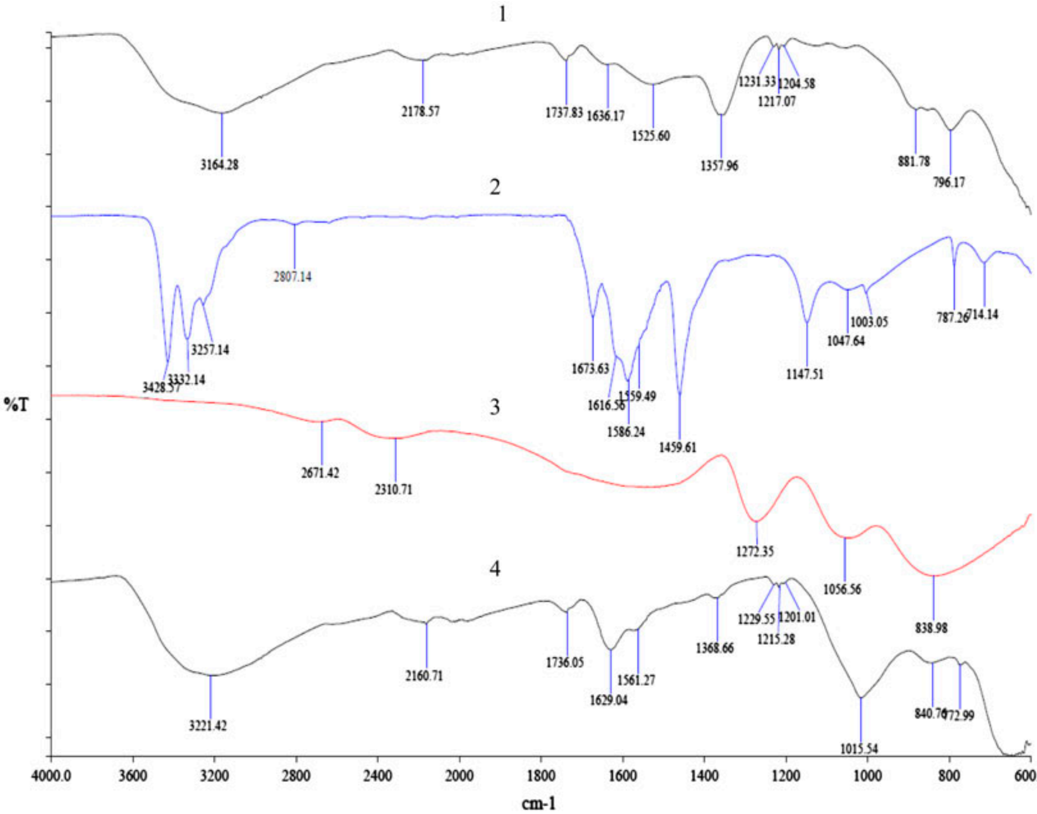


Fig. 9. FTIR adsorption spectra of the 600–4,000 cm^{-1} region: (1) iron hydroxide, (2) urea, (3) phosphate, (4) iron hydroxide with nutrients.

phosphate group (curve 2). The band at 1,272 cm^{-1} can be associated with vibrations on the P–O bond. The band at 838 cm^{-1} is attributed to the overlap of P–O stretching and hydrogen-bonded OH.

IR spectrum of the iron hydroxide with nutrients (urea plus phosphate) repeats, at the first approximation, the spectra of individual molecules. However, in some cases band shifts are observed, that can happen due to the weak adsorption bonds with iron hydroxide formation for both urea and phosphate anions.

For FTIR sediment (curve 3), the band extension is observed at 3,221 cm^{-1}, that is related to the hydrogen bonds formation and frequency of vibrations decrease. Characteristic band ν(OH) at 3,371 cm^{-1} shifts to 3,221 cm^{-1} and becomes wider. Peak at 1,636 cm^{-1} (C–O stretching of urea) does not change its position. There are a few weak bands at 1,200–1,230 (curve 3) instead of the band at 1,272 cm^{-1} (curve 2), and a deeper band appears at 1,015 cm^{-1}, that points to the reaction between iron and phosphate groups under the sediment formation. The band at 838 cm^{-1} keeps its position unchanged.

3.6. Operating cost

Operating cost during yellow wastewater treatment includes the costs of electricity, labor, maintenance, chemical reagents, sludge disposal, and equipments. In electrochemical process, the most important parameters that affect operating cost are consumed electrical energy and the cost of electrodes material. The operating costs investigated in this paper were the power cost for the electrocoagulation cell and the material cost due to the consumption of the iron electrode. The iron plate costs 0.87 €/kg, and electricity costs 0.09 €/kWh (Turkey). The applied current density is 3 A/m^2, and the average applied voltage is 8 V. Processing time is 60 min.

The energy consumptions and some operating parameters were presented in Table 2. When optimized, the total cost for yellow wastewater treatment by electrocoagulation is 0.20 €/m^3.

Table 2
Estimated cost for pilot-scale electrocoagulation process

Item	Cost (€/m^3)
Electrical energy prices (€/kWh)	0.09
Iron plate consumption ((€/kg)	0.02
Energy consumption (kWh/m^3)	0.08
Chemicals (€/m^3)	0.01
Approximate operating cost	0.20

4. Conclusion

In this study, the electrochemical removal of phosphate from synthetic human urine by using iron plates as electrode materials has been investigated. From the obtained results, it is seen that phosphate removal from synthetic human urine with iron solution produced by electrocoagulation is an effective process. It represents very promising novel method for those pH value of fresh and stored urine that are around pH 6 and pH 9 naturally. Therefore, chemical additions might not be needed. At the next experimental step, it will be better obtaining new results by using real human urine samples (fresh and stored).

Optimal selection of the operational conditions of EC indicated that current density, retention time, electrode type, and pH value affected the pollutants removal. The research elicited that the current density should be 3 A/m^2, the retention time is to be 60 min, and pH 8 for synthetic human urine, as the optimal reaction conditions. For the synthetic urine with initial pH adjusted as 8, nearly 100% efficiency of P removal and 20% TN removal was reached with iron electrodes after 60 min of the electrolysis time. The P removal efficiency increased, but TN removal efficiency decreased with the increase in iron concentration generated during the EC process. Also, 26% of TOC removal was obtained at pH 8 and Fe/Nt molar ratio equal to 0.3. The electrocoagulation processes come into prominence as compared with conventional nitrogen/phosphate removal methods because of less chemicals usage, simple equipment requirements and easy operation. EC is an evolving technology that is being effectively applied today for the nutrients removal.

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